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A Parameter Study of the Effect of a Plasma-Induced Ozone Colour-Fading Process on Sulphur-Dyed Cotton Fabric

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Abstract: A plasma-induced ozone colour-fading treatment was used for treating a blue sulphur-dyed knitted cotton fabric. Since the process parameters of plasma-induced ozone colour-fading treatment are inter-related with one other, the final colour-fading results are affected. An orthogonal array testing strategy (OATS) method was used for determining the optimum conditions of the plasma-induced ozone colour-fading treatment in this study. Three process parameters used in the plasma-induced ozone colour-fading treatment, i.e., oxygen gas concentration (%), water content in fabric (%), and treatment time (minutes), were used in the optimization process. Experimental results reveal the optimum conditions for fading the colour by plasma-induced ozone colour-fading treatment are: (1) oxygen gas concentration = 70%; (2) water content in fabric = 35%; and (3) treatment time = 30 min. The order of importance of these parameters is: oxygen gas concentration > water content in fabric > treatment time. In addition, the plasma-induced ozone colour-fading treatment can effectively remove the colour from the dyed fabric and the colour-fading effect is uniform and even.

Keywords: cotton fabric; sulphur dye; plasma; ozone; colour-fading

1. Introduction

Colour fading of textile apparel has attracted much attention recently because of high customer acceptance of such materials in the market [1,2]. In textile applications, the colour-fading process can be classified as a finishing method to provide aesthetic effect of worn and vintage look in the products. There are many different colour-fading processes that can be used for such textile applications [3–5]. However, much of the conventional colour-fading processes involve the use of large quantities of water and chemicals, which increases the generation of effluents [6,7]. Recently, some have contemplated the use of sustainable technologies for replacing conventional chemical processes in textile applications. Among different sustainable technologies, plasma treatment is viewed as a possible way to substitute conventional chemical processes because it is a dry process and with the use of suitable reactive gas, the desired final effect can be achieved [8–13]. Table 1 compares the advantage of plasma treatment over conventional wet chemical processes [6]. In our previous studies, we have investigated the possibility of using oxygen plasma treatment in atmospheric pressure conditions for achieving a colour-fading effect on dyed cotton fabric [6,14]. In plasma treatment using oxygen, the oxygen can generate different active plasma species (O_3 and O•) [15,16]. The ozone (O_3) generated is a strong oxidising agent [17,18] which is readily dissolved in water to become an effective oxidant (•OH radical) for colour fading [15]. In addition, UV light is generated as a by-product during plasma treatment with oxygen. The UV light assists in the generation of hydroxyl (●OH) radicals which are responsible for colour fading in dyed fabric [15–18]. Since the effectiveness of the plasma-induced ozone colour-fading treatment can be controlled by process parameters [6,14], this study is aimed to study the effect of process parameters of plasma-induced ozone colour-fading treatment on the colour properties of a blue sulphur-dyed knitted cotton fabric.

Table 1. Comparison of plasma treatment over conventional wet chemical processes [6].

Methods	Advantages	Disadvantages	References
Chemical process e.g., hydrogen peroxide or sodium hypochlorite	Fast operating processes Simplest Application	Most Expensive Wastewater Water Pollution Disposal problem as concentration of sludge Excessive chemicals usage High electrical energy Toxic Chemicals usage (e.g., Acid) Time consuming Inability to create standard designs Not possible on all textile surfaces Loss of quality	[19–22]
Plasma Treatment	Low cost Most rapidly for surface modification No solid waste No air pollution No water pollution Dry operation Wide range of textile surface can be done Even modification result	Modification of the parameters before starting operation Less skilled operating skills	[19,20,23]

2. Experimental

2.1. Knitted Cotton Fabric and Sulphur Dye

A 100% ready-for-dyeing knitted cotton fabric (Lacoste type, yarn count = 32 s/2, fabric weight = 220 g/m^2) was used. The knitted cotton fabric was conditioned at 21 ± 1 °C and 65 ± 2% relative humidity for at least 24 h before use. Sulphur dye (Diresul Blue RDT-2G liq 150 pre-reduced solubilised sulphur dye; supplied by Archorma, Shanghai, China) was used (as received) for dyeing the knitted cotton fabric.

2.2. Dyeing of Knitted Cotton Fabric with Sulphur Dye

The dyeing of knitted cotton fabric with sulphur dye in this study consisted of three stages: dyeing → oxidation → soaping [14].

Stage 1: Dyeing

The dyeing process was conducted in a liquor-to-goods ratio of 10:1 and the dyeing profile is as shown in Figure 1. The dyeing depth is 1.5% on-weight of fabric (owf).

Stage 2: Oxidation

After dyeing, the sulphur-dyed knitted cotton fabric was oxidized with a liquor-to-goods ratio of 10:1 by using 35% hydrogen peroxide (conc. 2% owf). The pH for the oxidation process was 4–4.5. The oxidation temperature and time were 50 °C and 20 min, respectively [14].

Stage 3: Soaping

After oxidation, the sulphur-dyed knitted cotton fabrics were washed with running water for at least 5 min (until no colour was seen in the running water) followed by soaping with detergent. The soaping process was carried out at 90 °C for 10 min for removing unfixed and surface-deposited sulphur dye from the fabric. After soaping, the fabrics were washed with running water. Finally, the knitted cotton fabrics were dried completely in an oven at temperature of 70 °C for at least 30 min.

The dried knitted cotton fabrics were then conditioned at relative humidity of 65 ± 2% at 20 ± 2 °C for at least 24 h before use [14].

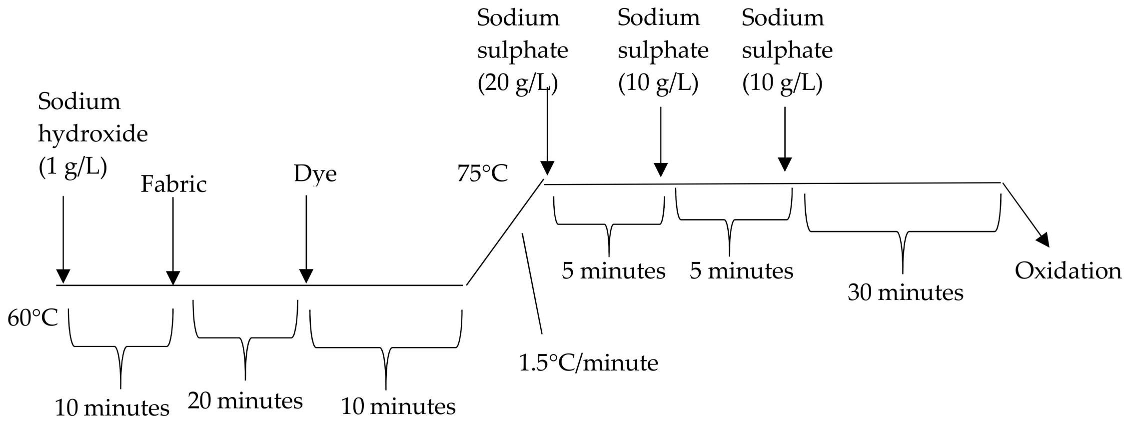

Figure 1. Dyeing profile of sulphur dye.

2.3. *Plasma-Induced Ozone Colour-Fading Treatment*

The plasma-induced ozone colour-fading treatment was carried out in an industrial-scale plasma machine (G2, Jeanologia, Spain) [24–26]. Process parameters of plasma-induced ozone colour-fading treatment affect by each other [27–29]. An orthogonal array testing strategy (OATS) [30–32] method was used for analysing the optimum conditions of plasma-induced ozone colour-fading treatment. Three process parameters in the plasma-induced ozone colour-fading treatment, i.e., (1) oxygen gas concentration (%); (2) water content in fabric (%); and (3) treatment time (minutes), were used for finding the optimum conditions and the experimental arrangements were as shown in Tables 2 and 3 respectively [30–32]. After plasma-induced ozone colour-fading treatment, the knitted cotton fabric samples were conditioned at 21 ± 1 °C with 65 ± 2% relative humidity for at least 24 h before further evaluation.

Table 2. Parameters and levels used in the orthogonal array testing strategy (OATS).

Level	Process Parameters		
	Oxygen Gas Concentration (%)	**Water Content in Fabric (%)**	**Treatment Time (Minutes)**
I	10	35	10
II	50	40	20
III	70	45	30

Table 3. Experimental arrangement.

Test Run	Process Parameters		
	Oxygen Gas Concentration (%)	**Water Content in Fabric (%)**	**Treatment Time (Minutes)**
1	I	I	I
2	I	II	II
3	I	III	III
4	II	I	II
5	II	II	III
6	II	III	I
7	III	I	III
8	III	II	I
9	III	III	II

2.4. Colour Measurement

Spectrophotometer (Color-Eye 7000A, GretagMacbeth, Hong Kong, China) was used for measuring colour properties (K/S value and CIE L*a*b* values) of the knitted cotton fabric. The measuring conditions were: (1) illuminant D_{65}; and (2) 10° standard observer. Five measurements were obtained for each knitted cotton fabric sample and the results were averaged.

2.5. Colour Levelness Measurement

The levelness of colour in the knitted cotton fabric samples was assessed by the relative unlevelness index (RUI) as proposed by Chong et al. [33]; Table 4 summarizes the interpretation of RUI values.

Table 4. Interpretation of the relative unlevelness index (RUI) [33].

RUI Value	Interpretation
<0.2	Excellent levelness
0.2–0.49	Good levelness
0.5–1.0	Poor levelness
>1.0	Bad levelness

3. Results and Discussion

3.1. Optimum Conditions for Colour Fading of Sulphur-Dyed Knitted Cotton Fabric Based on Plasma-Induced Ozone Colour-Fading Treatment

The K/S value is a measurement of colour yield in a dyed fabric: the higher the K/S value, the higher the colour yield [6,14]. The purpose of the colour-fading process is to remove the colour from the dyed fabric. Thus, for a better colour-fading effect introduced by plasma-induced ozone fading treatment, K/S value of the sulphur-dyed fabric knitted cotton fabric is reduced. Table 5 shows the K/S value of dyed fabric after plasma-induced ozone colour-fading treatment according to the OATS arrangement.

Table 5 shows that all the three process parameters of plasma-induced ozone colour-fading treatment, i.e., (1) oxygen gas concentration; (2) water content in fabric; and (3) treatment time, result in different colour-fading effects in terms of the K/S value of sulphur-dyed knitted cotton fabric. The order of importance of these parameters is oxygen gas concentration > water content in fabric > treatment time. On the whole, the optimum conditions for colour fading of sulphur-dyed knitted cotton fabric by plasma-induced ozone colour-fading treatment are identified. By calculating the results obtained from the nine trials, the optimum conditions for the plasma-induced ozone colour-fading process for decolourising the dyed sulphur-dyed knitted cotton fabric are: (1) oxygen gas concentration = 70%; (2) water content in fabric = 35%; and (3) treatment time = 30 min. These optimum conditions were then used for treating the sulphur-dyed knitted cotton fabric for further evaluation.

The effect of concentration of oxygen gas on the sulphur-dyed knitted cotton fabric is shown in Figure 2; when the oxygen concentration increases, the K/S value decreases accordingly, which means that a better colour-fading effect is achieved. When the oxygen concentration increases, more oxygen molecules are affected by the plasma and more active plasma species, e.g., hydroxyl radicals are generated, leading to a higher oxidation effect on the sulphur dye in the dyed fabrics resulting in higher degree of colour fading [6].

Table 5. Orthogonal table for optimising the plasma-induced ozone colour-fading treatment of sulphur-dyed knitted cotton fabric.

Test Run	Parameters			K/S Value
	Oxygen Gas Concentration (%)	Water Content in Fabric (%)	Treatment Time (Minutes)	
1	I	I	I	14.97
2	I	II	II	14.74
3	I	III	III	14.50
4	II	I	II	11.88
5	II	II	III	12.39
6	II	III	I	13.99
7	III	I	III	10.08
8	III	II	I	10.75
9	III	III	II	11.63
∑ in K/S Value	**Parameters**			
	Oxygen Gas Concentration (%)	**Water Content in Fabric (%)**	**Treatment Time (Minutes)**	
∑ I	44.21	36.93	39.71	
∑ II	38.26	37.88	38.25	
∑ III	32.46	40.12	36.97	
Difference	*11.75*	*3.19*	*2.74*	

Values in bold exhibits the smallest value among all the different factors used (the smaller the value, the better the colour-fading effect). Values in italics exhibits the level of importance of each factor (the larger the value, the more important the factor).

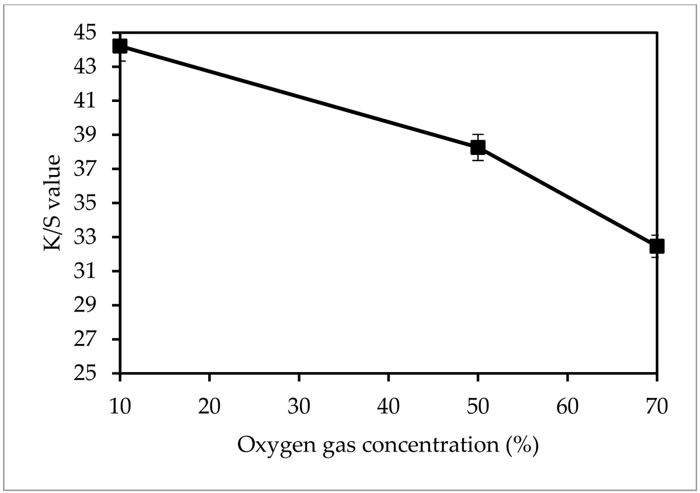

Figure 2. Effect of oxygen gas concentration in plasma-induced ozone colour-fading effect on the sulphur-dyed knitted cotton fabric.

The effect of water content in the fabric treated for plasma-induced ozone colour-fading effect on the sulphur-dyed knitted cotton fabric is shown in Figure 3. Figure 3 shows that the lowest K/S value is obtained at water content of 35%. When water content in the fabric is increased from 35% to 45%, K/S values increase accordingly. Water content in the fabric is very important because water helps promote the bleaching effect caused by the hydroxyl radical in the plasma leading to the colour-fading effect. If the water content in fabric is too low, the water content is insufficient to have a good colour-fading effect. If the water content in fabric is too high, dilution reduces the bleaching effect [14]. In this study, a 35% water content in fabric yielded the best colour-fading effect.

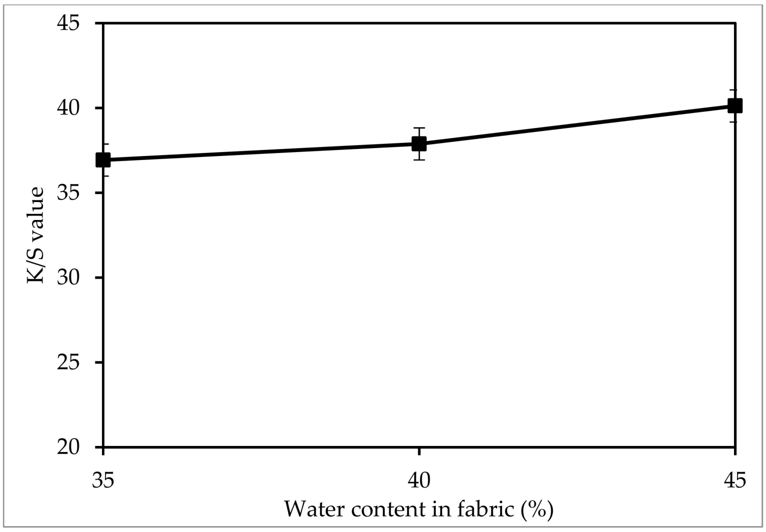

Figure 3. Effect of water content in fabric in plasma-induced ozone colour-fading effect on the sulphur-dyed knitted cotton fabric.

The effect of duration of the plasma treatment on the sulphur-dyed knitted cotton fabric is shown in Figure 4; when the treatment time is increased, the K/S values decrease accordingly. The lowest K/S value is obtained at a treatment time of 30 min. Generally speaking, long treatment times should enhance the interaction between the active plasma species for a greater oxidation effect on the sulphur-dyed knitted cotton fabric, leading to a greater colour-fading effect [11,14].

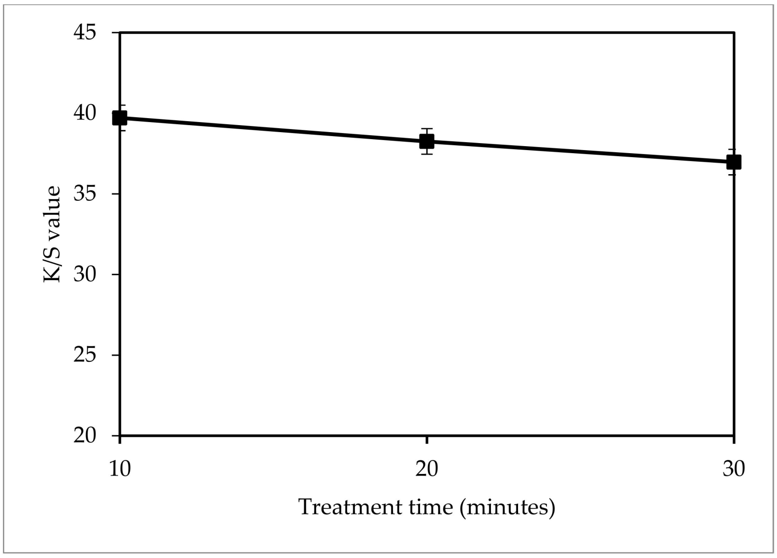

Figure 4. Effect of treatment time of plasma-induced ozone colour-fading effect on the sulphur-dyed knitted cotton fabric.

Sulphur-dyed knitted cotton fabric was plasma treated at the identified optimal conditions, i.e., (1) oxygen gas concentration = 70%; (2) water content in fabric = 35%; and (3) treatment time = 30 min, and the colour yield was measured. Figure 5 shows that the colour yield of the sulphur-dyed knitted cotton fabric plasma treated under the optimum conditions had the lowest colour yield compared to other different treatment conditions. Figure 5 shows that the colour yield of all sulphur-dyed knitted cotton fabric treated with plasma-induced ozone colour-fading treatment obtain a lower colour yield than the untreated sulphur-dyed knitted cotton fabric. This indicates that the plasma-induced ozone colour-fading treatment is good and suitable for colour-fading applications.

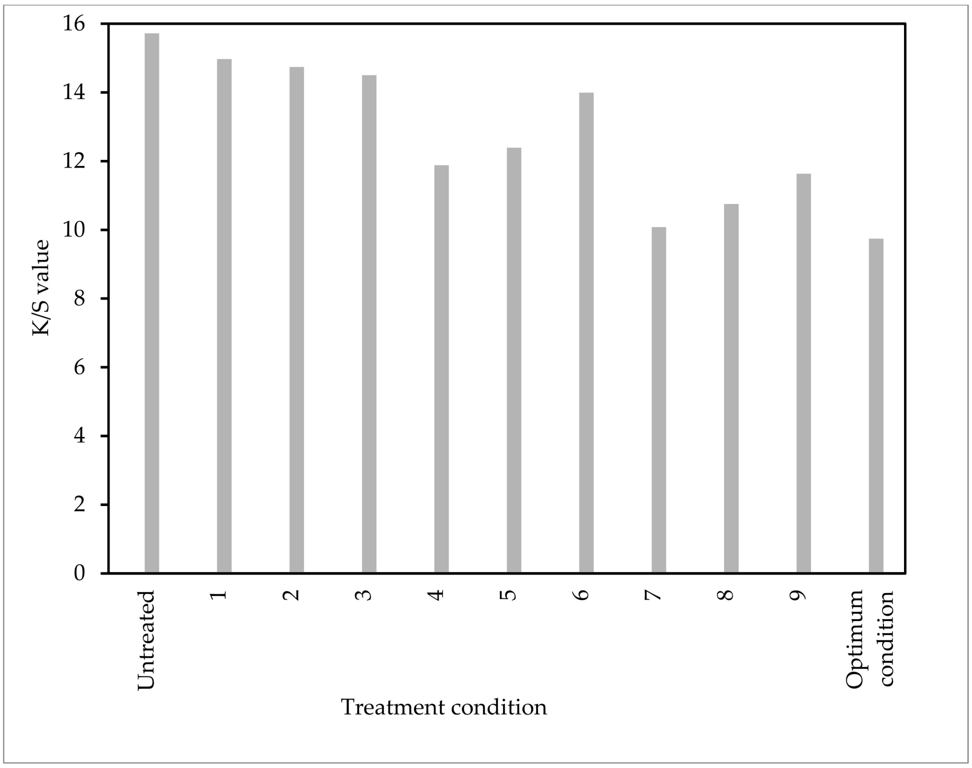

Figure 5. Verification of optimum treatment conditions for plasma-induced ozone colour-fading treatment of the sulphur-dyed knitted cotton fabric.

3.2. Colour Properties Measurement

After obtaining the optimum conditions for plasma treatment a sulphur-dyed knitted cotton fabric specimen was treated under these optimum conditions and its colour properties were measured. Table 6 shows the colour properties (CIE L*a*b* value and levelness) of sulphur-dyed knitted cotton fabric treated under the optimum conditions.

Table 6. Colour properties.

	CIE L*	**CIE a***	**CIE b***	**ΔE**	**Levelness (RUI)**
Untreated	58.15	1.40	−12.86	-	0.30
Plasma-induced ozone colour-fading treated (sample)	79.35	−0.86	7.47	29.46	0.30

Remark: Colour difference (ΔE) = $[(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{\frac{1}{2}}$ (where $\Delta L^* = L^*_{sample} - L^*_{untreated}$; $\Delta a^* = a^*_{sample} - a^*_{untreated}$; and $\Delta b^* = b^*_{sample} - b^*_{untreated}$).

The CIE L* value refers to lightness of the colour in the fabric. The higher the CIE L* value, the lighter/paler is the colour in the fabric. In case of untreated sulphur-dyed knitted cotton fabric, the CIE L* value is 58.15 and the CIE L* value is further increased to 79.35 after plasma-induced ozone colour-fading treatment. There is an increase of 36.5% in the CIE L* value after the plasma-induced ozone colour-fading treatment. This indicates that the colour in the plasma-induced ozone colour-fading treated sulphur-dyed knitted cotton fabric is paler than the untreated sulphur-dyed knitted cotton fabric.

In the CIE system, a* value refers to the measurement of redness/greenness in the fabric sample. A positive a* value generally means a redder shade, while a negative a* value refers to a greener shade of a fabric sample. The untreated sulphur-dyed knitted cotton fabric has a CIE a* value of 1.40 and after plasma-induced ozone colour-fading treatment, the CIE a* value is reduced to −0.86. Although the change in the CIE a* value is not great, the result indicates that the shade of sulphur-dyed knitted cotton fabric changes to slightly green after the plasma-induced ozone colour-fading treatment.

The b* value in CIE system refers to measurement of yellowness/blueness colour properties in the fabric sample. A positive b* value means yellowish shade, while a negative b* refers to a bluish shade in the fabric sample. Obviously, after plasma-induced ozone colour-fading treatment, the b* value of the sulphur-dyed knitted cotton fabric changes significantly from a negative to a positive value. This change in the CIE b* value reveals that the blue shade of the sulphur-dyed knitted cotton fabric is diminished after the plasma-induced ozone colour-fading treatment.

Based on the results of CIE L*, a* and b* values, the colour difference (ΔE) between the untreated sulphur-dyed knitted cotton fabric and the plasma-induced ozone colour-fading treated sulphur-dyed knitted cotton fabric can be determined. If there is no colour difference between the two fabric samples, the ΔE value should be zero. However, the difference between ΔE values of the two sulphur-dyed knitted cotton fabric samples is 29.46 in this study. This ΔE value indicates that the colour differences between the two sulphur-dyed knitted cotton fabric samples are obvious and significant.

Finally, the degree of levelness of colour in a fabric is an important requirement for assessing a dyed fabric. The levelness refers to the uniformity/evenness of the colour in the dyed fabric. Although plasma treatment removes colour from the dyed fabric, it is also important that the colour is uniform, even in the case of a colour-faded fabric. Experimental results reveal that the levelness of the untreated and colour-faded sulphur-dyed knitted cotton fabric have the same RUI value of 0.30, which means they have good levelness when compared instrumentally. Based on colour properties measurement, it can be concluded that the plasma-induced ozone colour-fading treatment can effectively remove the colour from the dyed-fabric sample and the colour-fading effect is uniform and even.

4. Conclusions

In this study, we investigated the effect of the plasma-induced ozone colour-fading treatment on a sulphur-dyed knitted cotton fabric (blue colour). The effect of the three operational parameters of plasma-induced ozone colour-fading treatment, i.e., oxygen gas concentration, water content in fabric, and treatment time, on the K/S value was investigated through OATS. The optimum conditions for the plasma-induced ozone colour-fading process for the dyed sulphur-dyed knitted cotton fabric were: (1) oxygen gas concentration = 70%; (2) water content in fabric = 35%; and (3) treatment time = 30 min. The order of importance of these parameters was oxygen gas concentration > water content in fabric > treatment time. In accordance with the measured colour properties (i.e., CIE L*a*b* value and levelness), it can be concluded that the plasma-induced ozone colour-fading treatment can effectively remove colour from the dyed-fabric sample, and the colour-fading effect is uniform and even.

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Conflicts of Interest: The authors declare no conflict of interest.

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