Journal of Applied Science

Biannual Peer Reviewed Journal Issued by Research and Consultation Center , Sabratha University

Issue (12)
April 2024

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Editorial

We start this pioneering work, which do not seek perfection as much as aiming to provide a scientific window that opens a wide area for all the distinctive pens, both in the University of Sabratha or in other universities and research centers. This emerging scientific journal seeks to be a strong link to publish and disseminate the contributions of researchers and specialists in the fields of applied science from the results of their scientific research, to find their way to every interested reader, to share ideas, and to refine the hidden scientific talent, which is rich in educational institutions. No wonder that science is found only to be disseminated, to be heard, to be understood clearly in every time and place, and to extend the benefits of its applications to all, which is the main role of the University and its scholars and specialists. In this regard, the idea of issuing this scientific journal was the publication of the results of scientific research in the fields of applied science from medicine, engineering and basic sciences, and to be another building block of Sabratha University, which is distinguished among its peers from the old universities.

As the first issue of this journal, which is marked by the Journal of Applied Science, the editorial board considered it to be distinguished in content, format, text and appearance, in a manner worthy of all the level of its distinguished authors and readers.

In conclusion, we would like to thank all those who contributed to bring out this effort to the public. Those who lit a candle in the way of science which is paved by humans since the dawn of creation with their ambitions, sacrifices and struggle in order to reach the truth transmitted by God in the universe. Hence, no other means for the humankind to reach any goals except through research, inquiry, reasoning and comparison.

Editorial Committee

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Journal Address:

Center for Research and Consultations, Sabratha University

Website: **https://jas.sabu.edu.ly/index.php/asjsu**

Email**:** **jas@sabu.edu.ly**

Local Registration No. (435/2018)

ISSN 💻 2708-7301

ISSN 📖 2708-7298

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All submitted research manuscripts must follow the following pattern:

- Title, max. 120 characters.
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- Keywords, max. 5 words.
- Introduction.
- Methodology.
- Results and Discussion.
- Conclusion.
- Acknowledgments (optional).
- References.

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The Editorial Committee invites all researchers "Lectures, Students, Engineers at Industrial Fields" to submit their research work to be published in the Journal. The main fields targeted by the Journal are:

- Basic Science.
- Medical Science & Technology.
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Refereeing

The Editorial Committee delivers researches to two specialized referees, in case of different opinions of arbitrators the research will be delivered to a third referee.

Editorial Committee

CONTENTS

DEGRADATION OF REACTIVE BLACK 5 DYE IN WATER FALLING FILM DIELECTRIC BARRIER DISCHARGE REACTOR (DBD)

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Abstract

Reactive Black 5 (RB5) is one of the synthetic reactive dyes frequent used in the textile industry, due to its solubility in water and reactive groups which form covalent bonds within the fibre.

The aim of this study was to investigate the degradation of the Reactive Black 5 dye by using an advanced oxidation process in a non-thermal plasma reactor based on water falling film dielectric barrier discharge (DBD).

In the past decades, an increasing number of procedures have been reported to remove pollutants from wastewater. An example of these is what is known as the Advanced Oxidation Processes (AOPs), which are employed for textile wastewater treatment. These (AOPs) are environmentally friendly methods based on chemical, photochemical or photocatalytic production of hydroxyl radical (HO•). This powerful oxidizer can react with most organic compound found in wastewater, such as dyes.

The effects of different initial pH (9, 7 and 5) of the dye solutions on the degradation during subsequent recycling of the Reactive Black 5 dye solution through the DBD reactor. Change of pH values and the effect of pH adjustments of the dye solution after each recycling on colour removal were also tested. It was found that the initial pH of the dye solutions and the pH adjustments of the dye solution after each recycling process did not affect the colour removal.

Decolonization of the Reactive Black 5 was studied using advanced oxidation processes (AOPs) in a non-thermal plasma reactor based on the coaxial water (DBD) film. The initial dye concentration in the solution was 40 mg /L.

The dye solution was recycled through a DBD reactor with an applied energy density of 45-315 kJ/ L. The effect of residence time was studied after 5 minutes and 24 hours of plasma treatment.

Changes in the pH values of the dye solution were tested after each recycling process. The decolourization and degradation effectiveness of high concentration dye Reactive

Black 5 (40, 80, 200 and 500 mg/L) was examined. Efficiency degradation and possible degradation pathways were monitored by measuring the COD value.

Experimental results confirmed the effectiveness of the procedure in the Reactive Black 5 degradation. The final degradation products did not cause any significant toxic effects to Artemia salina.

Keywords: Reactive Black 5; degradation; DBD; water treatment; advanced oxidation processes.

Introduction

The textile industry is considered one of the most important industries in the world. The global textile and apparel business in 2017 is estimated to be worth US $ 4.395 trillion. The current global apparel market is estimated at approximately 1.15 trillion US $, which constitutes approximately 1.8% of the world Gross Domestic product (GDP) (Textile Mates., 2018). Wastewater generated from textile industry is generally coloured and non-biodegradable with high BOD and COD due to the high concentration of dyes (Handoko et al., 2022).

Textile wastewater is difficult to treat by any conventional methods because it is non-biodegradable and soluble in water. Wet textile processing requires significant water consumption resulting in the generation of a large amount of effluent (Hussain et al., 2018; Handoko et al., 2022).

Reactive dyes are widely used in the textile industry to colour cellulosic fibres. Their reactive groups are able to form covalent bonds with hydroxyl groups on the fibre. A strong covalent bond would be expected but the efficiency of the dye-fibre reaction can change from 90 to 50% as found by (Esteves et al., 2007).

Azo dyes are the most common leachates in textile mills, accounting for 70% of their aqueous waste (Chong et al., 2007) due to its stability and high chemical resistance. Reactive Black 5 makes up 50% of the azo dyes used in the textile industry. However, these properties make it a toxic compound that is resistant to natural aquifers (Nabil et al., 2014).

Synthetic dyes are aromatic compounds with structural diversity, high chemical stability, and resistance to microbial attack. Approximately 8 × 105 tons per year of synthetic dyes are produced worldwide, with over 100,000 dyes commercially available, which are widely used in many industries, including the textile, cosmetics, pharmaceutical and food industries. Consequently, these industries produce large amounts of wastewater with high concentrations of synthetic dyes (Brillas et al., 2015). Large amounts of drinking water (150 L/ kg of textile material) are consumed in the textile industry, due to the inefficiency of the dyeing process. There are many studies relating synthetic dyes with cases of toxicity, carcinogenesis, mutations and teratogenicity in humans (Rafatullah et al., 2010).

Dying using reactive dyes with concentrations at about 1.5 g/L requires a large amount of salts, at about 5 – 6% of sodium chloride and sodium sulphate in alkaline solutions (Ogumka, C., 2020).

The chemical nature of synthetic dyes gives them resistance to degradation by conventional methods, including precipitation adsorption oxidation with sodium hypochlorite, filtration, coagulation, flocculation, and biodegradation (Martínez et al., 2012). Therefore advanced oxidation processes (AOPs) have emerged as an alternative to synthetic dyes.

The mechanism of advanced oxidation processes (AOPs) is based on the formation of transient species with high oxidation capacity, mainly hydroxyl radicals OH which has a very high effect on the oxidation of organic substances and are ideal for the treatment of unconventional compounds, i.e. synthetic dyes from industrial wastewater (Gómez et al., 2000).

Reactive dyes are very recalcitrant to conventional waste treatment processes, which although they account for approximately 80% of the dyes are reduced, and leave most of them absorbed into the sludge (Kunz et al., 2002).

This study presents experimental results on the Reactive Black 5 degradation by using advanced oxidation process (AOP) in a non-thermal plasma reactor based on coaxial dielectric barrier discharge with water falling film (DBD).

Materials and Methods

The Reactive Black 5 dye was purchased from Clariant (Switzerland). Reactive Black 5 is a dissociated anionic sulfonate in aqueous solution. UV-VIS spectrum of Reactive Black 5 in aqueous solution was obtained in order to check the wavelength at which maximum absorbance is (λ_{max} = 590 nm). The reason for this is that absorption at this wavelength is less sensitive to changes in pH value that occur during the colour oxidation process (Gibbs et al. 2004).

UV-VIS Characterization of reactive dye Reactive Black 5 was performed using spectrophotometric method of analysis and wavelength that show maximum of absorption of aqueous. Solution was checked λ_{max}, and dye concentration was 40 mg/L.

Artemia salina cysts (Artemia-mix) and sea salt are products of Sera Germany. Artemia- mix contains lyophilized cysts and food. Synthetic seawater was made by dissolving 100 g of sea salt in 3 L of deionized water.

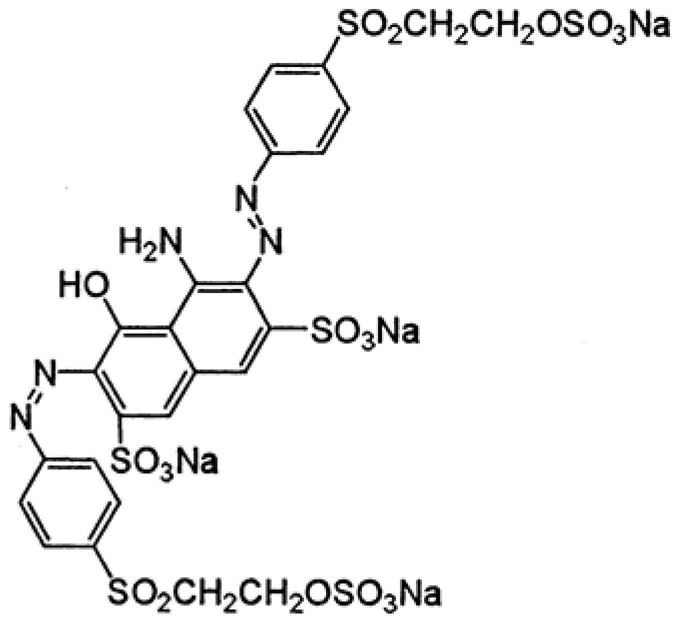

Figure (1): Molecular Structure of the Reactive Black 5 dye: Molecular Weight = 991.8 g/mol.

DBD Reactor

The degradation of Reactive Black 5 dye using a coaxial dielectric barrier discharge (DBD) was designed as an atmospheric non-thermal plasma reactor for the treatment of various water solutions. In this reactor water forms a falling film that is direct contact with the plasma. A reactor of this design successfully removed both phenol and pesticides from water (Jović et al, 2013). The coaxial DBD is a source of a wide range of reactive species created in both gas and liquid phase. This discharge is able to produce O_3, H_2O_2, OH and other active species (Jović et al., 2013).

A cylindrical reactor was prepared with Pyrex tubes that had an internal diameter of 27 mm and length of 600 mm. The outer electrodes were made with aluminium foil, which was sealed on an outer glass tube with 400 mm in length. The inner electrode was a glass cylinder with a diameter of 20 mm that was silver-plated on the inside (Jović et al., 2013).

A solution of Reactive Black 5 dye flows up through a vertical hollow cylindrical electrode and flows down, thus making a thin dielectric film over the inner electrode. A barrier discharge is generated in air within a 3.5 mm gap between the dielectric and the water layer by applying a sinusoidal voltage of 17 kV on the peak. To increase the total flow of the treated solution, three discharges are connected in parallel. The plug in power for this system of discharges was 150 W.

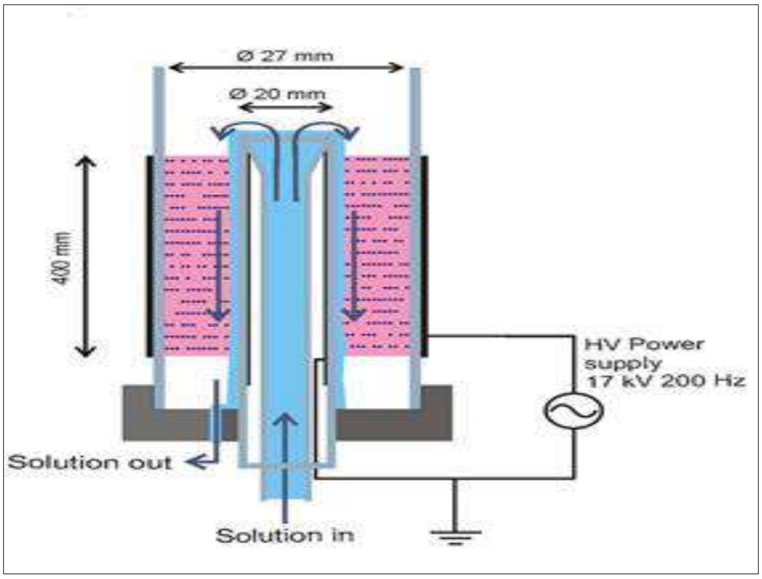

Figure (2): Water Falling Film Dielectric Barrier Discharge DBD Reactor Used in Experiments.

The thin layer of water is in direct contact with the plasma, and oxidative species are transferred from the plasma into the liquid phase, where the reactions with the pollutants occur, which is of great importance, especially for short-living active species. To increase the total flow of the treated solution, three discharges are connected in parallel.

An energy density (ED) of ~ 45 kJ L-1 was introduced into the solution with one pass through the reactor. Applied energy density was increased by recirculating the solution up to the point when reached 450 KJ/ L.

In all experiments, the treatment was started with 2 L of the surfactant solutions at a concentration of 100 mg/L. Furthermore it should also be noted that in all these experiments the solution of the azo dye Reactive Black 5 was prepared by dissolving commercial dye in demonized water without any further purification or pH adjustment. In all cases samples of dye water samples containing different concentrations of dye (40.0 mg/L – 1000 mg/L) were passed through the discharge from one to seven times.

The efficiency of degradation is defined as a percentage decrease of absorbance of surfactant derivatives according to Eq:

$$\boldsymbol{Decolorization}\ (\%) = \frac{\boldsymbol{A_0 - A}}{\boldsymbol{A_0}} \times \mathbf{100}$$

Where A0 presents the absorbance at the wavelength which shows the maximum absorption (λ_{max}) of the initial dye solution, while A presents the absorbance at (λ_{max}) of the dye solution after the plasma treatment. The absorbance measurements were performed using a UV–Visible Cintra 6 spectrometer (GBC Scientific Equipment, Australia) 5 min and 24 h after the plasma treatment.

All measurements of pH value were done using a pH meter (Microcomputer pH-vision 6071, JENCO Electronics Ltd. Taiwan) with combined electrode type HI 1131 (Hanna Instruments).

The Effect of the Initial pH Value of the Dye Solution on Degradation Efficiency

The effect of the initial pH value of the dynamic colour dye solution was determined for the active ingredient dye to remove reactive black 5. The initial dye concentration was 40mg/L. The initial pH value was adjusted to three different values 9, 7 and 5 by adding sodium hydroxide (0.05 M). Dynamic degradation measurements were performed for 5 min and 24 h for each of the seven areas of dye solutions through the reactor. UV-VIS spectra were obtained through these tests the pH value of each were determined.

Determine Chemical Oxygen Demand (COD)

Chemical oxygen demand was measured for all initial reactive black 5 samples in which the initial dye concentrations were 40, 80, 200 and 500 mg/L. The COD value was determined for plasma-treated samples with applied energy densities of 90, 135 and 450 kJ/L for each of the initial concentrations except 40 mg/L.

Toxicity Test (*Artemia Aquarium Test Organisms*)

To test acute toxicity, Artemia salina cysts in brine shrimp were hatched in artificial seawater at a salinity of 32 ± 0.5 % that was vigorously aerated from the bottom with an aerobic system.

Toxicology testing was performed using Reactive Black 5 solution at a concentration of 200 mg/L. The toxicity of the initial solution as well as of the solutions in which the decolonization rate reached 50% and 90% was examined after 24 hours of plasma treatment. Toxicology testing was performed on these samples ten days after treatment.

Results and Discussion

In the present study, the decolorization of the commercial reactive azo dye C.I. Reactive Black 5 was studied using an advanced oxidation process (AOP) in a non-thermal plasma reactor based on coaxial dielectric barrier discharge (DBD). In this reactor, water forms a falling film that is in direct contact with the plasma. As water is constantly flowing over the top of the reactor, thin water film constantly regenerates, i.e. reactor works on the principle of flow reactor.

Removal of Reactive Black 5 dye from solutions using electrical discharge are increasingly investigated in order of environmental protection (Magureanu et al., 2007).

The goal of the experimental work was to determine the influence of different starting parameters as pH value high concentrations and time on efficiency of decolorization process. Degradation efficiency and possible degradation pathways were tracked by determining the COD value and using the ion chromatography. Initial toxcicity and toxicity of the solutions after the treatment were examined by Artemia Salina test organisms.

(Magureanu et al., 2008) used the most structurally similar reactor for dye decolorization as the reactor used in this work. Both reactors work on the dielectric barrier discharge principle have a coaxial electrode geometry, and the treated solution was in the form of a thin layer and continuously poured over the inner electrode.

Effect of Initial pH Value on Decomposition Efficiency

In order to optimize the parameters affecting the degradation process, the effect of the number of solution recirculations, i.e., the energy density and initial pH value of the color solution, was investigated. Figure (3) illustrates dependence between the percentage of decolorization and the applied energy density 9,7,5 pH values.

The initial pH value does not significantly affect the dependency curve between the decolorization ratio and the applied energy density. In all cases, colour removal after the first treatment measured 5 minutes after recycling through the DBD reactor was 40%, while the colour removal after 24 hours increased to 60%.

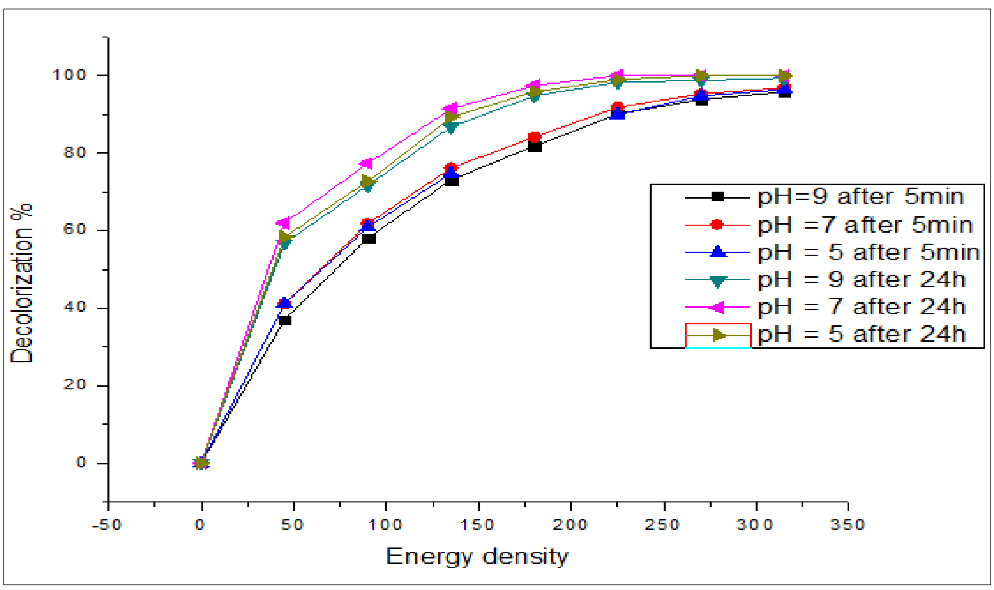

Figure (3): Effect of Initial pH Value on Decolorization Efficiency of Reactive Black 5 in DBD.

Ozone and hydrogen peroxide are the only oxidation species that are generated inside the plasma, and these are stable enough to react with dye molecules outside the plasma reactor, i.e. after the treatment.

Examination of the Degradation Efficiency of High Concentrations of Reactive Black 5 Dye

The effect of high concentrations of Reactive Black 5 on the decolorization efficiency was examined in function of the applied energy density using different concentrations of Reactive Black 5. Colour removal efficiency was measured after 5 minutes and 24 hours of plasma treatment as shown in Figure (4).

Based on the results of these experiments, it can be noted that the colour removal efficiency decreases when the concentration increases. Experimental results prove a greater decrease for lower concentrations (up to 80 mg/L), while the decrease for concentrations 80 -500 mg/L were almost linear.

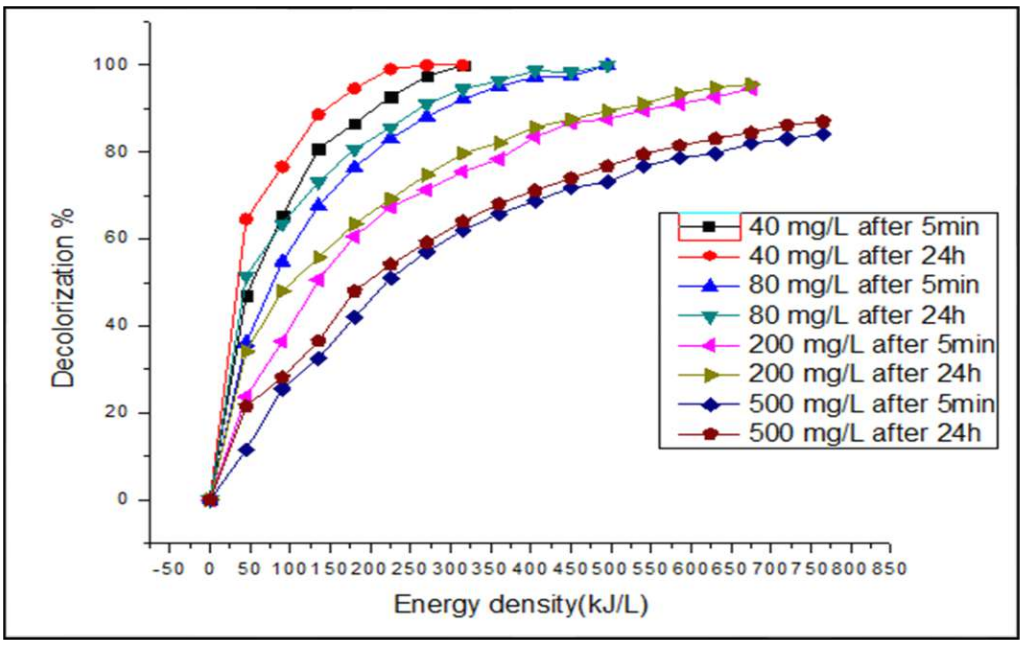

Figure (4): Change of Decolorization Percentage of the Reactive Black 5 (c0= 40 - 500 mg/L)

Determine Chemical Oxygen Demand (COD)

Organic matter is oxidized by using potassium dichromate in an acidic environment at high temperature and the organic matter is oxidized to carbon dioxide and water. Also, under these conditions, inorganic compounds are also oxidized such as Fe^{2+} to Fe^{3+}. The COD value of the primary reactive dye (O kJ/L) Reactive Black 5 (80, 200 and 500 mg/L) and solutions was measured after DBD treatment with applied energy densities of 90, 135 and 450 kJ/L. The COD value of plasma-treated samples was measured 24 hours after treatment.

Measured COD values are show in histogram Figure (5).

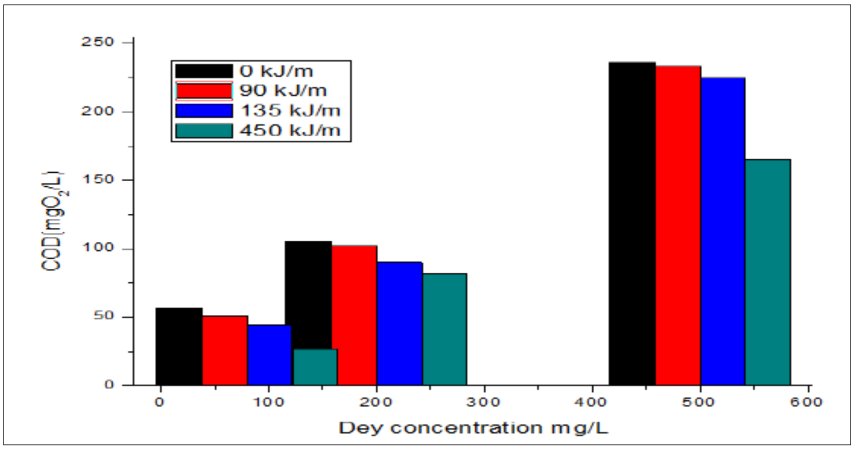

Figure (5): COD Values of Reactive Dye Reactive Black 5 (c0= 80, 200 and 500 mg/L) before and after the Plasma Treatment.

Determination of Sulfate by Ion Chromatography (IC)

Ion chromatography (IC) was used for the quantitative and qualitative determination of inorganic and organic anions of the primary reactive dye Reactive Black 5 solutions of 40, 80, 200 and 500 mg/L. Also, ion chromatography of the anions of plasma-treated dye samples was performed at applied energy densities of 90, 135 and 450 kJ/L. The measurement was performed 24 hours after plasma treatment. Qualitative and quantitative analysis was performed for sulfate.

The sulfate concentration increases via increasing initial dye concentration with the same applied energy intensity. Also, as the applied energy density increases from 90 to 450 kJ/L, the sulfate concentration increases. Figure (6) shows the change in sulfate concentration as a function of the concentration of reactive dye Black 5 after plasma treatment and applied energy densities of 90, 135, and 450 kJ/L.

For initial dye concentration of 500 mg/L, with applied energy densities of 90 kJ/L, the measured concentraton of sulfate was 62.667, with energy density 135kJ/L, the measured concentraton of sulfate was 74.987 while energy density of 450 kJ/L, the measured concentration of sulfate was 86.79 mg/L.

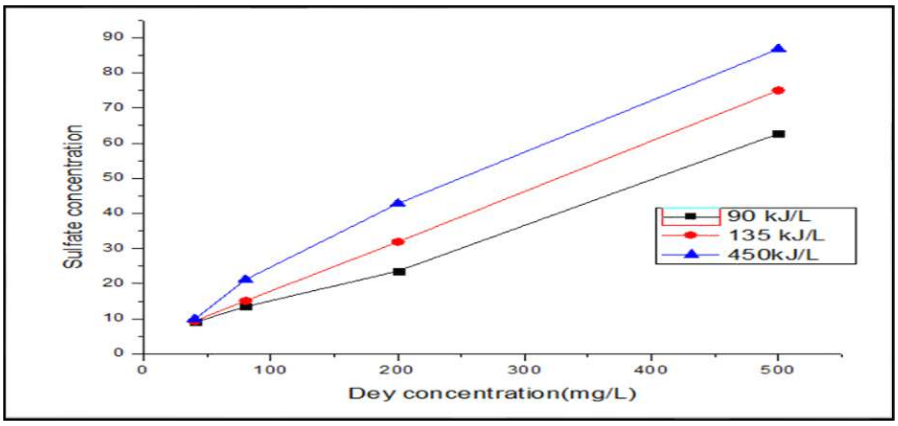

Figure (6): Change of Sulfate Concentration in Reactive Dye Function Reactive Black 5 Concentration after Plasma Treatment with Applied Energy Density of 90, 135 and 495 kJ/L.

The Toxicity Test (*Artemia Salina Test Organisms*)

The toxicity screening test, cysts of *Artemia salina* were hatched and reared in synthetic seawater at 32 ± 0.5‰ (Reef Salt, Aqua Medic, Germany) (ISO 11348-3:2007).

The toxicity of Reactive Black 5, which was performed using *Artemia salina* test organisms, is shown in Table (1). The toxicity of the dye is given as a percentage of mortality of the test organisms, *Artemia salina*, before plasma treatment for the dye concentration tested (50 and 100 mg/L). Toxicological tests, performed using *Artemia salina* test organisms, showed that the toxicity of the Reactive Black 5 dye solution was reduced or maintained at the same level after treatment but for the case of 50 mg/L dye concentration with 50% and 90% of decolonization where toxicity decreased to 0% of mortality.

Table (1): Mortality Rate (%) of *Artemia Salina* Test Organisms in Dye Solutions.

<table>
<tr><th rowspan="4">Dye</th><th rowspan="4">Dye Concentration (mg/L)</th><th colspan="3">Mortality (%)</th></tr>
<tr><th rowspan="3">Before Treatment</th><th colspan="2">After Treatment</th></tr>
<tr><th colspan="2">Decolonization</th></tr>
<tr><th>50 %</th><th>90 %</th></tr>
<tr><td rowspan="2">Reactive Black 5</td><td>100</td><td>5</td><td>2</td><td>5</td></tr>
<tr><td>50</td><td>3</td><td>0</td><td>0</td></tr>
</table>

Conclusion

In the present paper, the effectiveness of decolourization of aqueous reactive textile dyes Reactive Black 5 was tested. The test was performed by using a plasma reactor that works on the principle of dielectric barrier discharge with a thin water film of the solution being treated (dielectric barrier discharge with water falling film, DBD).

The influence of the initial pH value (pH= 9, 7 and 5) of the dye solution on the effectiveness of decolonization during plasma treatment (45-315 kJ/L) was examined. It was confirmed that the initial pH value, basic, neutral and acidic, has no influence on the kinetics of decolourization of the Reactive Black 5 dyes.

Ion chromatography (IC) confirmed a very efficient release of sulfates, which indicates that a larger part of the energy is spent on the separation of aliphatic sulfo groups than on the decolourization process itself, i.e., the oxidation of the diazo bond.

Toxicology tests done with *Artemia salina* test organisms showed that the toxicity of solution is decreased after the plasma treatment.

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