*Egypt.J.Chem.* **Vol. 62**, No. 6. pp. 1025 - 1036 (2019)

**Egyptian Journal of Chemistry**
**http://ejchem.journals.ekb.eg/**

# Evaluation of Synthesized Ozone by Dielectric Barrier Discharge Plasma for Degradation of Anionic Dyes from Their Solutions

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DEGRADATION of hardly degradable textile dyes in wastewater using dielectric barrier discharge (DBD) plasma method becomes a competitive technology for primary decomposition. In the same trend, new complex Dielectric Barrier Discharge Plasma device has been used in this study to evaluate its results in degradation of textile dyes. The six dyes used are from different classes (acid, basic and reactive) and structure to cover all the bases of dye. Different parameters as exposure time, ozone and dye concentration and their effect on% efficiency (η) of color removal of dyes from their solution were investigated. The de-colorization kinetics and both chemical oxygen demand (COD), the effect of final product of the degradation process on the aquatic live is expressed using biological oxygen demand (BOD) analysis. The resultant study shows that, the maximum color removal was obtained by treating the dye solution for 90 sec using ozone concentration 20g/m$^2$ and concentration of dye 0.5g/l for all the dye. Kinetic studies were been also considered and the obtained results were following first-order reaction.

**Keywords:** Plasma, Anionic dye, BOD, COD, Kinetic studies.

## Introduction

Textile industries used a huge amount of water and the produced effluents were been filled with dyes, auxiliaries and chemicals, which are – in general- toxic and resistant to color resulting from dyes remaining in the water. This colored waste water has a great bad effect on aesthetic merit, the purity and transparency of water that body needs and receives. [1-4] In addition, it is against the environment and causes many concerns about the possible toxicity and carcinogenicity of some organic dyes. Treatment of textile dyes house effluent is necessary because of its inherent color, which is unacceptable to environmental regulatory agencies and aesthetically unpleasant. Since, removal of color is one of the main problems in the textile dyeing wastewater. Therefore, it must come up with another effective solution that has the ability to get rid and remove both strong color and toxic organic compounds from textile wastewater. [5, 6]

Different devices of progressing oxidation were applied for the decomposition of organic dyes, including ozone ($O_3$) at pH > 8.5; $O_3$ and hydrogen peroxide ($O_3 + H_2O_2$); $O_3$ and catalyst; Fenton system ($H_2O_2 + Fe^{2+}$); $O_3$ and UV; $H_2O_2$ and UV; $O_3$, $H_2O_2$ and UV; photo-Fenton system; and photocatalytic oxidation ($TiO_2$ + UV).[7]These techniques are generally based on the formation of HO radical and its devastating behavior on organic matter. Recently, the applications of plasma-construct oxidation technique on the disintegration of pollutants have been increasingly explored due to their complexity, variation and high oxidant function.

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Received 25/11/2018; Accepted 9/1/2019
DOI: 10.21608/EJCHEM.2019.6370.1536

Many plasma generation techniques have been applied such as electrohydraulic discharge, corona discharge, dielectric barrier discharge (DBD), microwave discharge, radio frequency and others. [8, 9]DBD technology is one of the most promising, because it allows for various reactor configurations, such as axial with metal–glass/quartz electrodes, [10-13] axial with glass electrodes, [14] and DBD falling liquid. [15, 16] Low temperature, atmospheric pressure plasma is very stable and relatively easy to operate. This method, depending on conditions, can produce large amounts of ozone and UV radiation. HO radical was produced directly inside the plasma and during dissolution of $O^3$ in water. Reactions of ozone and radicals with organic matter have been discussed extensively. [17] Moreover, this method does not require additional chemicals and does not produce dangerous chloro-organic by-products [18, 19] compared to chlorination. It has been reported that the products of dye degradation by ozonation revealed low to zero toxicity [16] or at least lower than the original compound. [20]

The efficacy of ozonation process for the textile industries effluents depends largely – on the dye's concentration and the dose of ozone and treatment temperature. Due to the high cost of ozone generation, it is necessary and crucial to optimize its utilization needs. This point verified largely by using dielectric barrier discharge (DBD). Based on this point, the aim of our present study is removing the colorants from wastewater with low cost using physical technique instead of chemicals, which have bad effect on the environmental.

## Experimental

*Materials*

Six commercial reactive, basic and acid dyes were selected and used without any further purification. The following table shows the dyes used with their chemical structures, which are taken from the Color Index and published literature.[21]

*Methods*

*The ozone reactor*

A cylindrical ozonize has been built with a simple design from cheap materials (Fig. 1).

*Ozone generation*

The DBD plasma is well known as a useful method for production activates radicals and excited species such as H., $O_3$, $N^{2+}$ and OH, which are generated, by dissociation and ionization of the ambient gases because of energy transfer from energetic electrons to the gas molecules. The DBD generated ozone has been applied in treatment of polluted water, decomposition of exhaust gas and surface modification.[22-24]

*Analysis & measurements*

*Chemical and Biological Oxygen Demand (COD &BOD)*

Both of the untreated and treated aqueous solutions were measured in the Micro Analytical Centre of Cairo University.

*Dye adsorption procedure*

Aqueous solutions of the selected dyes were prepared by dissolving different amounts of dyes-range from 0.01 to 4 g in l Liter of distilled water. The dye solutions were treated using DBD plasma at various conditions (current, ozone concentration and plasma exposure time). At the end of each run, the dye absorbance was evaluated calorimetrically at the maximum wavelength of each one of the six dyes used. The absorbance of the dye is measured using a double beam spectrophotometer thermoelectric corporation unicorn 300 England. Color removal efficiency is calculated according to the following equation:

$$\eta(\%) = \frac{A_o - A}{A_o} \times 100$$

where $A_o$, A is the initial and final absorbance of dyes.

*The Kinetics Analysis*

Several investigators have reported that, [25-29] the color removal curves obey the first order kinetic reaction. The rate equation of a first order reaction is represented as:

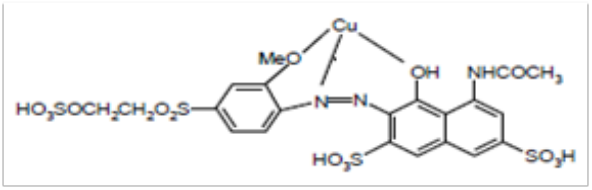
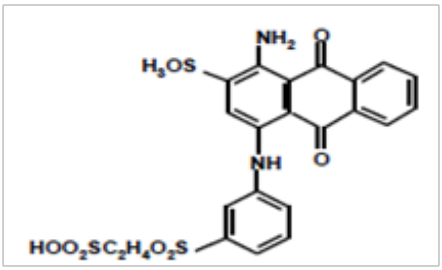
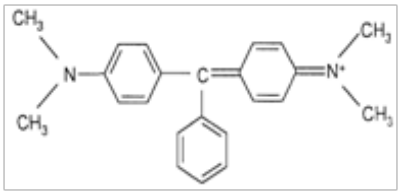
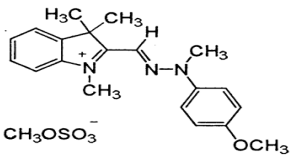
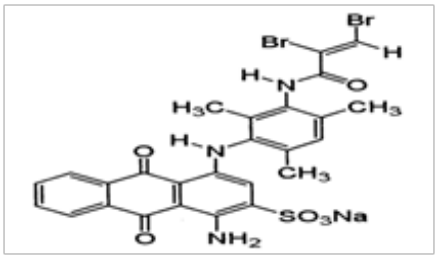
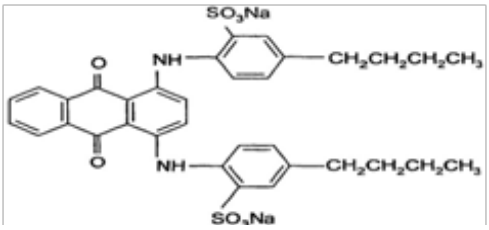
$$\frac{-d_c}{d_t} = K_c$$

where dc/dt is the rate of change of dye concentration, the negative sign indicates that concentration of dye decreases with time t, k= the rate constant ($min^{-1}$) and C= concentration of dye at time t.

Thus, the initial concentration of dye at $t_0$ is equal to $C_o$, while at some later when time= t, so, the concentration will be $C_t$.

By integrating equation (1) between times $t_0$

**TABLE 1. Color index and structure of used dyes.**

| Color Index | Structure | $\lambda_{max}$ |
|---|---|---|
| Reactive violet 5 |  | 560 |
| Reactive Blue 19 |  | 590 |
| Basic Green 4 |  | 617 |
| Basic Yellow 28 |  | 438 |
| Acid blue 225 |  | 584 |
| Acid Green 27 |  | 605 |

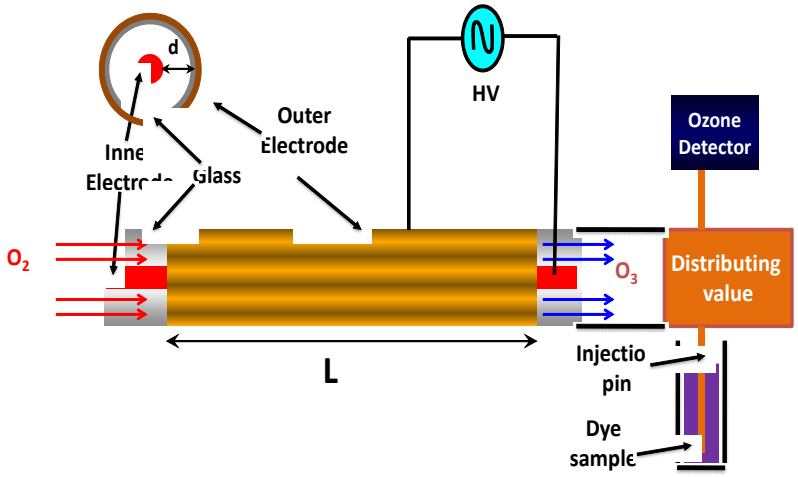



**Fig. 1. DBD- plasma ozone- generator scheme.**

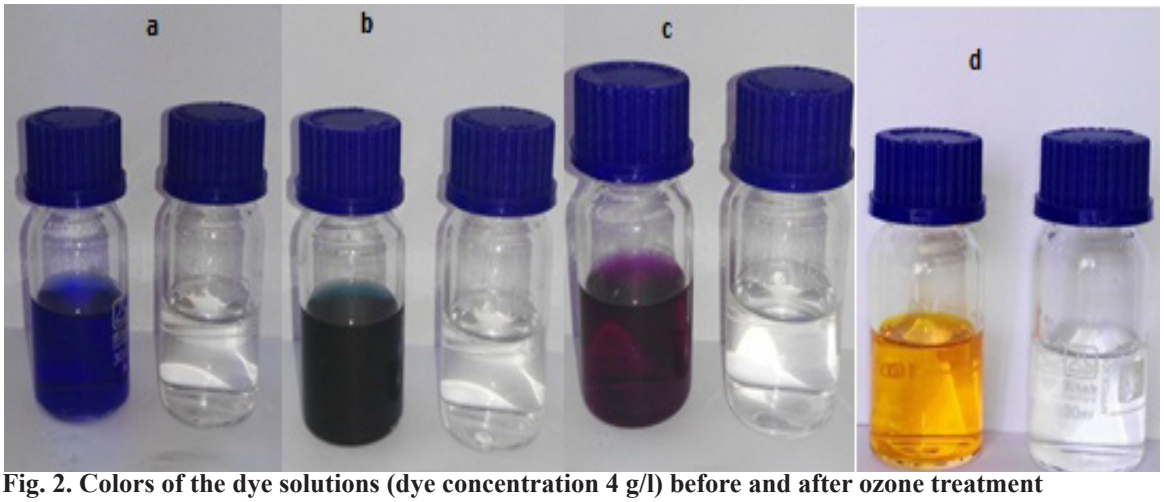



**Fig. 2. Colors of the dye solutions (dye concentration 4 g/l) before and after ozone treatment a) Acid blue 225, b) Basic Green 4, c) Reactive violet 5 & d) Basic Yellow 28.**

and $t_t$ it gives:

$$ln\frac{C_t}{C_o} = -Kt$$

thus the kinetic equation for a first order reaction is represented by

$$K = -\left(\frac{1}{t}\right)\left(\ln\frac{C_o}{C_t}\right)$$

Where $C_o$ is the initial dye concentration instant at $t_0$, $C_t$ is the dye concentration at $t_t$, k is the first order rate constant ($min^{-1}$) and t is the time of reaction in minutes.

## Results and Discussion

*General behavior of ozone generator system*

Both voltage and current wave are represented in Fig. 3. Once, the voltage 3.5 Kv is set between the gap spaces, the current flows as a filamentary current due to its formation in narrow channels that called the micro-discharge filaments. [30, 31] Whenever, the applied discharge voltage-between the two electrodes exceeds the onset voltage the electric field accelerate the free electrons- in the gap space to energies that are equal or exceed the ionization energy of the gas, thus the electron number is doubled with respect to each generation of ionization collision. The electron swarm could be moving in nanoseconds across the gap due to the high mobility of the electrons comparing to

the ions. As a result, thus slower ions, various excited and active species were left behind the electrons may undergo further chemical and physical reactions with the treated samples sited between the discharge gaps.

Once, the collect electron reverses the electrode they are spreading through the dielectric layer, inverse the positive charge on the instant anode. This factor joint the slower ions left behind and decreases the electric field in the proximity of the filament, and hence disconnects any extra ionization over the premier track in tens of nanoseconds. [32]As a result, micro-discharge filaments are created. Figure 3 declared the difference between current and voltage that is owed to the DBD cell. The operation cost of treatment process is depending therefore; the consumed power in the DBD discharge cell is the only factor that is controlling the operation cost of treatment process. Lissajous method [33]is used to measure the consumed power via this present work. Figure 3 shows the discharge current and the consumed power in the DBD cell as a function of the discharge voltage. It is clear that, always the DBD cell consumed power gives a very low value even at high voltage. Under these conditions, temperature varies between 30-40°C, while it is elevated to unrequired values at higher discharge voltages and in turns decrease the efficiency of the process treatment.

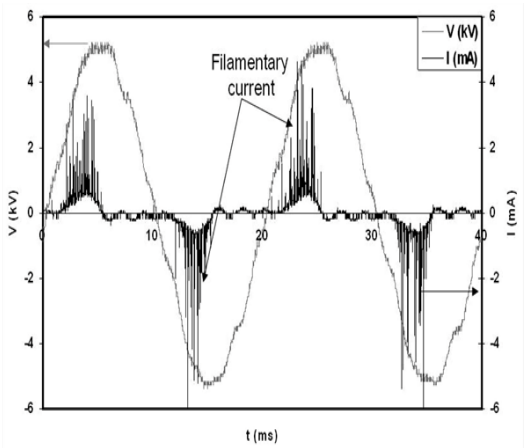



**Fig. 3. Voltage and current wave resulting from DBD.**

Figure 4 to 6 represent the general behavior of the power (P) and discharge current (I) as functions of the applied potential (V), while identifying the ozone efficiency (eff.) and its concentration as functions of the discharge power. In low temperature plasma treatments, the increase for oxygen can be formed through electron impact dissociation of oxygen, which resulted in a larger generation of ozone. $H_2O_2$. It was also produced by electron dissociating of $H_2O$ after a series of reactions. Some electrons and radicals decomposed both $O_2$ and $H_2O_2$ generated at the same time and consumed them for organic generation. Related formulas describing the generation reactions of $O_3$ and $H_2O_2$were exhibited as follows:[34-36]

$$e^- + O_2 \rightarrow e + O + O^* \quad (1)$$
$$O^* + O_2 \rightarrow O_3 \quad (2)$$
$$e^- + H_2O \rightarrow OH^* + H^* \quad (3)$$
$$OH^* + OH^* \rightarrow H_2O_2 \quad (4)$$
$$H_2O_2 + OH^* \rightarrow H_2O + HO_2^* \quad (5)$$

Figure 7 shows the relation between ozone concentration and the discharge power where, the result shows a great impact of increasing the power on the ozone formation up to 6 watts and then the concentration starts to decrease.

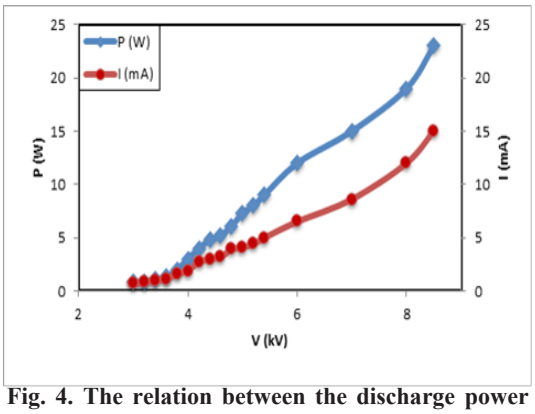



**Fig. 4. The relation between the discharge power (P) and the current (I) as a function of the discharge voltage (V)at fixed oxygen rate.**

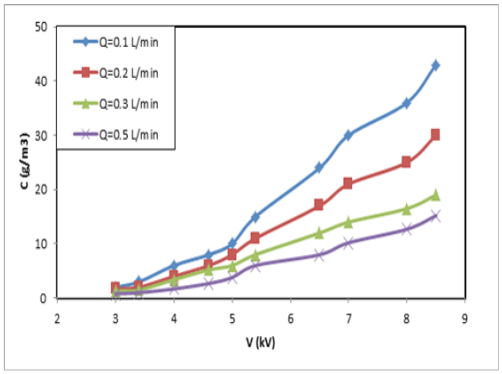



**Fig. 5. The ozone concentration as a function of the discharge voltage at different oxygen flow rates.**

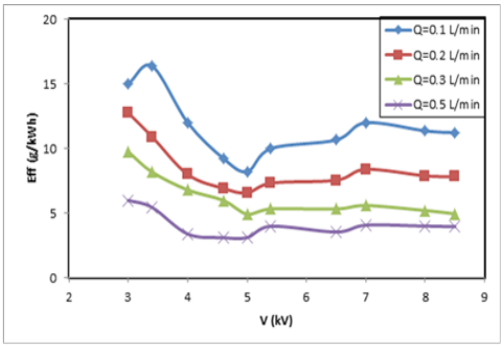



**Fig. 6. The ozone efficiency as a function of the discharge power at different oxygen flow rates.**

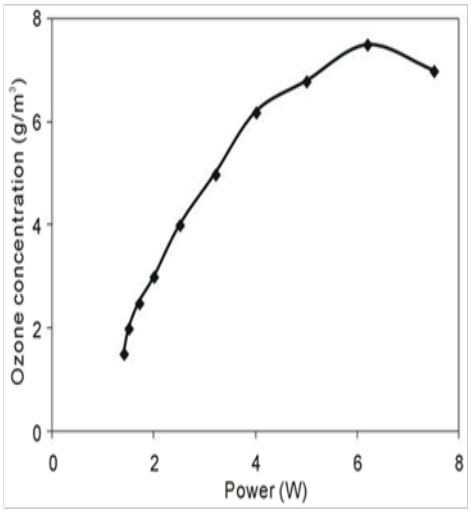



**Fig. 7. Ozone concentration in the exhaust gas of the DBD cell as a function of the discharge power.**

*Effect of plasma exposure time*

The relation between plasma treatment time and the (η) efficiency % of color removal for the six dyes- at ozone concentration 5g/m$^2$ and fixed oxygen flow rate 0.2 l/min is represented by Fig. 8. It is cleared that, as plasma treatment time increases, the (η) % of color removal increases, regardless the type of dyes. The optimum plasma exposure time is 90 sec that gives the highest efficiency (η) of color removal for dyes RV5, RB19, AB & AG while 120 sec is more efficient for dyes BG and BY. After these times, the η % of color removal becomes constant. These differences of dyes behaviors return to its configurations, structures and groups attached.

*Effect of ozone concentration*

Ozone plays a great role on dyes removal and this holds true for all dyes used in this work. It is clear from Fig. 9 that, as ozone concentration increases, the (η) % of dye removal is improved and gained high results. Where a great leap happened from 5-10 mg/m$^2$ reaching the maximum dye removal at 10 mg/m$^2$, the ozone concentration was 99.9, 94.5, 90.5, 88.1, 85.9 and 84.1 % for dyes: acid blue, acid green, reactive violet, reactive blue, basic green and basic yellow respectively. With the increase of ozone concentration, more active spices ($OH^*$, $H_2O_2$, $O_3$, $O^*$) in the reactor attacked the dyes molecules reflecting in higher results of the (η) % of color removal. So, the increase in ozone concentration more than this limit and up to 20 mg/m$^2$ caused no more effect on dye removal while, caused loss of time and effort.

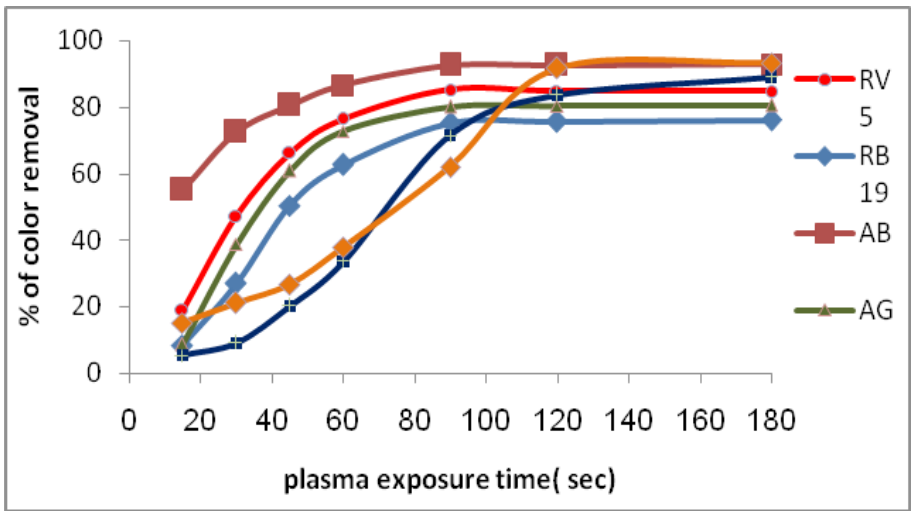



**Fig. 8. The effect of plasma exposure time on the percent of color removal for the six dyes.**

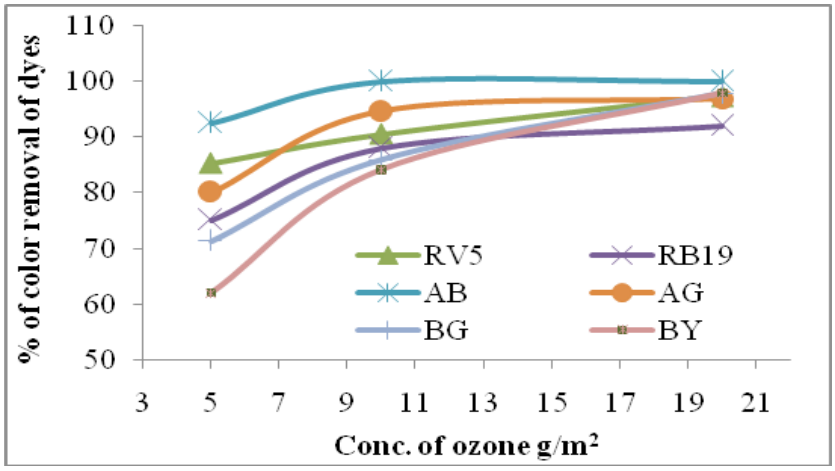



**Fig. 9. The effect of ozone concentration on the % of color removal for the six dyes.**

*Effect of dyes concentration*

The effect of dye concentration on the (η) % of color removal is represented by Fig. 10. As expected at high dye concentrations will obtain the lowest (η) % results. Although this fact, there is-still- a reasonable (η) % of color removal ranges from 45-65% for primary degradation process at highest concentration of dyes used (4 mg/l) at the same time lower concentration (<0.5) the (η) % of color removal reached 99% for all dyes.

Similar work has established using A pilot DBD plasma semi-continuously operated reactor at rate flow=14.5 l/min, AC high voltage power supply from 30 to 50 kv, AC frequency was set to 8000 HZ. [37]The yield of ozone production ranged between 0.19 and 0.46 mg/s by varying the discharge power between 3 and 33 W. The demand of energy to decompose separate dyes ranged from 18.7 to 866 kJ/g. 10 of 13 dyes were decomposed up to 95% during 300 s of reactor operation. While in this study the rate of flow=0.2 l/min, AC power supply high voltage transformer (0-20Kv), AC frequency was set to 50 Hz this gave concentration of ozone is 20 g/m$^2$. In this research we have been treated with high concentrations of dye as we have reached 4 grams per liter at 90 sec. This comparison ensures the success of the new complex plasma device and its ability to be established on pilot scale.

*Ozone effect on biodegradability (BOD) &chemical oxygen demand (COD)*

Table 2 indicates the performance of ozonation on the reduction of COD and the investigation of the change in biodegradability (BOD) of the effluent during the treatment process. In addition, it represents the results of the % dye removal obtained at the optimum conditions of ozonation; treatment time, 90 sec., ozone concentration, 20 mg/m$^2$and dye concentration is 0.5g/l. The results show that COD removal was enhanced partially not completely from the effluent of the three tested dyes where, complete degradation of the dye might need more time compared to the time needed for color removal. [38-40] Ozonation of the effluent was more effective for decoloration than for COD degradation where, it gives high efficiency of color removal as Table 2 cleared. Moreover, the BOD value was tripled after the treatment indicating enhanced biodegradability of the effluent after ozonation process. The increase in the BOD indicates to the decomposition of ozone- resistance substances and producing more biodegradable compounds. [39] Earlier reports presented mixed results from the toxicity tests of dye decomposition products by DBD ozonation. Dojcinovic et al. and Arslan-Alaton et al. in this perspective, [13, 17] the results obtained in this study are promising, keeping in mind the differences in model pollutants, concentration ranges and methods for toxicity testing.

*Kinetics of Decolorization*

In this part, three dyes were selected(RB19, AB225 and BY27). to study their kinetics in the decolorization process. The rate constants k for these three dyes at the optimum treatment time of ozonation 90 sec., where the maximum (η) % of dye removal has been obtained.

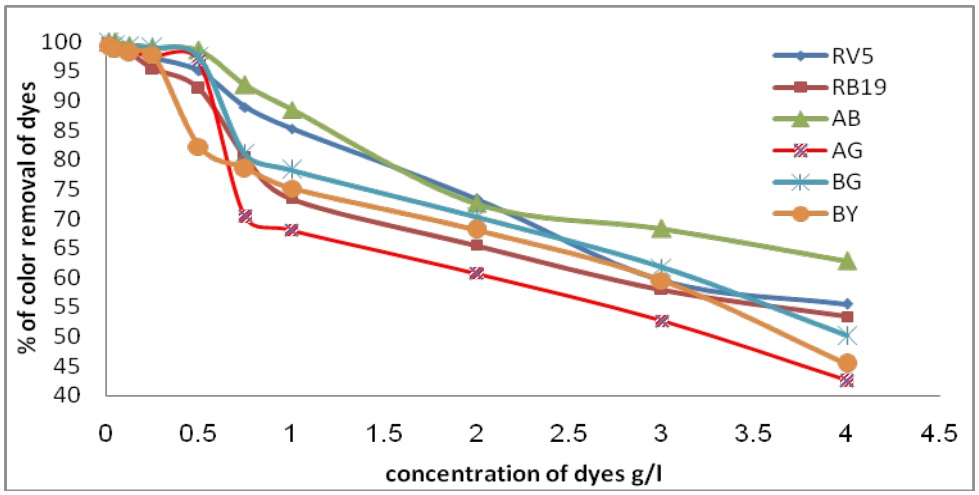



**Fig. 10. The effect of dye concentration on the % of color removal for the six dyes.**

**TABLE 2. Biological and chemical oxygen demand (BOD and COD) for untreated and treated dyes.**

| Dyes | Biological Oxygen Demand (BOD) | | Chemical Oxygen Demand (COD) | | % of color removal |
|---|---|---|---|---|---|
| | untreated | Treated | untreated | Treated | |
| Acid blue 225 | Less than 0.5 | 1.8 | 124 | Less than 100 | 99.99 |
| Basic green 27 | 0.5 | 1.24 | 131 | Less than 100 | 97.8 |
| Reactive violet 5 | 0.6 | 1.90 | 126 | Less than 100 | 97.9 |

Figure 11 (a, b and c) represent the relation between the reaction constant k ($min^{-1}$) and the dye concentration for the dyes RB, AB and BY respectively. It is clear from the figure that, the value of reaction rate constant k ($min^{-1}$) is varying significantly with dyes concentrations for all the classes. For reactive blue, it has the values 0.008, 0.041and0.053$min^{-1}$for concentrations 0.01, 0.25 and1g/ml, while for acid blue it was 0.0042, 0.048, 0.052 $min^{-1}$and for basic yellow it was 0.027,0.058,0.066 $min^{-1}$ at the same dye concentrations. This is expected where; increasing the dye concentration must enhance the possibility of collision between dye molecules and ozone, leading to an increase in the de - colorization reaction rate. The maximum rate constant is obtained at concentration of dye 0.5g/l for AB and BY while for RB19 the maximum rate constant was at 1g and more than this limit, a slightly constant result is obtained.

Previous investigations [40] have reported that, the de-colorization process mainly follows the first – order kinetics. The corresponding first order correlation is shown in Fig. 12 that illustrates the typical plot of linear regression (ln $C_t/C_0$) versus time of color removal and the relation is typical a pseudo first-order reaction plot.

The correlation coefficients that can explain the fitting extend of the function equation & experimental data is represented by $R_2$. For all classes of dyes, the values of these $R_2$ are almost greater than 0.9, which confirms the goodness of the assumed kinetics for de-colorization process. It is also obvious from the results (Fig. 12) that the de-colorization rates are increased slightly with the time of the treatment varies starting from 15 to 90 sec.

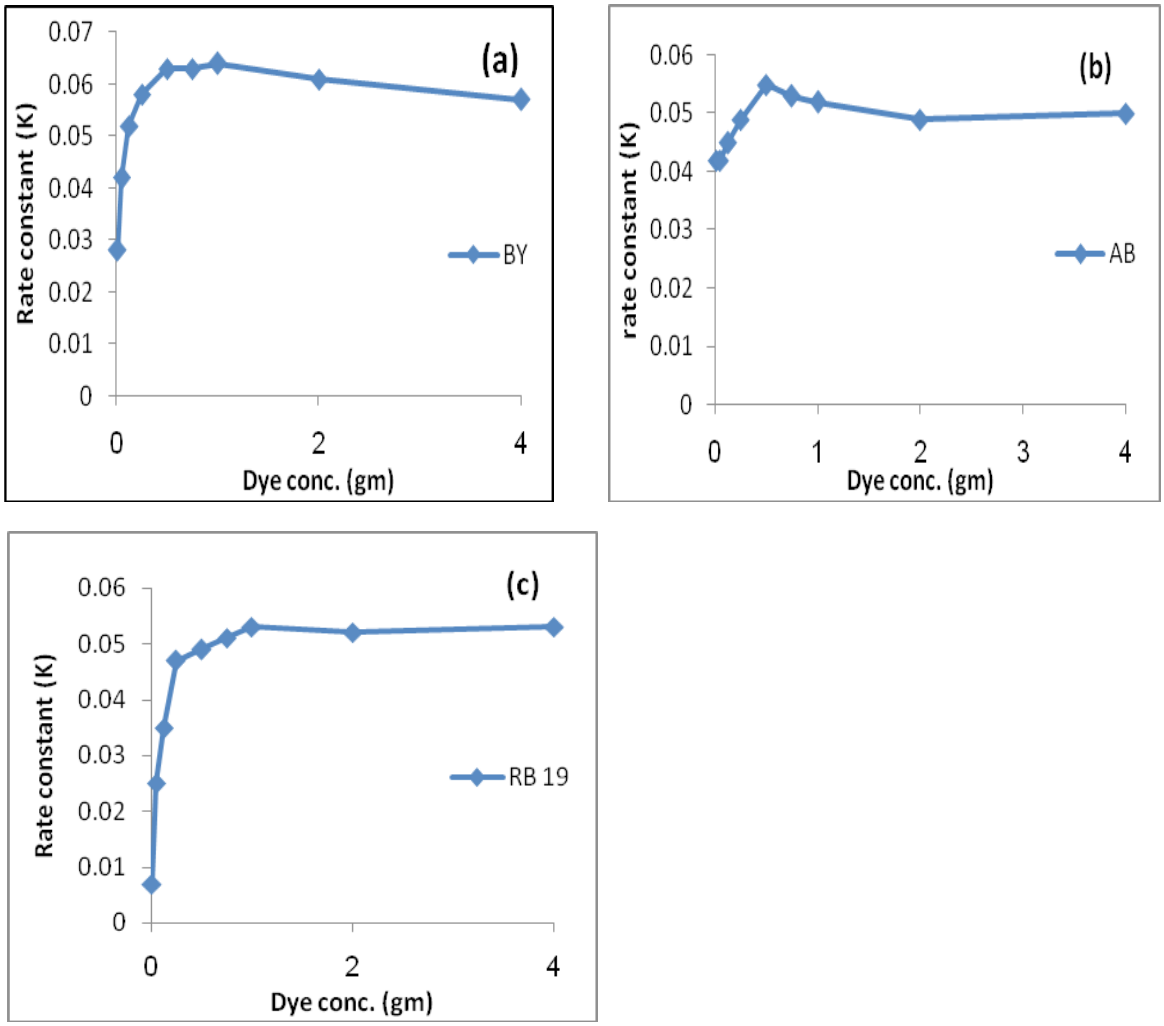



**Fig. 11. Effect of concentration of dyes on rate constant k.**

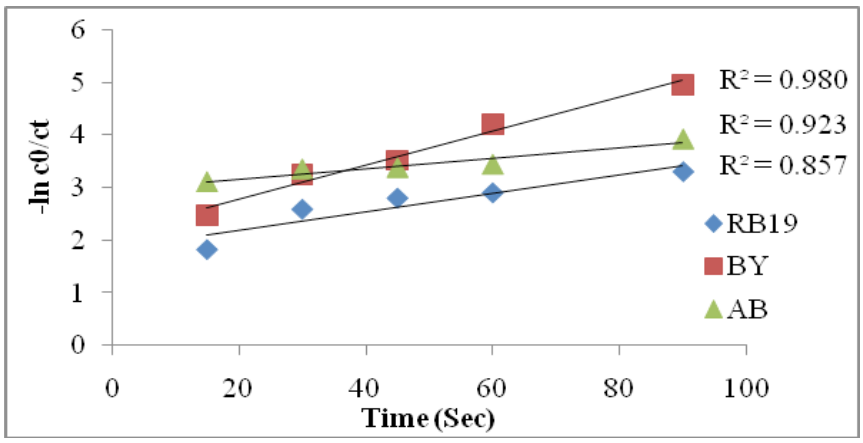



**Fig. 12. Decolorization kinetics of RB19, BY&AB dyes solution at different plasma exposure time (sec).**

## Conclusion

The new manufactured device has proved its efficiency in color removal and degradation of the six anionic at lower discharge power and exposure. Ozone concentration plays a great role on the (η) % of dyes removal for all the six dyes used in this work. The degradation of some dyes reached up to 95—99% at ozonation time 90 sec. while others needed more time up to 120 sec to achieve higher results. As plasma treatment, time increases, the (η) % of color removal increases- regardless the type of dye. The optimum time for plasma exposure is 90 sec that gives the highest efficiency (η) of color removal for dyes RV5, RB19, AB & AG while 120 sec is more efficient for dyes BG and BY. The results show that COD removal was enhanced partially not completely removed from the effluent for the three dyes; AB, BG & RV where, complete degradation of the dye might need more time than color removal. The values of the biological oxygen demand (BOD) was increased about three times compared to its values before the treatment, indicating enhanced biodegradability of the effluent after ozonation process. The relation between ($\ln C_t/C_o$) and exposure time is linear as the 1st order reaction and the values of reaction rate constant k ($min^{-1}$) varies depending on dye concentration and the class of dye; whether it is reactive, basic or acid dyes.

## Acknowledgment

The authors wish to express their great thanks to National Research Centre, Egypt for giving them the chance to complete this work and to have some new experiences.

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## استخدام غاز الاوزون الناتج من جهاز بلازما التفريغ الكهربائى فى تكسير الصبغات الابونيه فى محاليلها

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يهدف هذا البحث الى استخدام تقنية البلازما فى انتاج غتز الوزون واستخدامه فى تكسير الصبغات التى يصعب تكسيرها بالطرق العادية لازالة لونها. تم تعريض ست صبغات مختلفة الانواع (صبغات حمصية – صبغات قاعدية - صبغات نشطة) لبلازما التفريغ الكهربائى باستخدام عوامل مختلفة مثل تغيير زمن التعرض للبلازما، تغيير تركيز الاوزن و تركيز الصبغات المستخدمة مع دراسة تأثير هذه العوامل فى كفاءة ازالة لون الصبغات من محاليلها (η). كما تم دراسة تفاعلات ازالة اللون الحركية و كل من (BOD)، و (COD)، .

أوضحت النتائج أن أعلى درجة ازالة للون تمت بمعالجة محلول الصبغة (تركيزنصف جرام لكل لتر) لمدة 90 ثانية و تركيز الاوزن ( 20g/m) لكل انواع الصبغات. كما أوضحت الدراسات الحركية أن هذه التفاعلات من الرتبة الاولى.