

Simultaneous afterclearing and decolorisation by ozonation after disperse dyeing of polyester

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A set of trials have been conducted to examine the efficiency of ozonation on afterclearing of disperse dyed poly(ethylene terephthalate) fibres. Ozonation was performed in the cooled dyebath after the completion of the dyeing cycle. The ozone concentration used was 12.8 ± 0.3 mg/min at a gas flow rate of 400 ml/min. The results indicate that 3 min ozonation time is appropriate to achieve wash fastness results comparable to conventional reduction clearing. These results were obtained with simultaneous dyebath decolorisation ratios up to 67% and without significant colour yield (*K/S* value) losses of the dyed fabric. Ozonation periods exceeding 3 min caused significant colour yield (*K/S* value) losses, although dyebath decolorisation ratios increased up to 82% and wash fastness properties further improved. The chemical oxygen demand of the dyeing process decreased up to 62% by the ozonation afterclearing. The advantages of the ozonation afterclearing process are savings in terms of water, energy and time and reduction in environmental load.

Coloration
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Introduction

Afterclearing of disperse dyed polyester

The wash fastness properties of poly(ethylene terephthalate) (PET) fibre and its blends are a major problem when dyeing with disperse dyes. Disperse dyes aggregate and deposit on the surface of the fibre. The deposited disperse dyes on the surface decrease the wash fastness, sublimation fastness and crock fastness properties [1–5]. Afterclearing with reducing agents is the most preferred approach to remove the deposited disperse dyes on the surface. Disperse dye molecules adsorbed on surfaces are broken down into smaller, often colourless and more readily water-soluble fragments by reduction clearing. Reduction clearing is the process of treating the dyed fibre in a strong reducing bath, usually made up of sodium dithionite and caustic soda. The drawbacks associated with this method (i.e. conventional reduction clearing) include the requirement for strong alkaline conditions as well as large amounts of water and a discharge of sulphur with the wastewater [6–9]. Examples of other reducing agents, instead of sodium dithionite, are thiourea dioxide (TUDO), inorganic salts, reducing agents that do not require alkali and liquid reducing agents [6–9]. The selection of disperse dyes with high thermo fixation fastness and the selection of alkali-clearable disperse dyes are alternative ways to achieve high wash fastness, but dye selection is limited [6,10–12]. Oxidative clearing of PET with hydrogen peroxide was reported to yield high wash fastness results and lower chemical oxygen demand values (COD) compared with conventional reduction clearing. The colour of the dyed PET fabric did not change significantly during oxidative clearing [13,14].

Impressive colour and wash fastness results were reported with ozonation afterclearing for three disperse dyes of low, medium and high energy levels in neutral water at room temperature [15].

Decolorisation of dyebath effluent

Colour in textile effluent is a major problem and consumer demands for high colour fastness of fabrics require the use of dyes which are resistant to degradation and are not readily degraded in biological treatment plants [16–18]. Oxidative treatments seem to be the most promising among various decolorisation processes such as coagulation, flocculation, adsorption on activated carbon, filtration and oxidation methods [19–22]. Ozone combinations are widely used in oxidation methods [23]. Ozone (O_3) has a high oxidation potential of 2.07 V while the oxidation potential of hydrogen peroxide is 1.77 V. Ozone has been studied for colour removal from textile effluent and successful results have been reported for decolorisation of disperse dyes [24–29].

Decolorisation with ozonation occurs by breaking down the dye into colourless fragments. In fact, afterclearing of disperse dyed PET also targets the break down of disperse dyes deposited on the surface of the fibre. Therefore, ozonation can be utilised for afterclearing of disperse dyed PET fabrics if a significant change in colour does not occur.

The potential benefits of simultaneous decolorisation and afterclearing by ozonation are a decrease in environmental pollution by avoiding the use of reduction clearing chemicals, decolorisation of the dyebath effluent and savings in water, energy and time. In this study, the effectiveness of ozonation during the afterclearing of disperse dyeing of PET has been studied.

Experimental

Materials

The greige 100% PET woven fabric used in the experiments was supplied by Nergis Tekstil Co. (Turkey). The fabric had the following specifications: plain weave, warp density 16 ends/cm, weft density 15 picks/cm

and fabric weight 108 g/m². Both warp and weft were 300/72 denier/filament textured-intermingled filament yarns.

Three disperse dyes from low, medium and high energy levels were selected. These dyes were CI Disperse Blue 56 (low energy level anthraquinone disperse dye), CI Disperse Blue 60 (medium energy level anthraquinone disperse dye) and CI Disperse Blue 79 (high energy level azo disperse dye). The dyes used in the study were supplied by Ciba (Switzerland). The commercial names of the other chemicals used can be privately provided by the author.

Preparation and dyeing of the substrates

The greige fabric was scoured with 1 g/l anionic/nonionic detergent (Gemsan, Turkey) and 1 g/l caustic soda at 60 °C for 30 min at a liquor ratio of 15:1 to remove mill dirt and lubricants. The fabric was rinsed under tap water and dried at room temperature after scouring.

Samples of PET fabric were dyed at 3% depth of shade with the selected disperse dyes. Dyeing of fabrics was carried out using a laboratory-scale Dyetech Polybath dyeing machine (Boyap Co., Turkey) at a liquor ratio of 15:1. The dyeing recipe was: 3% owf disperse dye, 1 ml/l dispersing agent (anionic; Ciba), acetic acid to pH 4, at 130 °C for 45 min. The dyeing procedure is illustrated in Figure 1.

All dyes and chemicals used in the experiments were commercial grades and were used without further purification. The chemicals used during ozone and COD analysis were reagent grades. Distilled water was used in the dyeing and afterclearing baths.

Afterclearing of the dyed PET fabric

Dyed material was divided into pieces and each piece was subjected to different afterclearing conditions. The effectiveness of ozonation afterclearing to remove surface disperse dye was compared with conventional reduction clearing with sodium dithionite which was attained as the control after-treatment.

The conventional reduction clearing was applied using 2 g/l sodium dithionite and 2 g/l sodium hydroxide at 80 °C for 20 min at a liquor ratio of 15:1. The ozonation treatments were performed in the cooled dyebaths without further adjustment of the pH (pH 4 of the dyebath effluent). The ozonation treatment periods were 1, 3, 5, 10 and 15 min. All ozonation trials were performed at room temperature at a liquor ratio of 15:1.

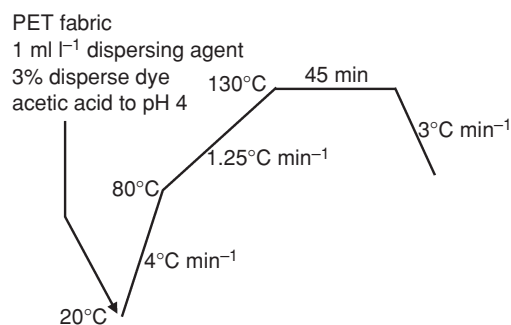


Figure 1 Procedure used for dyeing the PET fabric

Ozone application procedure

The ozonation experiments were conducted in a glass reactor with a closed cylindrical shape having a total volume of 140 ml and diameter of 27 mm. An Opal OS1 model ozone generator (Opal Ltd, Turkey) with an ozone production rate of 3 g/h generated the ozone needed in the reactions. The bottom of the reactor was equipped with a diffuser to produce fine ozone bubbles, which was generated by feeding high purity oxygen of 99.5% into the ozone generator. Teflon tubing was used for the connection between ozone generator and reactor. All experiments were performed at room temperature (20 ± 2 °C) using 45 ml dyebath solution. The ozone gas flow rate was adjusted to 400 ml/min before all the experiments.

Ozone concentration was measured according to the standard iodometric method (Standard Methods 2350 E, APHA, 19th edn, American Public Health Association, 1995). Outlet ozone from the ozone generator was directed to the gas washing bottles filled with 2% (w/w) potassium iodide (KI) solution for the determination of ozone concentration. The ozone concentration was measured as 12.8 ± 0.3 mg/min at 400 ml/min ozone gas flow rate by feeding high purity oxygen into the ozone generator.

The diffuser was placed at the bottom and consequently ozone bubbles introduced to the bottom of the reactor passed through the dyebath and the immersed fabric samples before leaving the reactor.

An intense foam formation was observed during ozonation of the dyebath effluent. Therefore, 0.1 g/l anti-foaming agent (nonionic; Setaş, Turkey) was added to the dyebath prior to ozonation to avoid foam formation. The anti-foaming agent was also added to the reduction clearing baths for consistency in COD evaluations.

The ozonated PET fabric samples were immediately rinsed under tap water with mechanical agitation (stirring) for 2 min to remove residual ozone and avoid any effects of residual ozone on the colour of the samples.

The ozonated dyebaths (i.e. afterclearing baths) were collected in glass tubes and the transmittance measurements were made after 24 h to ensure the consumption of dissolved ozone in the ozonated dyebaths.

Colour strength and colour difference measurements

After the respective after-treatment processes, the visual colour yield of each dyed substrate was measured, at the wavelength of maximum absorbance (λ_{max}) of the dye, using a Macbeth MS2020 spectrophotometer (GretagMacbeth LLC, USA) and expressed in *K/S* values (colour strength). The *K/S* values were calculated using the Kubelka–Munk equation and were determined from three different areas of each fabric sample. The measurements were taken twice on each side of the fabric for consistency and then the average value was calculated. Conventional reduction cleared fabric samples were used as control samples during colour difference measurements. The λ_{max} of the dyed fabric samples were 620, 680 and 600 nm for CI Disperse Blue 56, 60 and 79, respectively.

Dyebath and afterclearing bath absorbance measurements

Optical absorbance of the dyebaths was measured at the start and at the end of each process. The transmittance/absorbance values were measured using a Macbeth MS2020 spectrophotometer (GretagMacbeth LLC).

The averages of 16 absorbance values of dyebath effluent between 400 and 700 nm with 20 nm intervals were calculated to indicate the colour of the dyebath effluent. The decolorisation (%) ratios were then calculated using Eqn 1:

$$\text{Decolorisation (\%)} = [(A - A_0)/A_0] \times 100 \quad (1)$$

where A is the average absorbance (nm) of the ozonated dyebath effluent and A_0 is the average absorbance (nm) of the dyebath effluent.

Wash fastness evaluation

Wash fastness was determined according to the ISO 105-C06:C2S test in a Termal model 410N wash fastness test apparatus (Termal Lab., Turkey). The tests were carried out at 60 °C for 30 min.

COD evaluation

COD values were measured according to the standard titrimetric method (Standard Methods 5220 C: Closed Reflux, Titrimetric Method, APHA, 19th edn, American Public Health Association, 1995).

Results and Discussion

Change in colour yield of dyed fabrics after different clearing treatments

The colour yields (K/S values) and colour differences of PET samples dyed with CI Disperse Blue 56, 60 and 79

after different clearing treatments, are shown in Table 1. Increasingly severe changes in shade were observed in the colour yields of the dyed PET samples during ozonation after exceeding 3 min application time for all selected disperse dyes.

The change in shade indicates that not only were the dyes deposited on the surface, but also the fixed dyes in the PET fibre were attacked during ozonation. Probably, oxidising compounds formed during ozonation are able to penetrate into the PET fibre and oxidise the fixed dye in the fibre to decrease the colour yields (K/S values). Ozone decomposes to produce hydroxyl free radicals at neutral and weakly acidic pH [30]. It was reported in an earlier work that ozonation of PET fabric caused strength loss and decrease in the maximum load [15]. The severity of the strength loss increased as the ozonation period increased and reached 13.5% for 15 min ozonation. The high strength losses, despite the known good stability of PET fibres to oxidation and reduction agents, were attributed to the high oxidation potential of O_3 as well as the ability of oxidising compounds to diffuse into the fibre [15].

There is no standard limit for colour difference (ΔE) but a value of $\Delta E = 1$ is a useful assumption as a limit to assess the significance of colour difference. Colour difference values in Table 1 show that 3 min ozonation time should not be exceeded to avoid significant shade changes for all selected disperse dyes. Colour difference values for 5 min ozonation were measured as 1.02 and 1.17 for CI Disperse Blue 56 and 60, respectively. These values are very close to the assumed limit of $\Delta E = 1$. However, they still fail as they are over the limit.

It is known that reduction clearing destroys azo disperse dyes through chemical reduction of the azo link.

Table 1 Colour yield (K/S) and colour difference of PET fabric samples dyed with CI Disperse Blue 56, 60 and 79 individually and subjected to different clearing treatments^a

Clearing treatment	Time (min)	K/S	ΔE	ΔL	ΔC	ΔH
CI Disperse Blue 56						
Reduction clearing	20	23.0	Reference	26.8 ^b	40.3 ^b	283 ^b
Ozonation	1	23.4	0.52	-0.37	-0.36	0.03
Ozonation	3	23.9	0.58	-0.52	-0.24	0.03
Ozonation	5	23.5	1.02	-0.37	-0.89	-0.35
Ozonation	10	22.7	2.08	-0.28	-2.31	-1.56
Ozonation	15	22.5	3.07	-0.11	-2.49	-1.79
CI Disperse Blue 60						
Reduction clearing	20	17.6	Reference	48.7 ^b	39.9 ^b	244 ^b
Ozonation	1	18.0	0.38	-0.31	-0.14	-0.15
Ozonation	3	17.9	0.24	-0.21	-0.12	-0.03
Ozonation	5	17.3	1.17	0.00	-0.98	-0.64
Ozonation	10	16.2	3.69	0.25	-3.19	-1.85
Ozonation	15	15.6	4.87	0.35	-4.40	-2.05
CI Disperse Blue 79						
Reduction clearing	20	24.9	Reference	20.9 ^b	21.2 ^b	273 ^b
Ozonation	1	24.1	0.30	0.30	0.03	0.05
Ozonation	3	24.8	0.87	0.37	-0.09	-0.78
Ozonation	5	22.5	3.09	2.03	-0.07	-2.33
Ozonation	10	22.4	4.18	2.67	0.15	-3.21
Ozonation	15	20.1	5.82	4.21	0.51	-3.98

C, chroma; H, hue; L, lightness; ΔE , colour difference

^a Reduction clearing with 2 g/l sodium dithionite + 2 g/l sodium hydroxide at 80 °C; ozonation at room temperature in the dyebath, 12.8 mg/min ozone concentration at 400 ml/min gas flow rate; 0.1 g/l anti-foaming agent and liquor ratio 15:1

^b Absolute L , C , H values of the reference reduction cleared samples

Anthraquinone disperse dyes are not fully destroyed during reduction clearing but converted to alkali-leuco form [7,31]. Lightness did not show a significant change (ΔL) for anthraquinone disperse dyes CI Disperse Blue 56 and 60, although a considerable change occurred for azo disperse dye CI Disperse Blue 79 for ozonation periods exceeding 3 min. This difference can be attributed to the differences in the chemical structures of the dyes. The high ΔL values for CI Disperse Blue 79 indicate the decomposition of this dye during ozonation, whereas the ΔL values of the anthraquinone disperse dyes indicate that these dyes did not decompose. However, the ΔC and ΔH values for CI Disperse Blue 56 and 60 indicate that these dyes were partly destroyed resulting in the changes of chroma and hue. These results indicate the similarity between oxidation during ozonation and reduction during reduction clearing.

The colour yields and colour differences of PET fabric samples presented in Table 1 show that ozonation of dyed PET fabric can be safely applied without significant shade changes for up to 3 min ozonation periods at an ozone concentration of 12.8 mg/min and gas flow rate of 400 ml/l. Significant shade changes occur above 3 min application time.

Discussion on the shade changes of the dyed fabric samples after ozonation in neutral distilled water was made for the same dyes in an earlier publication [15]. Similar findings were also reported for the changes in shades of the dyed fabrics.

Decolorisation of dyebath effluent after ozonation (afterclearing) treatments

The results of the decolorisation ratio calculations of the dyebath effluent for different ozonation periods are given in Table 2. The results indicate a simultaneous decolorisation in the dyebath effluent during ozonation afterclearing of the dyed PET samples. The decolorisation ratios increased with the prolonged ozonation periods. However, the increase in decolorisation ratios decelerated above 10 min.

In fact, the decolorisation ratios for 3 min ozonation are important here, because this treatment targets not only the decolorisation of the dyebath effluent, but also the

afterclearing of dyed PET fabric samples. Significant colour yield losses occurred for ozonation periods exceeding 3 min, limiting the application of the treatment above 3 min. The decolorisation ratios reached for 3 min ozonation were 67%, 31% and 57% for CI Disperse Blue 56, 60 and 79, respectively.

The final absorbance values (A) of the anthraquinone dyes were found to be close to each other and much greater than that of the azo dye (Table 2). The reason is thought to be the more rapid destruction of the azo disperse dye through chemical oxidation of the azo link. There is a considerable difference between the decolorisation ratios of the dyebaths of two anthraquinone dyes, although the final absorbance values of their dyebaths are close to each other. Actually, the difference in the decolorisation ratios between the dyebaths of these two anthraquinone dyes essentially arises from the difference of the initial absorbance (A_0) values. The initial absorbance (A_0) of the dyebath of CI Disperse Blue 56 is greater than twice the initial absorbance (A_0) of the dyebath of CI Disperse Blue 60.

Effect of ozonation on wash fastness

The ISO 105-C06:C2S wash fastness results after different clearing treatments are shown in Table 3. The wash fastness ratings of the ozonated samples were measured by the grey-scale method indicating no staining on the adjacent multifibre test fabric for 15 min ozonation. However, the fixed dyes in the fibre were also destroyed above 3 min ozonation periods resulting in significant shade changes, as discussed above. Therefore, the wash fastness results of 1 and 3 min ozonation periods are significant here.

The wash fastness ratings for the 1 min ozonation period were quite insufficient for CI Disperse Blue 79, and were half a grey-scale degree lower for CI Disperse Blue 56 and identical for CI Disperse Blue 60 when compared with conventional reduction clearing. In fact, CI Disperse Blue 60 showed very good wash fastness at all tested conditions.

The poor wash fastness results of CI Disperse Blue 56 and 79 for 1 min ozonation improved by increasing the ozonation period to 3 min. The wash fastness of the

Table 2 Absorbance (nm) and decolorisation ratios (%) of the dyebath effluent of CI Disperse Blue 56, 60 and 79 subjected to ozonation^a for the afterclearing of dyed PET samples in the dyebath

Ozonation time (min)	A (nm)/decolorisation (%)		
	CI Disperse Blue 56 ^b	CI Disperse Blue 60 ^c	CI Disperse Blue 79 ^d
1	0.58/36	0.36/14	0.21/36
3	0.30/67	0.29/31	0.14/57
5	0.21/76	0.26/38	0.09/72
10	0.17/81	0.22/47	0.07/78
15	0.17/81	0.22/47	0.06/82

^a Ozonation at room temperature in the dyebath, 12.8 mg/min ozone concentration at 400 ml/min gas flow rate; 0.1 g/l anti-foaming agent and liquor ratio 15:1

^b $A_0 = 0.89$ nm

^c $A_0 = 0.42$ nm

^d $A_0 = 0.33$ nm

Table 3 Wash fastness properties of CI Disperse Blue 56, 60 and 79 on PET fabric samples after different clearing treatments^a

Clearing treatment	Time (min)	Acetate stain	Nylon stain	PET stain
CI Disperse Blue 56				
Reduction clearing	20	4–5	4	4–5
Ozonation	1	4	3–4	4–5
Ozonation	3	4–5	4	5
Ozonation	5	5	4–5	5
Ozonation	10	5	4–5	5
Ozonation	15	5	5	5
CI Disperse Blue 60				
Reduction clearing	20	5	5	5
Ozonation	1	5	5	5
Ozonation	3	5	5	5
Ozonation	5	5	5	5
Ozonation	10	5	5	5
Ozonation	15	5	5	5
CI Disperse Blue 79				
Reduction clearing	20	4–5	4	4–5
Ozonation	1	2	2–3	3
Ozonation	3	4	4	4–5
Ozonation	5	4–5	4–5	5
Ozonation	10	5	5	5
Ozonation	15	5	5	5

^a Reduction clearing with 2 g/l sodium dithionite + 2 g/l sodium hydroxide at 80 °C; ozonation at room temperature in the dyebath, 12.8 mg/min ozone concentration at 400 ml/min gas flow rate; 0.1 g/l anti-foaming agent and liquor ratio 15:1

samples dyed with CI Disperse Blue 56 and 60 were found to be the same with the conventional reduction cleared samples for 3 min ozonation afterclearing. The staining of the acetate fibre was half a grey-scale degree higher while the staining of the PET and nylon fibres were the same as that of the conventional reduction cleared CI Disperse Blue 79 samples for 3 min ozonation. The colour yields and colour differences of dyed PET fabric did not show a significant change in colour shade for the 3 min ozonation period, as discussed above. Therefore, these wash fastness ratings, comparable to

conventional reduction clearing, for 3 min ozonation afterclearing can be regarded as successful wash fastness results.

The wash fastness ratings presented in Table 3 showed that the wash fastness of the disperse dyed PET fabrics can be effectively improved by ozonation. The wash fastness results further improve at prolonged ozonation periods, but the increasing colour difference is a limiting factor for longer ozonation periods.

Environmental benefit of the ozonation afterclearing

The results of the COD measurements are shown in Table 4. COD removal ratios up to 70% were reached with 15 min ozonation afterclearing. However, the appropriate time for ozonation afterclearing was found to be 3 min at the studied circumstances considering colour evaluations, as discussed above.

The COD removal ratios after 3 min ozonation were 48%, 58% and 62% for CI Disperse Blue 56, 60 and 79, respectively. It must be stated here that these COD removals mainly arise from the exchange of the reduction clearing step by the ozonation afterclearing. The COD removals of the dye bath effluent for 3 min ozonation were only 3, 5 and 6 for CI Disperse Blue 56, 60 and 79, respectively.

Conclusions

A novel study has been conducted by applying a decolorisation procedure (ozonation) to a textile process (afterclearing of disperse dyed PET). Individual studies have been made on the decolorisation of textile dyeing effluent and on fastness properties of disperse dyed PET. However, these studies were never combined in previous research.

In this study, three disperse dyes of low, medium and high energy levels were subjected to ozonation afterclearing. Promising decolorisation and wash fastness results were obtained with a 3 min ozonation period in the dyebath at room temperature. Decolorisation ratios up to 67%, COD removal ratios up to 62% (mainly

Table 4 Relative chemical oxygen demand (COD) values (%) of the dyebath effluent, reduction clearing effluent and ozonated^a dyebath effluent of CI Disperse Blue 56, 60 and 79

	COD (%)		
	CI Disperse Blue 56 ^b	CI Disperse Blue 60 ^c	CI Disperse Blue 79 ^d
Dyebath effluent	53	44	40
Reduction clearing effluent ^e	47	56	60
Total	100	100	100
<i>Ozonated dyebath effluent</i>			
Ozonation time (min)			
1	53	44	39
3	52	42	38
5	50	40	38
10	48	38	36
15	37	32	30

^a Ozonation at room temperature in the dyebath, 12.8 mg/min ozone concentration at 400 ml/min gas flow rate; 0.1 g/l anti-foaming agent and liquor ratio 15:1

^b 100% COD = 5760 mg/l

^c 100% COD = 4032 mg/l

^d 100% COD = 4320 mg/l

^e Reduction clearing with 2 g/l sodium dithionite + 2 g/l sodium hydroxide and 0.1 g/l anti-foaming agent at 80 °C for 20 min

arising from the exchange of the reduction clearing step by the ozonation afterclearing) and wash fastness results comparable to reduction clearing were obtained by ozonation afterclearing.

Ozonation afterclearing yields simultaneous afterclearing of dyed PET fabric and decolorisation of dyebath effluent and can lead to the following advantages: energy and time savings, as it is performed at room temperature only for 3 min; and water savings, by utilising the dyebath for the afterclearing treatment instead of using fresh water; and environmental load reduction, by avoiding the use of harsh conventional reduction clearing chemicals and by the removal of the colour of the dyebath effluent (i.e. decolorisation).

The ozonation period should not exceed 3 min for the maintained conditions to avoid significant colour yield losses. The optimum conditions were room temperature and an ozone concentration of 12.8 ± 0.3 mg/min at an ozone gas flow rate of 400 ml/min. Ozonation afterclearing of the dyed fabric was performed in the cooled dyebath effluent without further adjustment of pH.

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