

Article

Brine Recycling from Industrial Textile Wastewater Treated by Ozone. By-Products Accumulation. Part 2: Scaling-Up

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Abstract: Extremely high volumes of salty wastewater are produced by textile manufacturers daily. Therefore, brine recycling from the wastewater should be regarded as a crucial issue within the textile industry. Ozonation was used in this two-part study as a purification method for industrial textile wastewater polluted by low-molecular-weight salts (LMWS). Part 1 revealed the accumulation of ozonation by-products in a multi-recycling system. The objective of Part 2 was the scaling-up of the process and the investigation of the occurrence of by-products. It was found that ozonation works well in an alkaline reaction medium, which was characteristic of the wastewater from a dye house; an almost complete color removal was achieved within 30 min of treatment. The brine that was produced from the wastewater treated by ozonation in a 20 L bubble column reactor was recycled successfully. Dyeing of cotton with five types of reactive dyes in various shades resulted in very good values of DE_{CMC} , which is the normative color matching parameter, and were between 0.15 and 1.2. The color fastness obtained for upcycled fabrics were satisfactory, and not worse than standard values. Although accumulation of the side products was detected in Part 1, the fabric discharges produced in the scaled-up process were free from carcinogenic amines and heavy metals. The study indicated that ozonation can be applied in the industry as a method for textile wastewater recycling.

Keywords: industrial textile wastewater; wastewater recycling; laboratory and scaled-up ozonation; by-products of ozonation

1. Introduction

Numerous literature reports have revealed great pollution of textile wastewater by low-molecular-weight salts (LMWS) [1–6]. Part 1 of this study pointed out that extremely large consumption of NaCl or Na₂SO₄ is characteristic in textile manufacturing, because these salts are common textile auxiliary agents during dyeing operations. The problem of salty wastewater emission seems to be severe in industrial areas of massive textile production. Bhatia and co-workers [7] indicate that the total dissolved solids (TDS) concentration measured in a local river (Punjab, India) even reached 3000 mg/L. The pollution revealed by Bhatia and co-workers [7] was caused by direct emission of textile wastewater into surface water. However, it should be kept in mind that the danger of an increase in the salinity of water bodies is real, even for industrial districts with regulated environmental policy. The areas of extensive textile production in Europe, such as the Prado region (Italy) and the Lodz region (Poland), are truly exposed to water body salinity. Even though the environmental

regulations are inflated there, and textile wastewater is directed to municipal wastewater plants, these plants have not been designed to eliminate LMWS. Therefore, the idea of keeping LMWS in a closed loop by means of an on-site recycling system seems to be a solution worth testing out.

Part 1 of this study indicated that textile wastewater after dyeing operations (dyeing of cotton fabric with reactive dyes) can be successfully used as a source of “ready-to-use brine” without diluting it. However, keeping the same water in a closed loop for a few cycles resulted in accumulation of by-products. Accumulation of intermediates observed after the second recirculation of process water disturbed the dyeing process, but only to a small extent. The greatest issue of the study is how the ozonation side products can affect the final textile outfit which contacts human skin.

The objectives of Part 2 were, firstly, to investigate the possibility of ozone system industrial implementation, realized in a bubble column reactor, and secondly, to check the quality of textiles dyed with recycled process water. Both goals were extremely important from the industrial point of view. However, the eco-human-testing of textiles properties, which gives information on how textiles affect human health, was crucial. Therefore, the Textile Research Institute (Poland), assigned for Oeko-tex™ labeling, was employed for this part of the investigation.

2. Experimental

2.1. Materials

Ozone treatment. The experiments were carried out for the dye solution and the wastewater from the dyeing operation.

The dye solution was based on Reactive Black 5 (RB5) dye. It was obtained from Boruta-Zachem (Poland) as a purified reagent, molecular weight 991 g/mol, λ_{\max} 569 nm, di-azo type. The buffer components were NaOH A.R. purchased from Stanlab (Poland) and H_3PO_4 , KH_2PO_4 , Na_2HPO_4 all were A.R grade and purchased from Chempur (Poland). Na_2SO_3 A.R was purchased from Chempur (Poland).

The substances present in industrial textile wastewater were: Setazol Black DPT (industrial product based on RB5, Setas-Kiyima (Turkey)) and industrial dyeing assistant—Perigen LDR (SAA—naphthalenesulfonic acid and carboxylates mixture, Textilchemie Dr. Petry Co. (Germany)), as well as NaCl, NaOH, and Na_2CO_3 (technical products). The exact compositions of the dye bath (given by the textile company) and the industrial wastewater indicators (wastewater after the specific dyeing process) are given in Table 1.

Table 1. Dyeing bath composition A), dyeing process conditions B), industrial wastewater characteristics C).

| Wastewater Characteristics | | | | | |
|------------------------------------|----------|------------------------------|--|--------------------------|--------------------------|
| A) Dyeing Bath Content | | B) Dyeing Process Conditions | | C) Wastewater Indicators | |
| Setazol Black DPT (industrial RB5) | 5.36 g/L | Batch type | 85%Viscose 10%Polyester 5%Elastane | pH | 11.23 |
| Perigen LDR (SAA) | 0.76 g/L | Weight | 200 kg | conductivity | 78.41 mS/cm |
| NaCl | 65 g/L | Dyeing ratio | 1:14 | NaCl conc. | 45 g/L |
| Na_2CO_3 | 1.0 g/L | Bath volume | 2800 L | COD | 1715 mgO ₂ /L |
| NaOH 50% (a.s.) | 1.95 g/L | Machine type | Thies TRD | Dye conc. | 470 mg/L |

Textile dyeing. In the wastewater recycling experiment (cotton dyeing) industrial dyes were used. Synozol Yellow KHL (Reactive Yellow145), Synozol Red K3BS_{150%} (Reactive Red 195), Synozol Blue KBR (Reactive Blue 221) were purchased from KISCO (Turkey). Bezaktiv Turquoise VG_{150%} was obtained from Bezema GmbH (Germany). Setazol Black DPT was purchased from Setas-Kiyima (Turkey). The dyeing assistants were the same as listed above for the wastewater. A raw knitted fabric, made from 100% cotton, was obtained from Sontex (Denmark).

2.2. Experimental Procedure

Ozonation. A laboratory scale ozonation of the industrial textile wastewater and dye solutions was performed in a semibatch glass-stirred tank reactor with a capacity of 1 L (described in Part 1).

Scaled-up ozonation was performed in a glass bubble column with a capacity of 20 L (shown in Figure 1). Ozone was fed into the reactor from the bottom of the reactor (1) with a ceramic diffuser brandol 60®(3). Ozone was produced by TOGC8X TROGEN LTD generator (2), which was equipped with an integral compressor and oxygen concentrator. The ozone concentration in the gas phase at inlet and outlet of the reactor was measured by an ozone analyzer BMT 964 (4), manufactured by BMT MESSTECHNIK GMBH (Germany). Circulation of the liquid phase was forced by a peristaltic pump (5). Samples for analysis were collected using a cell (6), and the ozone concentration in the liquid phase was measured. The gas effluents from the reactor passed through a scrubber filled with silica gel with an indicator (7) in order to remove moisture contained in the gas, and then directed to an ozone destructor (8).

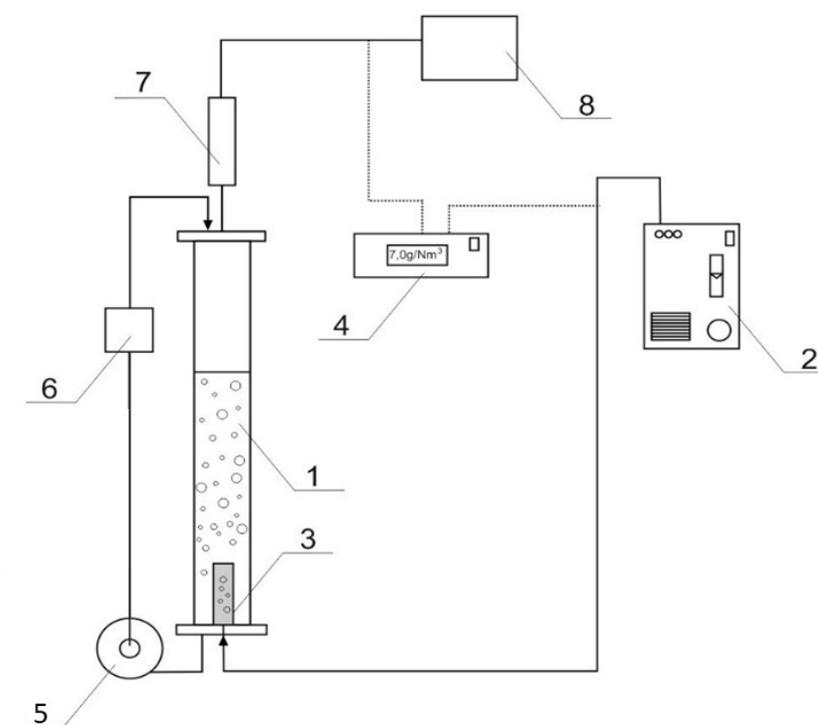


Figure 1. Experimental set-up for ozonation process: 1—bubble column; 2—ozonator with oxygen concentrator; 3—gas diffuser; 4—gasous ozone analyzer; 5—peristaltic pump; 6—cell for samples collecting; 7—scrubber filled with silica gel; 8—ozone destructor.

In both cases, the laboratory and the intermediate scale, the reaction progress was stopped by the addition of 0.01 M Na_2SO_3 to the samples.

Textile dyeing. The dyeing of the cotton was carried out in a LABOMAT BFA-12 system (laboratory dyeing machine made by Mathis AG, Switzerland) according to a standard exhaustion-fixation procedure in temperature of 60 °C (presented in Part 1). The weight of each sample was 10 g, and the liquor ratio was set at 1:12. The alkalis, which were NaOH and Na_2CO_3 aqueous solutions, were dosed partially to avoid excessive dye hydrolyzation. The electrolyte (NaCl) was dosed only into freshwater dyeing, not recycling ones.

2.3. Analytical Methods

The color of the samples collected at specified time intervals were measured by a spectrophotometer (Helios Thermo). A calibration plot based on Lambert-Beer's law was used to determine the concentration of the samples.

Chemical oxygen demand (COD) was obtained using the standard method with a HACH-LANGE apparatus (DR 3800) by the dichromate (VI)-LCK 514 and 314 tests.

The colors of the textile samples were measured using DataColor 400 reflection spectrophotometer in accordance with ISO 105-J03 [8]. Each measured DE_{CMC} (DE of Color Measurement Committee) value is an average of at least three measurements (measured up to a maximum error value—standard deviation from the average value set at 0.1)

The carcinogenic amines (in accordance to EN 14362-1:2012) and heavy metal (in accordance to PB/8/2005 internal methodology) detection were carried out by the Textile Research Institute (Poland) assigned by Oeko-tex® Organization for eco-label research and certification.

3. Results and Discussion

3.1. Ozonation

Ozonation was conducted at a laboratory scale (LS), as well as at an intermediate bench scale (IBS). Both experiments were run up to the moment at which the absorbed ozone dose was close to 0.7 g/L. This ozone dose was specific to obtain satisfactory wastewater discoloration (99% of color removal).

The LS experiment was run in a volume of 1 L, while the IBS was run in 10 L. The RB5 was decomposed in an aqueous solution and industrial wastewater, respectively. In Figure 2, it can be observed that the results obtained for LS and IBS are not fully comparable. However, they complement each other: LS gave sharper information about the process in the initial phase, and the IBS experiment made it possible to confirm the general trends.

Figure 2A presents the color removal during ozonation. Based on the LS data, it can be noticed that the process was divided into two specific steps. In the first, very short, one (about 3 minutes), the experimental points of the RB5 solution and the industrial wastewater were almost identical to each other, even though the wastewater was laden by much higher COD caused by its complex matrix. This means that decolorization of RB5 was nearly the same in the aqueous solution and the industrial wastewater. In the second phase, the color removal rate of the RB5 aqueous solution was much higher than the wastewater. The phenomenon is noticeable when absorbed ozone dose is concerned, as well (Figure 2B). This observation confirms the argument that decolorization is the first stage of dye decomposition via ozonation, as postulated in our previous work [9] and by other authors [10,11]. It is highly probable that color is removed first, due to chromophore oxidation, and then further oxidation of by-products or other components of the reaction mixture takes place. Moreover, it could be noticed that for the IBS the experimental points of the RB5 solution and the wastewater were much closer to each other than for LS, which suggests the more similar decolorization progress, probably due to longer contact time and much more effective ozone absorption. The ozonation of the wastewater was more efficient in the IBS than in LS.

Where COD removal was concerned (Figure 2C) it can be noticed that decolorization does not correspond to the total decomposition of RB5. Even though the color was removed (after applying 0.7 g/L of ozone) the 70% of COD remained. This suggests that a high concentration of colorless by-products might have occurred in the post-reaction mixture, as postulated in Part 1.

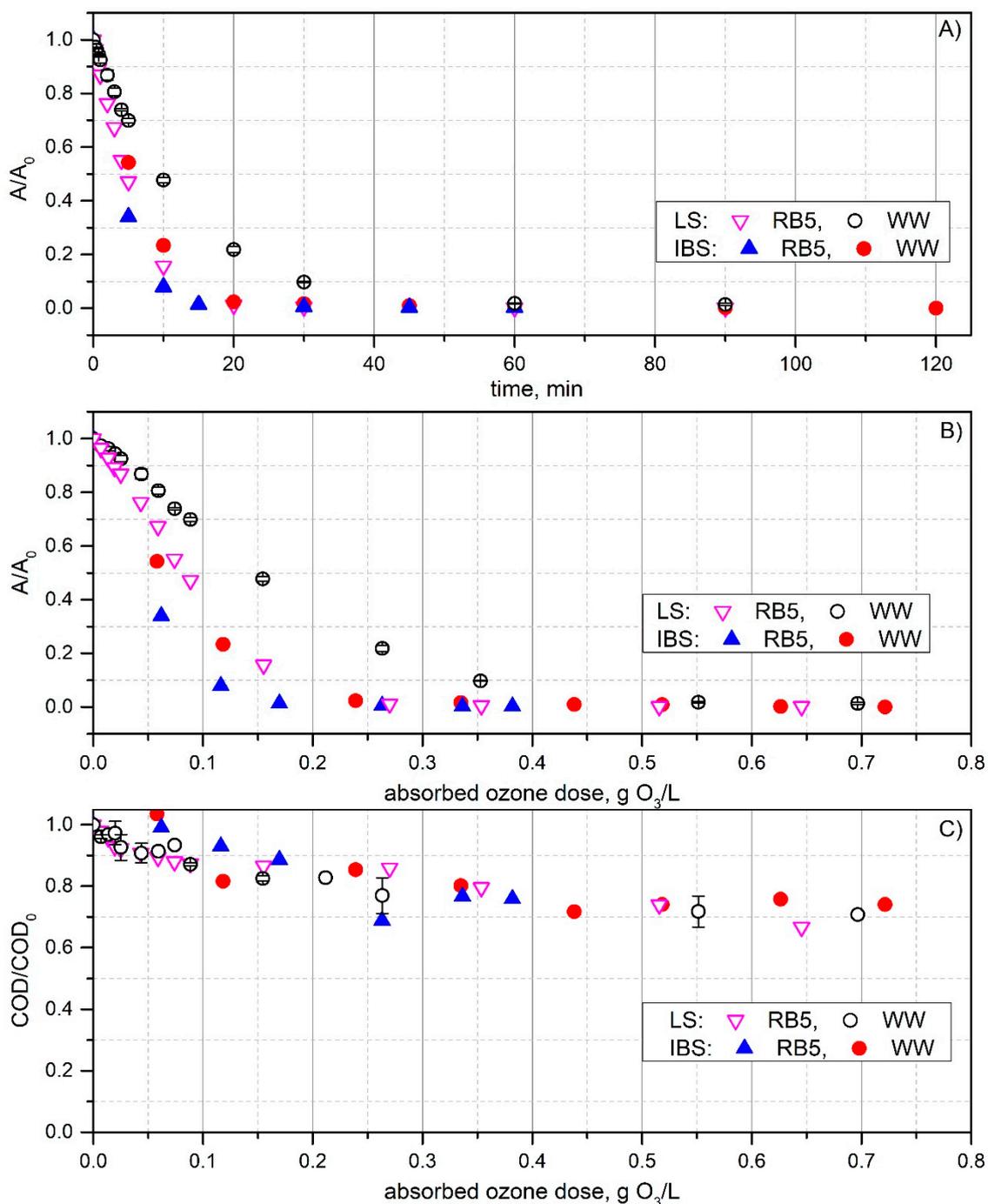


Figure 2. Ozonation LS (Q 0.66 L/min, C_{O_3} 42.3 mg/L) and IBS (Q 5 L/min, C_{O_3} 25 mg/L): (A) color removal (A/A_0) vs. time, (B) color removal (A/A_0) vs. absorbed ozone dose $\text{mg O}_3/\text{L}$, (C) COD removal (COD/COD_0) vs. absorbed ozone dose $\text{mg O}_3/\text{L}$; symbols: RB5—an aqueous solution of RB5 dye, WW—industrial wastewater after dyeing, which contains RB5 dye.

To keep similar ozonation conditions in terms of absorbed ozone dose in specific time, two ozone concentrations were used: C_{O_3} 42 mg/L for LS and C_{O_3} 25 mg/L for IBS. In Figure 3, the ozone concentrations in the gas phase on the inlet and outlet of the reactor were presented. Figure 3A shows data collected for LS. When the feed of the reactor was set at C_{O_3} 42 mg/L, the ozone concentration on the reactor outlet relatively quickly achieved a value close to the inlet value. Moreover, the experimental points of RB5 solution and the wastewater were almost identical. The data obtained for the IBS were much more informative (Figure 3B). It can be noticed that up to 40 minutes of the process,

the outlet ozone concentration for the wastewater was much lower than for the RB5 solution. Therefore, it can be concluded that a much higher ozone concentration was needed to oxidize substances in the wastewater compared to the dye solution. The construction of the IBS reactor enabled efficient wastewater purification due to there being sufficient interphase-contact time (ICT equal to 2 min, based on Equation (1)) and a fine bubble distribution system.

$$ICT = \frac{V_{liquid\ phase}}{Q_{gas\ phase}},\ min \quad (1)$$

where: ICT —theoretical interphase-contact time, min; $V_{liquid\ phase}$ —volume of the wastewater sample, L; $Q_{gas\ phase}$ —volumetric flow rate of the oxygen-ozone mixture, L/min

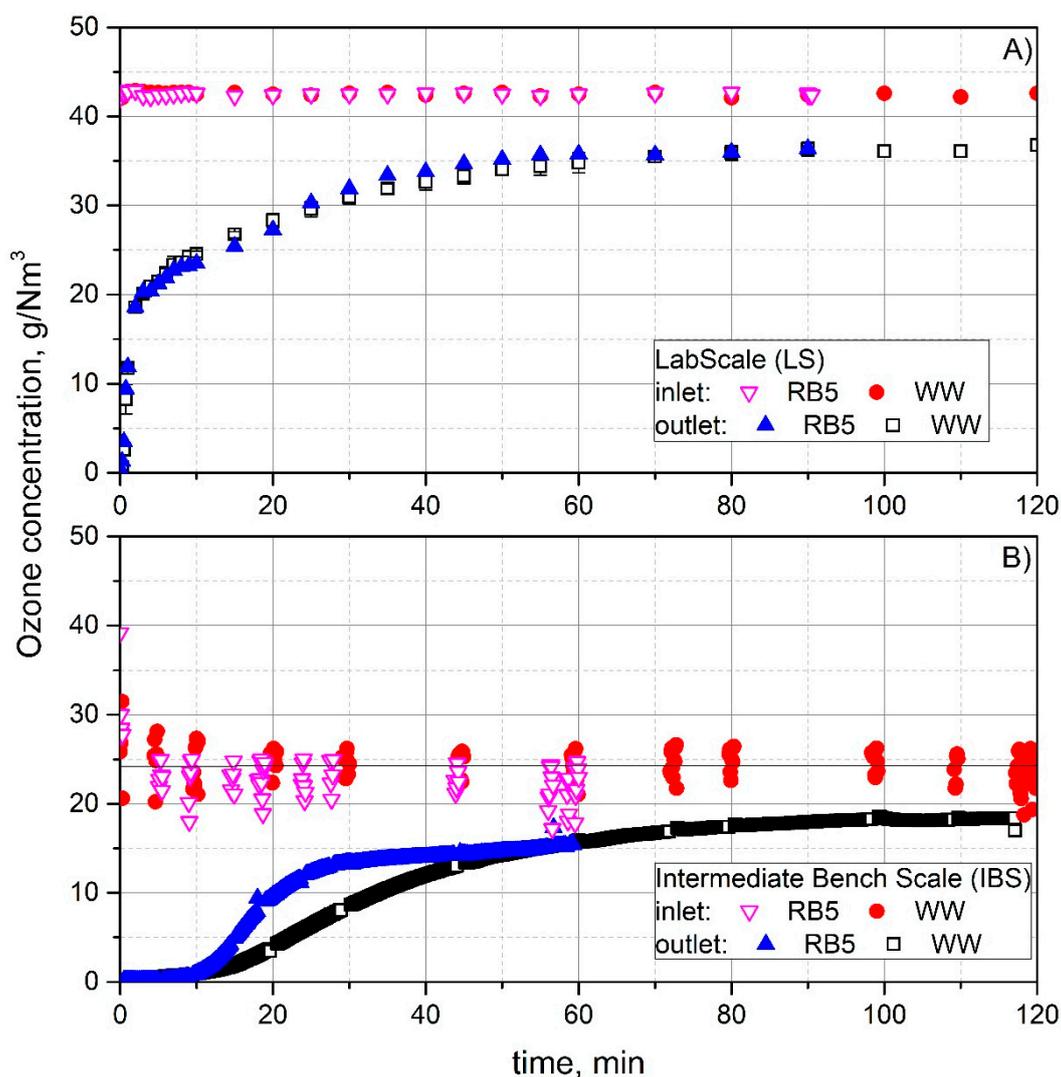


Figure 3. Ozone concentration in the inlet and outlet gases: (A) LS, Q 0.66 L/min, C_{O_3} 42.3 mg/L; (B) IBS, Q 5 L/min, C_{O_3} 25 mg/L; symbols: RB5—an aqueous solution of RB5 dye, WW—industrial wastewater after dyeing, which contains RB5 dye.

Based on this observation, it can be concluded that, in contrast to the IBS during the LS experiment, the ozone absorption into a liquid phase was very poor (even though the ICT of LS was 1.51 min).

3.2. Re-Dyeing

The ozonation experiments (LS and IBS) led to the production of ready to use brine from the wastewater. The initial salt concentration in this brine was equal 45 g/L. Following ozone treatment, reactive cotton dyeing in various shades was carried out using the purified wastewater without diluting it. The DE_{CMC} indicators, measured spectrophotometrically in accordance with ISO 105-J03 [8], which are color-matching parameters, are set out in Table 2. The DE_{CMC} values were between 0.15 and 1.2, with a limiting value of 1.5, for all the samples (I recycling). This means that there was no visible difference in shade between the standard fabric (a fabric dyed with fresh water) and the recycled fabric (a fabric dyed with purified wastewater-brine), even to the ‘experienced’ observer.

Table 2. DE_{CMC} values in accordance with ISO 105-J03.

| Re-Dyeing Type of Dye | Depth of Shade (%) | DE_{CMC} When Specified Ozone Dose Was Used | | |
|----------------------------|-----------------------|---|--------------|----------------|
| | | 0.55 g/L (LS) | 0.7 g/L (LS) | 0.72 g/L (IBS) |
| Synozol Yellow KHL | 1 | 0.48 | 0.68 | 0.97 |
| | 2 | 0.56 | 0.8 | 0.99 |
| Synozol Red K3BS150% | 1 | 1.09 | 0.89 | 0.66 |
| | 2 | 1.20 | 1.05 | 0.87 |
| Synozol Blue KBR | 1 | 0.65 | 0.21 | 0.49 |
| | 2 | 0.15 | 0.51 | 0.63 |
| Bezaktiv Turquoise VG 150% | 1 | 1.07 | - | 0.34 |
| | 2 | 0.8 | - | 0.61 |
| Setazol Black DPT | 6 | 0.86 | 0.56 | - |
| | 8 | 1.16 | 0.89 | 0.64 |

Afterward, the fabric discharges were examined in terms of their potential harm to human health. Even though the production standards of modern colorants (dyes and pigments) are rigorous and the use of harmful components is forbidden, the standard examination of textiles includes eco-testing [12]. Therefore, the fabric discharges were examined with regard to the occurrence of carcinogenic amines and heavy metals (determined by GC-MS and ICP, respectively). While the detection level was 20 mg/kg of textile material, the analysis showed that all the samples were free from the substances listed in Table 3. These substances are stipulated by EU legislation [12], and they are covered by standard tests on the quality of textiles, especially where its recycling is concerned.

Table 3. List of harmful aromatic amines, according to [12], examined by GC-MS.

| Number | Amine Type | CAS Number | Number | Amine Type | CAS Number |
|--------|------------------------------|------------|--------|---------------------------------------|------------|
| 1 | 4-aminodiphenyl | 92-67-1 | 12 | 3,3'-dimethylbenzidine | 119-93-7 |
| 2 | benzidine | 92-87-5 | 13 | 4,4'-methylenedi-o-toluidine | 838-88-0 |
| 3 | 4-chloro-o-toluidine | 95-69-2 | 14 | p-cresidine | 120-71-8 |
| 4 | 2-naphthylamine | 91-59-8 | 15 | 4,4'-methylene-bis-(2-chloro-aniline) | 101-14-4 |
| 5 | o-aminoazotoluene | 97-56-3 | 16 | 4,4'-oxydianiline | 101-80-4 |
| 6 | 5-nitro-o-toluidine | 99-55-8 | 17 | 4,4'-thiodianiline | 139-65-1 |
| 7 | 4-chloroaniline | 106-47-8 | 18 | o-toluidine | 95-53-4 |
| 8 | 4-methoxy-m-phenylenediamine | 615-05-4 | 19 | 4-methyl-m-phenylenediamine | 95-80-7 |
| 9 | 4,4'-diaminodiphenylmethane | 101-77-9 | 20 | 2,4,5-trimethylaniline | 137-17-7 |
| 10 | 3,3'-dichlorobenzidine | 91-94-1 | 21 | o-anisidine | 90-04-0 |
| 11 | 3,3'-dimethoxybenzidine | 119-90-4 | 22 | 4-amino azobenzene | 60-09-3 |

The values of heavy metal concentrations presented in Table 4 show that the recycled fabrics were free from cadmium, cobalt and nickel; however, except the yellow textile sample, all were characterized by a low copper content. In the case of textiles dyed with Synozol Blue KBR and Bezaktiv Turquoise VG_{150%}, it can be explained by the dye structure (copper is a part of the chromophore). However, for samples dyed with Synozol Red K3BS_{150%} and Setazol Black DPT the recycling residuals could be

a cause of copper content. Therefore, copper residual should be monitored when textile wastewater recycling is concerned; however, its concentration is low, close to the detection level.

Table 4. Heavy metal concentrations in fabrics (DL—detection level; UDL—under detection level).

| Re-Dyeing Type of Dye | Cadmium (Cd) mg/kg | | Cobalt (Co) mg/kg | | Copper (Cu) mg/kg | | Nickel (Ni) mg/kg | |
|--|-----------------------|------|----------------------|------|----------------------|-------|----------------------|------|
| | value | DL | value | DL | value | DL | value | DL |
| Synozol Yellow KHL | UDL | | UDL | | UDL | | UDL | |
| Synozol Red K3BS _{150%} | UDL | | UDL | | 0.84±0.026 | | UDL | |
| Synozol Blue KBR | UDL | 0.20 | UDL | 6.85 | 11.07±0.34 | 0.707 | UDL | 7.05 |
| Bezaktiv Turquoise VG _{150%} | UDL | | UDL | | 1.30±0.04 | | UDL | |
| Setazol Black DPT | UDL | | UDL | | 1.01±0.031 | | UDL | |

The color fastnesses against laundering, sweat, and rubbing were satisfactory, and were no worse than those obtained for the standard dyeing procedure with fresh water (Table 5); measurements were carried out in accordance to ISO 105-C06, ISO 105-E04, and ISO 105-X12, respectively [13–15].

Table 5. The color fastnesses against washing, sweat and rubbing in accordance with ISO 105-C06, ISO 105-E04, and ISO 105-X12, respectively (5—the best, 1—the worst).

| Type of Sample | Depth of Shade (% w/w) | Fastness Against | | | | | | | |
|--------------------------|---------------------------|------------------|-----------|----------------|-----------|--------------|-----------|-------------|-----------|
| | | Washing 40 °C | | Sweat Alkaline | | Sweat Acidic | | Rubbing Dry | |
| | | Standard | Recycling | Standard | Recycling | Standard | Recycling | Standard | Recycling |
| Synozol Yellow KHL | 1 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| C.I. Reactive Yellow 145 | 2 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| Synozol Red K3-BS | 1 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
| C.I. Reactive Red 195 | 2 | 4/5 | 4 | 4/5 | 4/5 | 4 | 4 | 4 | 4 |
| Synozol Blue KBR | 1 | 4/5 | 4/5 | 5 | 5 | 5 | 4/5 | 4/5 | 4/5 |
| C.I. Reactive Blue 221 | 2 | 4/5 | 4 | 5 | 5 | 5 | 4/5 | 4/5 | 4 |
| Setazol Black DPT | 6 | 5 | 5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
| C.I. Reactive Black 5 | 8 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 3 | 4 |

4. Conclusions

The findings of this study indicate that ozonation can be applied in the textile industry as a highly effective method for recycling of salty wastewater. High color removal was obtained for both laboratory (LS) and intermediate bench (IBS) scale ozonation.

Even though the ozone transfer from the gas phase into the liquid phase was far less effective in the case of LS compared to IBS experiment, the same main principles of dye ozonation could be observed in LS and IBS. An ozone dose of 0.7 g/L was found to be sufficient for complete decolorization of wastewater; however, the COD removal was not as satisfying as the color removal. 70% of COD remained after the process what confirms a high concentration of colorless by-products. Despite low COD removal, the industrial wastewater treated in both LS and IBS systems was suitable for recycling.

Excellent color parameters of fabrics dyed with recycled process water were achieved when a single recycling loop was performed (Part 1 and Part 2 of the study) and slightly satisfactory when the multi-recycling loop was carried out (Part 1). The multi-recycling-loop experiment indicated accumulation of the oxidation by-products which were recognized as naphthol and phenol derivatives by TLC method (Part 1). However, the by-products occurrence disturbed color removal by ozone treatment in the subsequent recycling loops and slightly affected the dyeing process in Part 1, the fabrics dyed with recycled process water in Part 2 were free from carcinogenic amines and heavy metals, and only a very low copper content, which was correlated with the dyes' structure, was detected.

This result cannot give the final health-safety assurance of up-cycled textiles, but it encourages for further investigations in this area, especially where industrial implementation is possible.

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