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Investigation of the Treatment of Textile Wastewater with Cold Atmospheric Plasma Reactor (Profoks) and Reuse of Recycled Water in Reactive Dyeing Process of Cotton

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ABSTRACT

In this study, Profoks reactor (Cold Atmospheric Plasma reactor based on Dielelectric Barrier Discharge) was used for the treatment of textile wastewater and then the usability of the recycled water in the reactive dyeing process was investigated. After processing of 30 L textile wastewater with Profoks system under laboratory conditions, 99% efficiency was obtained in chemical oxygen demand (COD) and total suspended solid (TSS) parameters. In the wastewater sample, the treated water color amount was reduced to less than 1.5 units in Pt-Co and the conductivity value was reduced to 7790 Us/cm. In dyeing of 100% cotton Ne 30/1 single jersey (190 g/m^2) fabric with reactive dyes having azo chromophore group under laboratory conditions, the dyeings made with recovery water with Profoks system were similar to those dyed with osmosis water in terms of color, levelness and fastness performance.

摘要

本研究采用教授反应器（基于双电阻挡放电的低温常压等离子体反应器）处理纺织废水，并对再生水在活性染料染色过程中的可用性进行了研究. 在实验室条件下，用教授系统处理30升纺织废水后，化学需氧量（COD）和总悬浮物（TSS）参数的效率达到99%. 在废水样品中，处理后的水在Pt-Co中的颜色量减少到小于1.5个单位，电导率值降低到7790 Us/cm. 在实验室条件下，用含偶氮生色基团的活性染料对100%纯棉ne30/1单面（190g/m2）织物进行染色时，用教授系统回收水进行染色，在色泽、匀染性、牢度等方面与渗透水染色相近.

KEYWORDS

Dielelectric barrier discharge; Profoks; cold atmospheric plasma; wastewater treatment; reactive dyeing; fastness

关键词

双电阻挡放电; 教授; 冷大气等离子体; 废水处理; 活性染料; 牢度

Introduction

Increasing population and consequently increasing needs create pressure on water resources. Domestic/urban and industrial water consumption amounts increase day by day and water resources are consumed. Worldwide, it is almost impossible to maintain the model of supplying and consuming water in this way, and these conditions will make water recovery mandatory.

Textile industry is a leading industry for most countries, such as China, Singapore, United Kingdom, Bangladesh, Italy, Turkey, etc. However, environmental pollution is one of the main problem of this industry. Due to usage of huge amounts of water and chemicals, the textile dyeing and finishing industry is one of the major polluters among other industrial sectors (Yonar 2011).

Major pollutants in textile wastewaters are high suspended solids, chemical oxygen demand, heat, color, acids, and other soluble substances (Savin and Butnaru 2008). Typical textile industry wastewater characteristics can be summarized with a COD of 150–12000 mg/L, total suspended solids of

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2900–3100 mg/L, total Kjeldahl nitrogen of 70–80 mg/L and a BOD range of 80–6000 mg/L. Textile wastewater has a BOD/COD ratio of around 0.25, showing that it contains large amounts of non biodegradable organic matter (Inditex 2015). Generally, effluent from textile dyehouses will exhibit some level of residual color, which if discharged into receiving waters is not only esthetically displeasing but also creates serious environmental concerns due to the possible existence of biocidal finishing agents, carriers, surfactants, sequestering agents, leveling agents, etc. that may be present (Yonar et al. 2005). It has been widely shown that advanced oxidation processes (AOP) have the greatest potential for the treatment of textile wastewater (Oller, Malato, and Sánchez-Pérez 2011). AOPs show specific advantages over conventional treatment alternatives because they can eliminate non-biodegradable organic components and avoid the need to dispose of residual sludge. This process implies the generation and subsequent reaction of hydroxyl radicals, which are the most powerful oxidizing species after fluorine radicals (Yonar et al. 2005). Corona discharge is emerging as a promising advanced oxidation process (AOP) for the treatment of a variety of organic contaminants, including compounds that are not effectively destroyed by more common AOPs (Even-Ezra et al. 2009).

In cold atmospheric plasma (CAP), temperature is not thermally equilibrated and varies widely between electrons and other particles (ions, atoms, and molecules). For this reason, CAP is also referred as non-thermal plasma. The temperature of the electrons is typically between 10,000 K and 250,000 K (1–20 eV). These highly energetic electrons produce free radicals from main molecules in multi-stage physical and chemical processes. These free radicals react with pollutants and form decomposed products (Kim 2004).

Nowadays, the reduction of clean water resources makes it necessary to reduce the use of water and to reuse the process water in industrial production. Studies on reuse of recycled water in textile processes are limited in literature. For this reason, in this study it was aimed to investigate the treatment (including decolorization) and recovery of textile wastewater by using Cold Atmospheric Plasma Reactor (Profoks) based on Dielectric Barrier Discharge and its reuse in reactive dyeing as an alternative to conventional osmosis water conventionally used in the textile sector. Although there are various studies on recycling textile wastewaters via electrochemical oxidation, ozonation, UV/H_2O_2 oxidation, nanofiltration, membrane processes, adsorption on activated carbon, Fenton's process, coagulation-floculation, biological treatments, etc., studies also including the reuse of recyled wastewaters in textile dyeing processes (Allègre et al. 2006; Buscio et al. 2015, 2016; Lee and Pavlostathis 2004; Leshem et al. 2006; Lu et al. 2010; Rosa et al. 2015; Rosa, Tambourgia, and Santana 2012) are limited. On the other hand to the best of authors' knowledge, there is no study in literature on the reuse of water, which was recyled by using a CAP reactor based on dielectric barrier discharge, in reactive dyeing. In this study, in the presence of a capacitor, the treatment/recovery efficiency of the Profoks reactor capable of producing the desired amounts of reactive oxygen species and UV radiation at the set wavelength was tried to be demonstrated. The Profoks system is a Reactive Oxygen Species (ROS) reactor where excited O_2 is produced by plasma discharge in gas phase and then bubbled through the wastewater to be treated. Electrical discharge reactors have been recently developed in which bubbles of distant plasma gas are introduced into the wastewater by means of a gas diffuser. The main difference of the Profoks system from other reactors is that only pure O_2 is used as the plasma feed gas. With a new design, the desired amount of reactive oxygen species can be produced.

In textile dyeing processes, it is essential to achieve the desired color uniformly and with desired fastness levels, economically and ecologically. Therefore, in this study, reactive dyeing by using osmosis water was used as a reference and properties (color, levelness, fastness, economy, and ecology) of dyeing carried out by using treated wastewater with Profoks/CAP reactor were compared.

Table 1. Characteristics of the waters used in the experiments.

Measurement Parameters	Osmosis Water	Wastewater Treated with Profoks
pH	8.34	7.95
Conductivity (uS/cm)	9.50	77.90
Hardness (°dH)	0.00	2.00
p-Alkalinity (ppm HCO_3^-)	0.00	0.00
m-Alkalinity (ppm HCO_3^-)	40.00	60.00
Chloride (ppm)	56.72	49.63

Materials and methods

Wastewater treatment trials

In this study, 30 L textile wastewater sample was taken from a textile dyehouse and wastewater treatment and water recovery studies were carried out with Profoks/CAP system under laboratory conditions. Two Profoks reactors producing 100 g of excited oxygen and UV radiation at set wavelength, one ultrafiltration unit and one nanofiltration unit were used. Thirty liters of textile wastewater was discharged into a stainless steel reactor and gas was discharged to the wastewater through two gas diffusers for 90 min. At the end of 90 min, mineralization of organic substances in wastewater and conversion of inorganics to their salts were realized. After oxidation-reduction processes with reactive oxygen species in a batch reactor, wastewater was first passed through ultrafiltration unit and then through nanofiltration unit. Subsequently, the pollutant concentrations (COD, TSS, conductivity, and color) of the influent water and the effluent water before the treatment study were tested and water characterizations were performed.

Investigation of usability of recycled wastewater in reactive dyeing process

All dyeing experiments were carried out using wastewaters treated (and also decolourized) with Profoks/CAP as well as the osmosis water conventionally used by the dyehouses. The results of analysis for each waters are given in Table 1 comparatively.

One hundred percent cotton Ne 30/1 single jersey (190 g/m^2) fabrics were dyed under laboratory conditions with reactive dyes whose C.I. No, reactive and chromophore groups are given in Table 2. Dyeing trials were performed on a laboratory dyeing machine (Mathis Labomat). All experiments were performed at 1:6 liquor ratio.

Reactive dyeing process

Dyeing was started at 40°C with a liqour containing 0.4 g/L sequestering agent and 1.5 g/L anticreasing agent and then reactive dye dosing was performed within 15 min, then salt was dosed within 15 min at 5-min intervals. Then the temperature was rised to 80°C with 2°C/min heating speed and after 10 min of migration at this temperature, the temperature was reduced to 60°C and after 5 min of treatment at this temperature two portions of soda was dosed within 50 min. It was continued for 45 min at this temperature and then the liqour was drained. Then, following washing steps were applied respectively; overflow rinse at 50°C for 5 min, neutralization with acetic acid at 50°C for 6 min; soaping (0.4 g/L) at 90°C for 8 min, soaping (0.4 g/L) at 80°C for 8 min, rinsing at 60°C for 6 min, cold rinsing for 6 min.

Table 2. C.I. No, reactive, and chromophore groups of reactive dyes used in experiments.

C.I. No	Reactive Group	Chromophore Group
Reactive Yellow 176	Vinylsulphone/monochlorotriazine	Azo
Reactive Red 239	Vinylsulphone/monochlorotriazine	Azo
Reactive Blue 221	Vinylsulphone/monochlorotriazine	Azo
Reactive Blue 21	Vinylsulphone	Azo
Reactive Yellow 167	Vinylsulphone/monochlorotriazine	Azo

- ***Color measurements***: Reflectance (R%) values of dyed samples were measured with spectrophotometer (D 65/10°) and color yields (K/S) of dyed samples were calculated by Kubelka Munk equation:

$K/S = (1\text{-}R)^2/2\ R$

R = Reflectance value in maximum absorption wave length (nm)

K = Absorption coefficient

S = Scattering coefficient

Variance analysis was performed over the K/S values obtained in three replicates by using Minitab 15 in order to determine whether the difference between samples dyed by using osmosis water and wastewater treated with Profoks/CAP system are statistically significant or not.

CIE L*a*b* color values were also measured.

L*: Lightness-darkness value (varries between 0 and 100; 0: perfect black, 100: perfect white)

a*: Red-green value (+ redder, – greener)

b*: Yellow-blue value (+ yellower, – more blue)

Fastness tests

Washing (at 50°C), water, perspiration (acid and alkali) and rubbing (dry and wet) fastness values of dyed samples were assessed according to ISO 105-C06 (2010), ISO 105-E01 (2013), ISO 105-E04 (1994), and ISO 105-X12 (1993) standards, respectively.

Results and discussion

Wastewater treatment and recovery results

Table 3 shows the pollutant concentrations and the water characterization analysis results of the influent water before and after the treatment.

COD is one of the most important pollution parameters in industrial wastewater. As can be seen from Table 3, COD parameter could be reduced by 99% after the treatment and it was decreased below 50 mg/L. These results indicate that treatment method provides advanced organic matter mineralization. As a result of treatment process, 99% efficiency was obtained in TSS parameter. The TSS value of treated wastewater indicates that low treatment sludge will be formed. Textile wastewater has high electrical conductivity values depending on the processes. High conductivity is caused by chlorinated compounds of organic and inorganic character, which are present in different forms in the wastewater. After treatment, the effluent water conductivity value was reduced to about 77.90 Us/cm.

Color is one of the most important problems in textile wastewater. Physicochemical techniques or advanced oxidation processes are often used in the treatment of these wastewaters. After the treatment with Profoks/CAP system, 99% removal of color parameter was realized. The effluent color in the wastewater sample was reduced to less than 1.5 units in Pt-Co. Physical appearance of the wastewaters before Profoks treatment, after 45 and 90 min of treatment is given in Figure 1. From Figure 1 it can be seen that a high level of color quality is achieved.

Table 3. The pollutant concentrations and the water characterization analysis results of the influent water before and after the treatment.

Parameter	Unit	Before Treatment	After Treatment
COD	mg/L	2643.65	<50
TSS	mg/L	256.6	<1
Conductivity	uS/cm	1829	77.90
Color	Pt-Co	1150	<1.5

Figure 1. Physical appearance of the wastewaters before Profoks treatment, after 45 and 90 min of treatment.

Results related to the investigation of usability of recycled wastewater in reactive dyeing process

The results of color measurements of dyeings carried out with same recipe and fabric by using osmosis water and wastewater treated with Profoks/CAP system are given in Table 4 and photographs of fabric samples are given in Figure 2.

When Table 4 is examined, it can be observed that the color difference between dyeings carried out by using wastewater treated with Profoks system and reference dyeings with osmosis water is insignificant except for dyeings with turquoise dye. As is known, if the total color difference value (ΔE) is less than 1, the color difference between two samples is considered insignificant. When the L* values expressing the lightness-darkness of the color are examined, it is seen that the L* values of those dyed with wastewater treated with Profoks system are slightly higher (except for the turquoise), i.e., the colors are slightly lighter. However the difference between them is insignificant. The K/S values, which represent the color yield, also gave a parallel result.

Furthermore, variance analysis was also performed over the K/S values in order to show whether the difference between samples dyed by using osmosis water and wastewater treated with Profoks/CAP system are statistically significant or not. Results of variance analysis are given in Table 5.

Table 4. The results of color measurements of dyeings carried out by using osmosis water and wastewater treated with Profoks/CAP system.

Dye	No	Treatment	L*	a*	b*	C	h	ΔE	%R	K/S	nm
2% Reactive Yellow 176	1	Profoks	72.19	28.96	72.95	78.49	68.34	0.75	5.15	8.73	440
	2	Osmosis	72.22	29.66	73.23	79.01	67.95	-	5.11	8.81	440
2% Reactive Red 239	3	Profoks	43.92	56.74	−4.01	56.89	356.01	0.49	4.30	10.64	550
	4	Osmosis	43.62	57.13	−3.98	57.27	356.01	-	4.12	11.15	550
2% Reactive Blue 221	5	Profoks	37.99	−0.94	−30.37	30.39	268.05	0.51	4.85	9.33	630
	6	Osmosis	37.56	−0.80	−30.60	30.61	268.50	-	4.68	9.71	630
2% Reactive Blue 21	7	Profoks	51.91	−34.51	−27.42	44.08	218.46	2.73	1.94	24.81	680
	8	Osmosis	54.55	−34.40	−26.76	43.46	217.87	-	2.19	21.79	680
0.0077% React. Yellow 167 + 0.162% React. Blue 21 + 0.0018% React. Blue 221	9	Profoks	74.60	−27.13	−13.80	30.38	207.02	0.75	12.97	2.92	680
	10	Osmosis	75.05	−26.57	−13.60	29.86	207.11	-	13.62	2.74	680

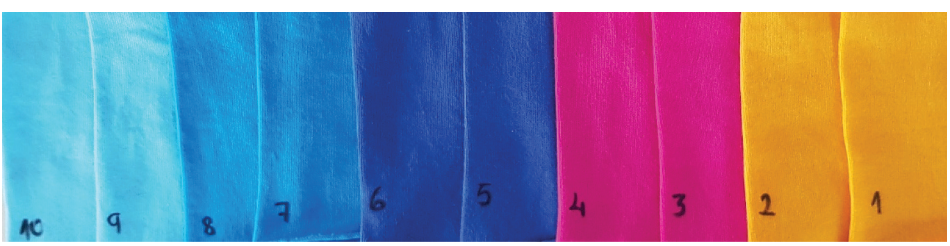

Figure 2. Photos of dyeings carried out by using osmosis water and wastewater treated with Profoks/CAP system.

Table 5. Variance analysis results of K/S values obtained in dyeings.

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Dye type	4	450.3	450.387	112.597	105.12	0.000
Water type	1	0.497	0.497	0.497	0.46	0.533
Error	4	4.285	4.285	1.071		
Total	9	455.16				

From Table 5, it can be seen that differences between K/S values of samples dyed by using osmosis water and wastewater treated with Profoks/CAP system are statistically insignificant ($p > .05$).

When the nuance of the color is examined, it is understood that the a* and b* values of the samples dyed with wastewater treated with Profoks/CAP system are smaller than the ones dyed with osmosis water, i.e., the nuance of the colors obtained are greener and more blue. However, the difference between the nuances of the colors obtained is also not significant even for turquoise dyeing. This means the hue of obtained colors are nearly same. This situation can be understood by checking the h values that indicate the hue of color. As can be seen from Table 4, h values are approximately same for both dyeings carried out with osmosis water and wastewater treated with Profoks/CAP system.

As mentioned before, in textile dyeing processes, it is essential to achieve the desired color uniformly, economically and ecologically with desired fastness levels. For this reason levelness of dyeings have great importance and it can be said that both dyeings carried out with osmosis water and wastewater treated with Profoks/CAP are level according to the visual assesments as can be seen from the photographs of dyed samples given in Figure 2.

Washings, water, perspiration (alkaline and acidic) and rubbing (dry and wet) fastness tests were also applied to the dyed samples and the results are given in Tables 6, Tables 7, 8.

When fastnesss test results given in Tables 6–8 are examined, it can be said that the difference between fastness values of dyeings carried out by using wastewater treated with Profoks/CAP system and reference dyeings with osmosis water is insignificant. In addition, it is remarkable that the overall fastness values of all dyeings are quite good.

Conclusions

The future potential of plasma technology, which has been intensively researched and studied in recent years, is quite remarkable. At this point, it is anticipated that plasma can be a solution to environmental problems and conventional treatment approaches cannot compete with plasma technique.

According to the results obtained from the prototype study to investigate the treatability of wastewater produced by the activities of a textile dyeing mill with Profoks/CAP system, it was demonstrated that an effective purification was achieved according to the measured parameters of

Table 6. Washing and water fastness test results of dyeings carried out by using osmosis water and wastewater treated with Profoks system.

		Washing Fastness						Water Fastness					
Color	Water	CA	CO	PA	PES	PAC	WO	CA	CO	PA	PES	PAC	WO
Reactive Yellow 176	Profoks	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4	4/5	4/5	4/5	4/5
	Osmosis	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4	4/5	4/5	4/5
Reactive Red 239	Profoks	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
	Osmosis	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
Reactive Blue 221	Profoks	4/5	4	4/5	4/5	4/5	4/5	4/5	4	4/5	4/5	4/5	4/5
	Osmosis	4/5	4	4/5	4/5	4/5	4/5	4/5	4	4/5	4/5	4/5	4/5
Reactive Blue 21	Profoks	4/5	3/4	4/5	4/5	4/5	4/5	4/5	4	4/5	4/5	4/5	4/5
	Osmosis	4/5	3/4	4/5	4/5	4/5	4/5	4/5	4	4/5	4/5	4/5	4/5
Reactive Yellow 167+ Reactive Blue 21 + Reactive Blue 221	Profoks	4/5	4	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
	Osmosis	4/5	4	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5

Table 7. Perspiration fastness test results of dyeings carried out by using osmosis water and wastewater treated with Profoks system.

		Perspiration Fastness											
		Alkali						Acid					
Color	Water	CA	CO	PA	PES	PAC	WO	CA	CO	PA	PES	PAC	WO
Reactive Yellow 176	Profoks	4/5	4	4/5	4/5	4/5	4/5	4/5	4	4/5	4/5	4/5	4/5
	Osmosis	4/5	4/5	4	4/5	4/5	4/5	4/5	4/5	4	4/5	4/5	4/5
Reactive Red 239	Profoks	4/5	4	4/5	4/5	4/5	4/5	4/5	4	4/5	4/5	4/5	4/5
	Osmosis	4/5	4	4/5	4/5	4/5	4/5	4/5	4	4/5	4/5	4/5	4/5
Reactive Blue 221	Profoks	4/5	4	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
	Osmosis	4/5	4	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
Reactive Blue 21	Profoks	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
	Osmosis	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
Reactive Yellow 167	Profoks	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
+ Reactive Blue 21 + Reactive Blue 221	Osmosis	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5

Table 8. Rubbing fastness test results of dyeings carried out by using osmosis water and wastewater treated with Profoks system.

		Rubbing Fastness	
Color	Water	Dry	Wet
Reactive Yellow 176	Profoks	4	4/5
	Osmosis	4	4/5
Reactive Red 239	Profoks	3/4	4/5
	Osmosis	3/4	4/5
Reactive Blue 221	Profoks	4	4/5
	Osmosis	4	4/5
Reactive Blue 21	Profoks	3	4/5
	Osmosis	3/4	4/5
Reactive Yellow 167 + Reactive Blue 21 + Reactive Blue 221	Profoks	4	4/5
	Osmosis	4	4/5

the textile wastewater passed through the Profoks/CAP reactor and the ultrafiltration and nanofiltration units. Chemical oxygen demand, color, and suspended solids, which are the main pollutants in textile wastewaters, have been removed with very high yields. Conductivity was reduced and salinity removal from wastewater at desired level was achieved. With this study, wastewater was not only treated according to discharge standards, but also water recovery was realized. Furthermore, in dyeing processes of cotton fabrics with reactive dyes, dyeing with Profoks/CAP-treated wastewater as an alternative to osmosis water showed similar performance in terms of color, levelness, and fastness compared to those dyed with fresh osmosis water.

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