Desalination and Water Treatment

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ABSTRACT

One of the main cultural heritages associated with South-East Asian people’s way of life and economy is batik. Not only batik is a part of textile industries, playing an important role in industrial development, it also, like other wet textile processes, generates huge amounts of hazardous wastewater. Due to the presence of wax in the batik production process an efficient wastewater pre-treatment is necessary to appropriately prepare related effluent conditions for further conventional wastewater treatment. Accordingly, in this study, the application of a baffle separation tank to treat simulated batik effluent as an innovative wastewater pre-treatment method was investigated. Therefore, two sets of simulated batik wastewaters containing constant amounts of chemical components (wax, dye, and sodium silicate with different reactive dyes) were tested. In the next step, the removal efficiency of the discussed samples’ chemical components was calculated based on pre- and post-process analytical experiments during an hour-long pre-treatment run. Wax, sodium silicate, and reactive dye removal through the runs were in the ranges of 92–95, 32–42, and 2–5%, respectively. The results showed slight decreases in pH, COD, and conductivity values in the post-treated samples, the differences of which were within the ranges of 0.2–0.6, 52–107 mg/L and 9–27 μS/cm, respectively. Moreover, this technique presented an effective rate of reduction for effluent samples’ existing heavy metal concentration.

Keywords: Textile wastewater pre-treatment; Batik wastewater; Baffle separation tank; Sustainable treatment technique

1. Introduction

Societies’ industrial and economic growth plays a significant role in future sustainable international action [1]. Inappropriate consumption of vital resources, as a result of this development process, can give rise to critical issues influencing every social habitat. Water resources, as the most basic, limited vital element, are seriously affected by industrial action [2].

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Among all industrial processes, the textile wet process is known to generate the highest hazardous wastewater because of the demand for it [1,3,4]. The batik industry, as a part of the textile industry, allocates the biggest share of local South-East Asian families' income source to itself based on its cultural and artistic value [5,6]. Batik's distinctive feature, which makes this industry different from other textile industries, is the application of paraffin wax as physical resistance in the motif design stage [7]. This means that not only batik wastewater is one of the most difficult effluents to treat by conventional wastewater treatment technique, but also that the application of effective pretreatment is vital [3,8]. Although various researchers have considered different techniques to treat industrial and domestic wax and oily effluents [9,10], fewer studies have highlighted the treatment of batik wastewater, including paraffin wax derivatives [11,12]. The applications of different synthetic polymer fibers as oil droplets and particle removal techniques have been studied by several researchers [13–17]. Some studies report on modified polymeric membranes and deployment of fibers' physical and chemical properties (oleophilicity and oleophobicity parameters) to reach high efficiency in continuous oil/water industrial wastewater separation [14,15,18–20]. These methods, due to operational benefits such as simple manufacturing operation conditions and user-friendliness, can be applied in petroleum and its subcategory industries. Moreover, some cellulosic natural fibers, based on hydrogen bond formation and van der Waals forces among hydroxyl groups with free hydrogen groups in effluents’ oil and wax, can be shown to have high wax absorption and separation during wastewater treatment stages [21,22]. On the other hand, some industrial oil and wax separation methods, such as gravity-based separation techniques followed by skimming methods, and dissolved air flotation and emulsifier applications, can generally be effectively applied in refineries, dairies, textiles, and other industrial processes [23–26]. Although the related methods, based on their operational advantages, can be deployed in different industrial areas, almost all batik industries employ routine cooling and skimming techniques for wax removal during the respective wastewater treatments [7], due to different types of applied physical and chemical components like wax (paraffin and plant resin) and to their traditional family-based structure [5,7]. Despite the fact that the method is easy to use and cheap to operate, it contravenes sustainability principles since it requires excessive operational time [21,27].

Not only these mentioned techniques are incompatible with continuous wastewater treatment, they also require a considerably long cooling down period, which intensifies the potential for harmful chemical and biological impacts on the environment [28]. Accordingly, this study investigates the baffle separation tank pre-treatment technique, which is based on baffle collision impact on the melted wax droplets' flotation, facilitation, and phase transformation effects on simulated batik wastewater's chemical and environmental parameters.

2. Experimental section

2.1. Materials

The wax, sodium silicate, and all the different types of fiber reactive dyes that were used in this study were obtained from TMS ART Company (KL, Malaysia), and were used directly without any purification. (Reactive Red 194, λ\text{max}: 505; Reactive Blue 15, λ\text{max}: 674; Reactive Black 5, λ\text{max}: 600; Reactive Orange 16, λ\text{max}: 492; Reactive Yellow 145, λ\text{max}: 419; sodium silicate, Na\text{2}O\text{3Si}, paraffin (wax), C\text{20H}4\text{2}–C\text{40H}8\text{2}).

2.2. Pre-treatment device equipment

The application of a specially designed baffle separation tank in a physical pre-treatment wax removal process for simulated batik industry wastewater has been investigated. Accordingly, a rectangular basin-shaped separation tank with eight different heights of parallel-positioned baffle layers, which are made by Plexiglas, has been used (Fig. 1). The related operation properties are represented in Table 1.

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Each pre-treatment cycle was set to run in an optimal tank volume of 30 L with a constant value of flow rate, at a temperature and time of 570 L/h, 70°C, and 1 h, respectively. Each experiment was repeated at least three times in order to ensure its accuracy and reproducibility.

2.3. Sample preparation

Based on the characteristics of actual untreated batik wastewater, each wastewater sample was simulated in 30 L for each separation run in constant temperature (70°C) [29–31]. All samples included a mix of fiber reactive dye, sodium silicate, and wax in concentrations of 16 mg/L, 1 g/L, and 7.7 g/L, respectively.

2.4. Sample coding

To simplify the presentation of the data, the dyes used in this study were coded in (Table 2).

2.5. Analytical methods

The wax (RW%), sodium silicate (RS%), and dye (RD%) removal efficiency of each run was calculated using Eqs. (1)–(3):

\[
RW% = 1 - \frac{W_R}{W_F} \times 100 \tag{1}
\]

\[
RS% = 1 - \frac{C_{Sp}}{C_{Sf}} \times 100 \tag{2}
\]

\[
RD% = 1 - \frac{C_{Dp}}{C_{Df}} \times 100 \tag{3}
\]

where \(W_R\) and \(W_F\) are removed wax and feed wax weight, \(C_{Sp}\) and \(C_{Sf}\) are feed and permeate sodium silicate concentration, and \(C_{Dp}\) and \(C_{Df}\) are permeate and feed dye concentration. The sodium silicate concentration of the pre-treated samples in each experiment was prepared for investigation using silicate (silicic acid) test reagents (1.00857.0001-MERCK, Germany). The efficiencies of dye and sodium silicate removal were measured by a UV–visible spectrophotometer (Perkin Elmer Lambda 25 UV/VIS Instrument L6020060, USA) at a visible wavelength (325–750 nm) and analyzed by integration with the absorbance curve.

The pH, conductivity, and chemical oxygen demand (COD) range of each simulated sample, pre- and post-treatment process, were measured using a Metrohm pH meter analyser, model 827 (Swiss); a Cheetah multi-parameter meter, model DZS-708 meter (China); HACH and a portable COD spectrophotometer, model DR/890 (USA). Furthermore, the value ranges of existing heavy metal elements in all pre- and post-treated simulated samples were measured using ICP Optima 4300 DV ICP-OES, Perkin Elmer (USA). Moreover, to simplify the exhibition of the achieved data in each pre-treatment run’s graphs and tables, pre- and post-baffle separation tank application named BB and AB, respectively.

3. Results and discussion

The effectiveness of the baffle separation tank on simulated batik wastewater pre-treatment efficiency in
relation to specific parameters including wax, dye, sodium silicate, and heavy metal removal is discussed in this section. Moreover, the COD, conductivity, and pH values of each sample pre- and post-treatment stage were surveyed.

3.1. Wax and sodium silicate removal efficiencies

Figs. 2 and 3 illustrate wax removal efficiency using a baffle separation tank for single- and mixed-simulated dyes, respectively. The related pre-treatment technique based on baffle layer arrangement has a strong influence on melted wax droplets during each cycle. In each cycle, the wax droplets hit the baffle layers in constant volume and speed. Consequently, this collision makes the floatation of wax droplets facilitate and accelerate the melted wax droplets’ phase change (from liquid to solid).

Generally, the baffle separation tank presents an operation based on two main factors: cooling area impact and collision effect. These factors principally influence the coalescence of the wax removal process. The baffle layers’ arrangements and the separation tank walls’ thermal properties, based on physical, coalescence, and thermodynamic laws [32,33], speed up coalescence of melted wax droplets and phase change. This phenomenon has also been reported in other studies [34–36]. As a result, more than 92.5% wax removal efficiency was achieved by using this pre-treatment technique in all simulated batik wastewater samples; the highest wax removal efficiency is related to navy blue and a mixture of five dyes (M5) with 94.9 and 94.5% efficiency in single and mixed dye samples, respectively (Figs. 2 and 3). However, the range of difference within all samples (single and mixed dyes) is insignificant (less than 2.4%). The operational conditions such as coalescence-melted wax droplet size, type of dye, and amount of air diffused during the treatment cycles can explain the minor changes [37]. The total achieved wax removal efficiency is absolutely in agreement with thermodynamic laws for changing kinetic energy into thermal energy and making the phase changes faster [38,39]. Correspondingly, based on the third law of thermodynamics, coalescence law and water media-specific heat capacity [32,33], the heat and phase transition actions happen easier and faster during pre-treatment stages. Moreover, the effect of sodium silicate on the wax removal process when using baffle separation tank pre-treatment is not negligible.

The sodium silicate concentrations after each pre-treatment demonstrate high reduction in value, which can be explained by two main parameters: the effect of existing melted paraffin wax in batik wastewater samples and the application of baffle pre-treatment operational conditions, such as air diffusion and temperature [40]. Generated CO₂ molecules (paraffin-free carbon and oxygen molecules) in the presence of dyes’ reactive groups during each cycle run not only make the wax droplets’ floatation increase, but also by a reaction of sodium silicate can generate more cracked, deposited silica [40]. In general, this phenomenon can improve efficiency of sodium silicate removal by the baffle separation tank pre-treatment technique. Table 3 illustrates the existing sodium silicate removal efficiency during each pre-treatment run. As the results show, here, the sodium silicate efficiency in all simulated samples is in the range of 32–44.5% in both single and mixed dye samples. The highest efficiency belongs to yellow and turquoise blue, and the mixes of five dyes and two dyes in single and mixture samples, respectively. The samples’ efficiency differences are directly related to the dye group’s reactivity and wax removal efficiencies [40,41]. Hence, the yellow, turquoise blue, and also the navy blue samples, with higher wax
removal efficiency, represent higher sodium silicate removal efficiency as well. The wax removal efficiency in mixed dye samples is in same range, but all related samples follow the same trend as single dyes.

3.2. Dye removal efficiency

The baffle separation tank pre-treatment technique, besides the wax removal application that is its main role and operational efficiency, can also slightly affect the batik wastewater’s other existing chemical components, such as sodium silicate and dye concentration. Although physical pre-treatment application demonstrates a high wax and sodium silicate removal efficiency ratio, it was negligible in reactive dye removal efficiency. Table 4 represents the results of fiber reactive dye concentration pre- and post-baffle separation tank application. The results indicate that the dye removal efficiency in all single and mixed dye samples is not considerable (2.7–4.9%). This can be explained by the lesser effect of baffle layers’ collision on dye component separation, which resulted in the same range of dye rejections in all simulated batik wastewater.

The highest dye removal values were seen in turquoise blue and navy blue with 4.3 and 3.8%, respectively. The higher removal rate of physical components results in more trapped dye separation. This in agreement with the higher wax and sodium silicate removal efficiencies within all single dye samples using the baffle separation tank. The mixed dye samples also follow the same trend, and the highest dye removal value belongs to the mixture of five dyes with 4.9%. This is not only due to higher removal efficiency of physical components’ removal, but also relates to higher concentrations of different reactive groups in the mixed samples. On the other hand, pre-treatment initial conditions including time, temperature, mixing, and air diffusion, as well as higher values of reactive groups in each samples, can cause the high hydrolysis rate of reactive dyes. This occurrence makes dyes’ reactivity rate lower; consequently, dye removal efficiency will decline. Therefore, the minority groups of the dye components can be removed by trapping in wax along with the other physical constituents.

3.3. COD value concentration

The existence of pollution of wax particles in batik wastewater, as well as the other common chemical and physical pollutants such as dyes and auxiliaries, in textile wastewater can have critical impacts on wastewater’s environmental and chemical parameters [5]. For instance, esthetic quality and dissolved oxygen concentration are directly affected by the existence of chemical components in batik wastewater [7]. One of the main parameters in each set of wastewater characterization criteria is COD. Not only dyes, sodium silicate, and other chemical auxiliaries can intensively increase COD value in batik wastewater, the presence of wax particles can also affect COD factor [42]. For this reason, the COD values in the baffle separation tank pre-treatment cycle run for each sample before and after the treatment process are measured and the results are shown in Figs. 4 and 5. The COD value reductions in all simulated wastewater samples after wax removal processing through use of the baffle separation tank are in the range of 55–107 mg/L. As the COD value results show, the baffle tank pre-treatment does not have distinct effects on COD’s rate of decrease. This phenomenon can be explained by removal of wax particles from simulated batik wastewater through application of physical separation by baffle separation tank. The COD value reduction results demonstrate that only the wax particles play a main role in pre-treatment runs, while the other chemical components such as dyes and sodium silicates do not have a vital role [7,42].

<table>
<thead>
<tr>
<th>Type</th>
<th>Dye rejection efficiency (%)</th>
</tr>
</thead>
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<tr>
<td>R</td>
<td>2.7</td>
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<tr>
<td>Y</td>
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<tr>
<td>TB</td>
<td>4.3</td>
</tr>
<tr>
<td>O</td>
<td>3.3</td>
</tr>
<tr>
<td>NB</td>
<td>3.8</td>
</tr>
<tr>
<td>M2</td>
<td>4.3</td>
</tr>
<tr>
<td>M3</td>
<td>3.5</td>
</tr>
<tr>
<td>M4</td>
<td>3.8</td>
</tr>
<tr>
<td>M5</td>
<td>4.9</td>
</tr>
</tbody>
</table>

Table 4
Dye removal efficiency in wastewater samples

Table 3
Sodium silicate removal efficiency in wastewater samples

<table>
<thead>
<tr>
<th>Type</th>
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<th>Y</th>
<th>TB</th>
<th>O</th>
<th>NB</th>
<th>M2</th>
<th>M3</th>
<th>M4</th>
<th>M5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium silicate removal Efficiency (%)</td>
<td>29</td>
<td>29.6</td>
<td>31.7</td>
<td>31</td>
<td>32.4</td>
<td>43.4</td>
<td>42.5</td>
<td>43.2</td>
<td>44.5</td>
</tr>
</tbody>
</table>
On the other hand, the chemical components used, specifically reactive dyes can play a huge role in generating and increasing COD value in batik wastewater by forming active groups. Consequently, in this study, the reactive dyes used affected COD value, making it higher. These phenomena happen due to reactive groups' reaction with sodium silicate and other chemical components in batik wastewater [40,41]. In addition, based on reactive dyes' hydrolysis reaction, they can easily react with water molecules, which increases COD values too. The largest decrease in COD value in simulated wastewater after the pre-treatment cycle runs were recorded for Reactive Black 5, Reactive Blue 15, M2 and M5 with 87, 76, 93, and 107 mg/L, respectively.

Significantly, navy blue and turquoise blue samples represent the largest COD reduction for single dye samples, which can explained by the presence of more leavening groups such as O\text{SO}_3Na, S\text{O}_3Na, and C\text{HCONH}; it can be concluded that there is higher reactivity with other chemical components such as sodium silicate and wax compared to the other single dye samples [43]. Moreover, the highest COD reduction in a mixture of dyes is recorded for (M5) followed by (M2), since they contained higher levels of O\text{SO}_3Na, S\text{O}_3Na, and C\text{HCONH} groups. Furthermore, the total number of charged groups in M5 was higher than in the other mixtures, since it contains all single dyes with the same ratio. On the other hand, due to the molecular size and weight in M2, this is rather higher than in the other mixed samples. Moreover, in comparison with other mixtures, the higher concentration ratio of reactive groups in turquoise blue and yellow reactive dyes in M2 supports the above explanation, although hydrolysis occurs in the separation process because of leavening groups (especially S\text{O}_3Na groups) [44].

3.4. Conductivity value

Each type of wastewater conductivity value is directly related to total amount of suspended and dissolved ion, which is in turn related to different chemical substances with various electrostatic charges, such as species of salts in the aqueous phase and electrical conductivity [3]. Accordingly, there are two main parameters playing tremendous roles in differences in conductivity value and reduction: (1) the chemical components used in batik processing, such as sodium silicate with paraffin's free carbon ions, and (2) the trapping and attachment to wax droplets of existing and generated chemicals during each pre-treatment run. These two parameters directly affect the concentration of electrostatically charged substances, such as ionized salts, in the aqua phase.

The conductivity values for each of the simulated batik wastewater samples after baffle separation pre-treatment cycles are shown in Figs. 6 and 7. The conductivity value decreased in a range of 7–27 μS/cm after baffle separation tank pre-treatment for all simulated wastewater samples. Mainly due to the physical separation of wax particles through baffle pre-treatment, differences of conductivity values do not represent great changes in paraffin wax's electrical charge behavior [45]. Although the highest conductivity reduction value was recorded for navy blue wastewater samples and the lowest for orange and yellow samples together, due to the low difference in all values, only physical and operational parameters in pre-treatment runs play roles in value differences.

However, the differences in conductivity value reduction in mixed dye wastewaters were higher. This is due to the presence of more reactive groups with higher numbers of leavening groups, such as O\text{SO}_3Na, S\text{O}_3Na, and C\text{HCONH}, which led to the generation of
more ions with more electrostatic charge [37,46]. The highest conductivity reduction values in single and mixed dye samples were reported for turquoise blue and M5, with 13 and 17 $\mu$S/cm, respectively.

As mentioned above, this small change in conductivity values is related to the lower amounts of chemical components such as dyes, sodium silicate, sodium carbonate, and the other chemical salts that can be physically attached to coalescence-melted wax, which does not represent an electrical charge, during the pre-treatment process [26,45,47]. This occurs due to wax undergoing a phase changing process, from liquid to solid phase during simulated wastewater pre-treatment. Moreover, the hydrolysis of reactive dye molecules with positive charge groups such as $\text{NaSO}_3^+$ throughout the pre-treatment cycle, and also the attachment of dye molecules onto wax droplets during the removal process, directly affects the decline of the conductivity value [48,49]. Accordingly, reaction between dye molecules and sodium silicate groups in the aqua phase can make efficient changes on the conductive value by, by-product generation. Thus, due to baffle separation pre-treatment, there is a loss of the chemical group with electrostatic charge removal efficiency, so that the decline of conductivity value is not distinctive.

### 3.5. pH value

The pH values for each simulated wastewater sample before and after baffle pre-treatment are measured and are demonstrated in Figs. 8 and 9. The results show the slight decrease in pH value after each treatment cycle run in all treated samples. This phenomenon can be explained by sodium silicate’s reaction with oxygen and carbon molecules, followed by their side-reaction effects on silica and sodium carbonate generation [40,41]. Through physical separation properties, the respective components can trap and attach to wax droplets by supporting $\text{O}_2$ and $\text{CO}_2$ floated bubbles after the phase change process. This is crucial for the removal of generated chemical components from batik wastewater [3].
Consequently, the related reactions during the pre-treatment cycle between CO₂ molecules, which result from paraffin’s carbon atoms and dissolved oxygen with sodium silicate molecules, can generate sediment silica and soluble sodium carbonate (NaCO₃) [40,41]. The removal process of these two components, through alkalinity behavior that involves trapping and attaching to wax droplets and a wax surface layer during the baffle pre-treatment cycle, makes slight changes and causes a decline in pH value in each of the tested samples. In addition, the separation of the wax droplet from sodium silicate and sodium carbonate molecules, based on neutral behavior of wax and alkalinity behavior of sodium salts, can directly affect pH value reduction [50].

Furthermore, a comparison between the results of baffle tank treatment’s effect on single and mixed dye samples reveals that the baffle pre-treatment has more effect on the decline of pH value in less operational time in all samples. Air blowing and oxygen supply during each treatment process by the baffle separation tank play a vital role in both physical and chemical separation and reaction, and show a better efficiency in decrease of pH value. This contributes to changes in values of other respective wastewater parameters, such as COD and conductivity.

### 3.6. Heavy metal removal efficiency

Tables 5 and 6 present results for concentration values of existing heavy metals in influent and effluent samples. The various types of heavy metals in the simulated batik wastewaters are related to the use of reactive dye components, sodium silicate, and tap

<table>
<thead>
<tr>
<th>Table 5</th>
<th>Heavy metal concentration in single dye wastewater samples</th>
</tr>
</thead>
</table>

<table>
<thead>
<tr>
<th>Heavy metal</th>
<th>Y</th>
<th>R</th>
<th>O</th>
<th>NB</th>
<th>TB</th>
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</thead>
<tbody>
<tr>
<td>Cd</td>
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<td>0.0018</td>
<td>0.0001</td>
<td>0.0023</td>
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<tr>
<td>Cr</td>
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<td>0.001</td>
<td>0.0017</td>
</tr>
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<td>As</td>
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<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Cu</td>
<td>0</td>
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<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Fe</td>
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<td>0.2316</td>
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<tr>
<td>Mg</td>
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<tr>
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</tr>
<tr>
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<tr>
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<table>
<thead>
<tr>
<th>Table 6</th>
<th>Heavy metal concentration in mixture dye wastewater samples</th>
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<table>
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<tr>
<th>Heavy metal</th>
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<th>M3</th>
<th>M4</th>
<th>M5</th>
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<td>0.00001</td>
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<tr>
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<td>Cr</td>
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<tr>
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</tr>
<tr>
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<td>0.0426</td>
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<tr>
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<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Pb</td>
<td>0.0322</td>
<td>0.0319</td>
<td>0.0326</td>
<td>0.0309</td>
</tr>
<tr>
<td>Se</td>
<td>0.0641</td>
<td>0.0635</td>
<td>0.0647</td>
<td>0.0638</td>
</tr>
<tr>
<td>Na</td>
<td>162.426</td>
<td>154.619</td>
<td>160.897</td>
<td>153.591</td>
</tr>
</tbody>
</table>
as a result, the concentration of heavy metals present decreased through the pre-treatment process. This phenomenon can be justified by attaching and trapping chemical components with wax droplets and baffle layers during each run [51,52]. In addition, due to sodium silicate and resin components, some heavy metal elements, such as potassium and sodium, can be detected in all simulated wastewater samples [51–53]. Moreover, some other heavy metal elements such as copper can be observed, based on each dye’s chemical structures [37,46]. For instance, the simulated wastewater including Reactive Blue 15 demonstrates the existence of copper elements, which is in agreement with the observation discussed in previous studies [3].

4. Conclusion

The wax removal efficiency of all simulated samples during each pre-treatment cycle was evaluated. The efficiencies in all samples were between 92.5 and 94.9%. The slight differences recorded in wax removal efficiencies were due to the pre-treatment techniques used. The sodium silicate removal rate in single and mixed samples was in the range of 32–44.5% and is based on wax removal and the various types of dye in each sample. The permeate pH in every simulated sample decreased slightly due to the effect of sodium silicate with an alkaline behavior. The COD in both single and mixed dye samples slightly decreased, which is a result of wax’s physical components, sodium silicate, and the separation of the dyes’ chemical components by baffle separation tank pre-treatment. Moreover, the conductivity and existing heavy metals concentration in all the samples declined slightly during the pre-treatment.

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