Wastewater treatment using flocculation method and water reuse for dyeing of polyester fibers

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Abstract. Textile industry uses and pollutes large quantities of water, especially for chemical treatments of textile materials, like bleaching, desizing, dyeing, printing and finishing. After each process, the water is usually drained into the sewage system and fresh water is used for new process of textile treatment. Re-using of wastewater for further processing would be of great importance in term of saving fresh water and diminishing the environmental burden. The goal of our research was to find out whether treatment of dye-house wastewater with flocculation could be efficient enough for water re-use in further dyeing of polyester fabric. Wastewater collected in Slovenian dye-house was treated with a cationic flocculant. Treated water was analysed for chemical oxygen demand (COD), total organic carbon (TOC), spectral absorption coefficient (SAC) and dry matter and further used for laboratory dyeing of polyester fabric. The fabric was dyed comparatively using technological and treated water under the same conditions in light, medium and dark shade. The colour of dyed samples was evaluated on spectrophotometrically. Wet fastness and colour fastness to perspiration (acid and alkaline) of differently dyed samples were investigated. Change of colour was acceptable for all dyed samples. Fastness to washing and perspiration were good, very similar when comparing samples dyed in technological and those dyed in cleaned water. The colorimetric and fastness results showed a high efficiency of flocculation for dye-house wastewater treatment and reuse of treated water in production.

1. Introduction

Textile industry uses and pollutes large quantities of water, especially for chemical treatments of textile materials, like bleaching, desizing, dyeing, printing and finishing. A large problem of wastewater represents the dyes, which even in small quantities are already very visible and disturbing for the environment. During dyeing most of the dyes are exhausted from the dye-bath and bound to the fibers, but a part of unbounded dye always remains in the dye-bath and consequently finishes in the effluent. After each process the water is usually drained into sewage systems and fresh water is used for new process of textile treatment. Re-using of wastewater for further processing would be of great importance in term of saving fresh water and diminishing of environmental burden. The composition of dye-house wastewater is very complex and varies daily because of different dyes and additives which are used in the dyeing processes [1]. Most dyes have a complex aromatic structure, consistent to light, bio decomposition, oxygen and other environmental influences. That is why it is difficult to remove them.
from water with classical treatment methods [2]. It is also impossible to find a process that would be suitable for all wastewaters [3].

Flocculation is a simple and economical process to remove the dyes from wastewater [4,5]. The goal of our research was to find out whether treatment of dye-house wastewater with flocculation could be efficient enough for water re-use in further dyeing of polyester fabric.

Wastewater collected in Slovenian dye-house was treated with cationic flocculants. Treated water was analysed for chemical oxygen demand (COD), total organic carbon (TOC), spectral absorption coefficient (SAC) and dry matter and further used for laboratory dyeing of polyester fabric. The fabric was dyed comparatively using technological and treated water under the same conditions in light, medium and dark shade. The colour of dyed samples was evaluated on spectrophotometer Datacolor Spectraflash SF-600X. Wet fastness and colour fastness to perspiration (acid and alkaline) of differently dyed samples were investigated.

2. Experimental

2.1. Flocculation

The representative wastewater from the dye-house was treated using different flocculants; Flocculant CHT-CV (Bezema, Switzerland), Flocculant CHT-VEL (Bezema, Switzerland), Rucofloc (Rudolf Group, Germany) and Sevofloc (Textilcolor AG, Switzerland). From 0,2 to 2,0 g/l flocculant at pH 6, 7, 8 and 9 was added to the water samples. pH was adjusted with Na$_2$CO$_3$ or CH$_3$COOH. The laboratory flocculator Velp Scientifica FP4 was used. The solutions were mixed for 2 min. at 100 o/min following 20 min at 20 o/min. After 120 min of sedimentation the best results were found at 0,3 g/l of Flocculant CHT-CV at pH 8, at 0,5 g/l of Flocculant CHT-VEL at pH 7, at 0,7 g/l of Rucofloc at pH 8 and at 0,7 g/l of Sevofloc at pH 8.

2.2. Dyeing

The treated water was used for dyeing of 100 % polyester yarn (PES) with the fineness of 25 tex in light (L), medium (M) and dark (D) shade. Before dyeing the yarn was washed at 70 °C for 30 min with 1 g/l Foryl OV (Pulcra). After dyeing the yarn was reductive cleaned with 1% Rucorit RAL (Rudolf Group GmbH) and 2 g/l NaOH 50% for 20 min at 85 °C. The same material was also dyed with regular technological water at the same conditions. The dyeing was done in a laboratory dyeing machine Ahiba Nuance ECO at 130 °C for 60 min at bath to liquor ratio of 1:10.

Dyes and additives: Foron dyes (Archroma, Switzerland), Alwiron PWE (Textilcolor, Germany); (dispersive agent), Tanacid SAB (Tanatex, Germany); (buffer).

| Dye | Concentration (%) | a) | b) | Chemicals and additives | Concentration (gl$^{-1}$) |
|-----|-------------------|---|---|--------------------------|------------------------|
|     |                   | L$^a$ | M$^a$ | D$^a$ | CH$_3$COOH 80% (pH 4,5) | L$^a$ | M$^a$ | D$^a$ |
| Foron brill.gelb SWF | 0 | 0 | 0,1500 | 1,10 | 1,10 | 1,10 |
| Foron goldgelb SWF | 0,0040 | 0,2480 | 0,5000 | 1,50 | 1,50 | 1,50 |
| Foron rot SWF | 0,0001 | 0,0003 | 0 | 0,75 | 0,75 | 0,75 |
| Foron blau SWF | 0,0002 | 0,0030 | 0 | 0,75 | 0,75 | 0,75 |
| Foron marineblau SWF | 0 | 0 | 0,5000 | 0,75 | 0,75 | 0,75 |

$^a$ Color tone

2.3. Analysis

Chemical oxygen demand (COD) - SIST EN ISO 6060:1996
Total organic carbon (TOC) - SIST EN ISO 8245:2000
Spectral absorption coefficient (SAC) - SIST EN ISO 7887:1996
Colour measurement – spectrophotometer Spectraflash SF-600X (Datacolor).
Colour fastness to washing - SIST EN ISO 105-C06:1999.
Colour fastness to perspiration - SIST EN ISO 105-E04:1999

3. Results and discussion

Table 2 represents the environmental parameters of representative sample of wastewater and water cleaned with different flocculants.

| Flocculant       | Dry matter (%) | COD (mgO₂/l) | TOC (ppm) | IC (ppm) | SAC (m⁻¹) |
|------------------|----------------|--------------|-----------|----------|------------|
|                  |                |              |           |          | 436 nm     | 525 nm     | 620 nm     |
| Untreated        | 0.23±0,01      | 680±10       | 615±10    | 92±1     | 420,0      | 219,0      | 190,0      |
| CHT- CV          | 0.25±0,01      | 580±10       | 597±10    | 113±1    | 1,6        | 0,6        | 0,5        |
| CHT- VEL         | 0.26±0,01      | 650±10       | 770±10    | 77±1     | 2,4        | 1,2        | 1,0        |
| RUCOFLOC         | 0.25±0,01      | 630±10       | 671±10    | 110±1    | 10,6       | 5,3        | 2,8        |
| SEVOFLOC         | 0.28±0,01      | 640±10       | 784±10    | 112±1    | 3,7        | 2,1        | 1,7        |

It can be seen that all flocculants thoroughly decreased the SAC values, while other parameters; dry matter, COD, TOC and IC, remained high or even increased. COD value of representative sample is 680 mg O₂/l. The best results of treatment were obtained for Flocculant CHT-CV, which decreased it for 15% to a value of 580 mg O₂/l. Even less has changed the TOC value, from 615 ppm for untreated water to 597 ppm for water treated with Flocculant CHT-CV. The content of dry matter even increased during treatment from 0.23% to 0.25%. On the other hand, very good results were obtained for SAC values, where the reduction in light absorption was almost 100% and the colour of the water was completely disappeared. This means that the flocculation removed almost all dyes from the wastewater although other components remained there in a considerable amount.

Polyester yarn was dyed in a water treated with Flocculant CHT-CV. Table 3 represents the CIE L*, a*, b* values for samples dyed with technological and treated water and colour differences between them.

| Water      | Colour tone | L*    | a*    | b*    | ΔEₘ₂₄* |
|------------|-------------|-------|-------|-------|--------|
| Technological | L           | 82.85 | 2.59  | 14.00 | /      |
|             | M           | 55.48 | 9.37  | 21.87 | /      |
|             | D           | 24.77 | 6.53  | 3.48  | /      |
| Treated    | L           | 83.36 | 3.33  | 14.05 | 0.90   |
|             | M           | 55.30 | 9.70  | 22.13 | 0.45   |
|             | D           | 25.01 | 6.57  | 3.47  | 0.24   |

The highest colour difference of ΔEₘ₂₄* = 0.9 was obtained for light shade. It is low enough to be accepted for further production. ΔEₘ₂₄* for medium shade is 0.45 and for the dark shade 0.24. Both values are very low and show that the material can be dyed with treated wastewater.

Table 4 represents the colourfastness’s values to wash and acid and alkaline perspiration of polyester samples dyed with technological and treated water.
Table 4: Colourfastness to wash and acid and alkaline perspiration of polyester.

| Water    | Colour tone | Evaluation |
|----------|-------------|------------|
|          |             | Acid perspiration | Alkaline perspiration | Washing |
| Technological | L  | 5 | 5 | 5 |
|            | M  | 5 | 4-5 | 5 |
|            | D  | 4-5 | 4 | 5 |
| Treated   | L  | 4-5 | 4-5 | 5 |
|            | M  | 5 | 4-5 | 5 |
|            | D  | 4 | 4 | 5 |

The fastness to washing are the same on polyester yarn dyed with treated or with regular water, excellent in both cases. Very good are also the fastness properties to acid and alkaline perspiration at both dyeing conditions.

4. Conclusions
In the first part of the investigation, it was found that the pollution of the wastewater from a dye-house oscillates during the day and during the week. The average pollution is high, the TOC values are above 900 ppm and the coloration is high. To obtain the average polluted wastewater, the samples of the water were taken from the collection pool once a day for one week and consequently all seven samples were mixed together. In this way the representative wastewater was obtained.

The wastewater from the textile dye-house was treated with 0.3 g/l CHT Flocculant-CV at pH 8. The SAC decreased for 99%, COD for 15%, whereas the amount of dry matter increased during the flocculation process.

The treated water was used for dyeing of polyester yarn in light, medium and dark shade. For comparison the polyester yarn was dyed in the same shades by using fresh process water. The dyeing results were appropriate for all three shades when using the treated wastewater and when using fresh process water. Change of colour was acceptable for all dyed samples. The lowest colour difference was obtained at dark shade, the highest at light shade. Fastness to washing and perspiration were good, very similar when comparing samples dyed in technological and those dyed in cleaned water. The colorimetric and fastness results showed high efficiency of flocculation for dye-house wastewater treatment and a possibility of reusing of treated water in further production.

Flocculation has proven to be a process, which successfully removes mainly dyes from the wastewater, while a part of other components remained there together with a part of flocculant. Despite high COD and TOC values the treated water is appropriate for preparing new dyebaths, especially for dyeing of synthetic polyester fibers in dark tones. Polyester is namely very hydrophobic, without functional groups, absorbs only few chemicals and is hard to dye. This is a reason why the flocculants and other chemicals that remained in treated water do not enter into the fibers during dyeing but remain in the dye-bath while small dispersive dyes enter into the fibers at appropriate conditions and successfully dye them.

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