Assessment of novel rotating bipolar multiple disc electrode electrocoagulation–flotation and pulsed plasma corona discharge for the treatment of textile dyes

Narasamma Nippatlapalli and Ligy Philip

ABSTRACT

The current study evaluates the performance of the designed novel electrolytic reactor with rotating bipolar multiple disc electrode (RBDE) in the electrocoagulation–flotation (EC-F) process and a pulsed plasma reactor for the removal of toxic textile dyes. Two different classes of dyes, Methyl Orange (MO), an azo group of dye, and Reactive Blue 19 (RB19), a reactive group of dye, were selected. Efficient removal of both the dyes at a faster rate was obtained with the designed RBDE reactor compared to the EC-F process with static electrodes. RB19 and MO were completely decolourized (100%) within 2 min of electrolysis time with rotating and 6 min with static (non-rotating) electrodes, respectively. Similarly, the maximum chemical oxygen demand removal of 86.4% and 93.2% was obtained for RB19 and MO, respectively, with the rotating electrode EC-F process. On the other hand, complete decolourization was obtained in 10 min and 12 min of pulsed corona discharge for MO (50 mg/L) and RB19 (50 mg/L), respectively. The comparison studies of RBDE and pulsed power plasma reactor (PPT) showed that MO removal was faster than RB19 removal in both RBDE EC-F and PPT processes. Relatively long treatment time was needed for RB19 compared to MO due to its complexity of structure and high solubility. RB19 and MO were completely degraded through pulsed corona discharge without any sludge production. The results show that the designed RBDE reactor performed much better than existing conventional electrocoagulation reactors. The RBDE reactor can be used as a pre-treatment unit for industrial wastewater, which can improve the treatment efficiency and reduces the energy consumption. Plasma technology showed complete degradation of pollutant without sludge production. The formation of a wide variety of reactive oxygen species during corona discharge helps in degrading the pollutants. Plasma technology can be used as a secondary treatment system along with the RBDE as pre-treatment process for complex industrial wastewaters. This will improve the quality of treated effluent and reduce the overall cost of treatment.

Key words | azo, comparison, electrolysis, pulsed corona, reactive, rotating bipolar electrodes

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INTRODUCTION

Textile industry wastewater with various dyes has led to severe environmental pollution especially in developing countries like India (Babaei *et al.* 2017). This industry widely uses a large amount of synthetic dyes and consumes a huge quantity of water and produces an equal quantity of coloured wastewater (Bilińska *et al.* 2019). Around 15–25% of textile dyes are directly discharged into the wastewater. The presence of dyes poses a severe threat to the aquatic ecosystem, if the wastewater is not properly treated before discharge.

Conventional treatment methods such as biological degradation and physicochemical processes are widely used to remove dyes and good removal efficiency has been reported. However, low dye degradation due to the xenobiotic nature (Dewil *et al.* 2017) and incomplete reactions are the major challenges while employing these treatment processes. On the other hand, advanced oxidation processes (AOPs) such as photocatalysis (Buscio *et al.* 2015), photo-Fenton, UV-H_2O_2, and ozonation provide good removal

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efficiency. But the high treatment cost and production of secondary pollutants in the effluent limit their applications (Miklos *et al.* 2018). Hence, an optimized treatment technique which can overcome these challenges is required to minimize the adverse effect to the complete ecosystem.

Electrocoagulation–flotation (EC-F) is a simple and cost-effective electrochemical technique. Mineral coagulants are produced by electrolytic dissolution of sacrificial electrodes. However, in a conventional EC-F process passivation of electrodes and energy consumption are the main limitations (Naje *et al.* 2016). Poor mass transfer of pollutants and accumulation of coagulants at electrode vicinity leads to the quick passivation of electrodes. Hence, the conventional EC-F process needs to be modified to achieve better performance. Sahu *et al.* (2014) reported that proper design of reactors helps in the mechanistic understanding of the EC-F process. Some other researchers (Naje *et al.* 2016; Xu *et al.* 2018) have reported the rotation of electrodes to address this problem. However, an EC-F reactor with multiple rotating bipolar disc electrodes has not been reported to the best of the authors' knowledge.

Pulsed corona discharge, one of the emerging AOPs, has been gaining importance for its versatility to completely degrade any organic pollutant (Singh *et al.* 2016). It is a process of sudden release of electrical energy that creates *in situ* strong oxidants such as H_2O_2, O_3, and OH. These reactive oxygen species (ROS) directly degrade various organic pollutants including dyes. The main advantage of the pulsed power plasma reactor (PPT) process is the production of a variety of ROS without any chemical addition. The short contact time required for the pollutant degradation makes it unique among all the AOPs. However, only a few studies have been reported on the application of non-thermal pulsed corona discharge for the degradation of various dyes.

Previous studies show that Methyl Orange (MO) is easily removed from the wastewater compared to Reactive Blue 19 (RB19). This is attributed to the complex structure of RB19 and its high solubility. Therefore there is a need to focus on removal of such complex structure molecules from the wastewater. In this view, the present study is devoted to the effective removal of RB19 (reactive) and MO (azo) dyes with a rotating bipolar multiple disc electrode (RBDE) EC-F reactor with multiple electrodes and PPT process. This is achieved by (i) carrying out a relative comparison between efficiency of rotating and static electrode EC-F processes for RB19 and MO removal, (ii) performance evaluation of corona discharge for the removal of RB19 and MO dyes and (iii) determining optimum values of various parameters such as current density and electrical conductivity for the EC-F process and pH and alkalinity for the PPT process based on the maximum removal efficiency of complex RB19 dye.

MATERIAL AND METHODS

Design and details of rotating bipolar multiple disc electrodes reactor

Figure 1 shows the RBDE EC-F reactor ($10 \times 8 \times 12$ cm) used in the current study. The reactor consisted of nine electrodes (surface area = 221.2 cm^2) with eight anode and cathode faces each. The electrodes are rotated using a DC motor at 0–1,000 rpm. DC power (model: APLAB; rating: 5 A, 64 V) was supplied to the disc electrodes. A volume of 450 mL dye solution was taken for all the experiments. The Fe electrodes were cleaned with acetone, 0.5 M

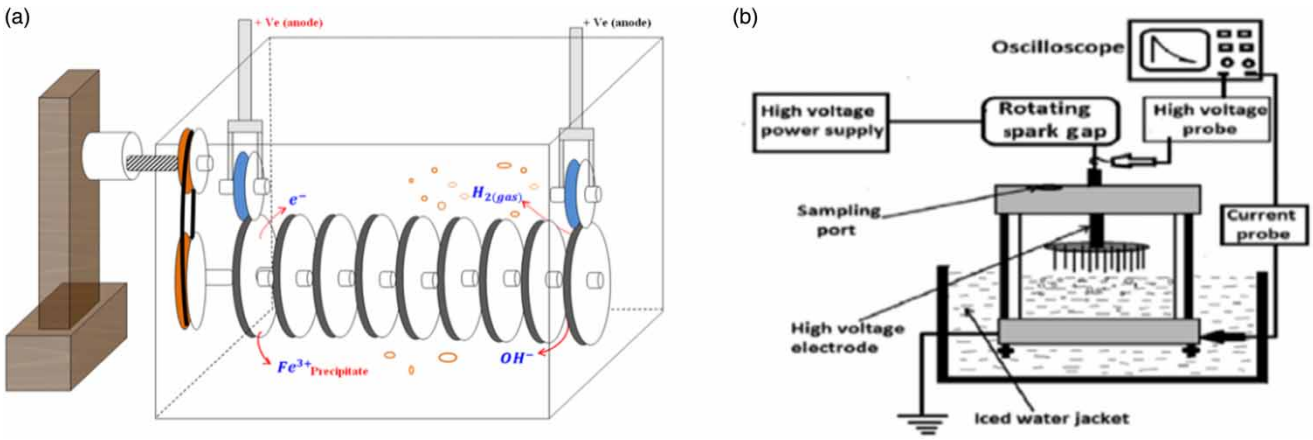

Figure 1 | (a) Rotating multiple disc bipolar electrode reactor set-up. (b) Pulsed corona discharge reactor set-up.

H_2SO_4 and again with distilled water before starting the experiments.

Description of pulsed power plasma reactor (PPT)

Figure 1(b) shows the experimental set-up of PPT containing a high voltage source and reactor cell. A discharge free transformer of 100 kV and 5 kVA was used to generate high AC voltage. Later the AC voltage source was converted to DC voltage using a high voltage diode (140 kV, 20 mA, 100 kΩ). The constant DC voltage was converted to square pulse using a rotating spark gap. Needle plate configuration was used to obtain pulsed corona discharge. A circular plate was used to hold the tungsten needle. It was connected to a high voltage terminal. The bottom of the reactor acted as plane electrode and it was grounded.

In RBDE EC-F and PPT processes, the treated samples were taken at predecided time intervals and stored in the refrigerator for further analysis of colour (UV-spectrophotometry, Shimadzu, Japan) and chemical oxygen demand (COD) (as per APHA method (APHA 2012)). RB19 and MO with initial concentration of 50 mg/L each were treated in EC-F and PPT processes, respectively.

RESULTS AND DISCUSSION

Performance evaluation of rotating and non-rotating bipolar disc electrodes for the removal of RB19 and MO dyes

Figure 2(a) and 2(b) show the removal of MO and RB19 at pH 7 using rotating and non-rotating bipolar disc electrodes. It is clear from the Figure 2(a) that complete decolourization of 50 mg/L of MO was obtained in 2 min and 6 min of electrolysis time with rotating and non-rotating electrodes, respectively. Rotating electrodes also showed high COD reduction from 125 mg/L to 7.2 mg/L (93.6%) compared to non-rotating electrodes (71.6%). For reactive dye, RB19 it took 4 min and 6 min for complete decolourization (100% decolourization) and the COD removal was 62.9% and 86.4%, with rotating and static electrodes, respectively. The experimental results show that MO removal was higher compared to RB19 under similar operating parameters. This could be because of the complexity of the RB19 and its high solubility in water that cause difficulty in degradation. The removal of two different classes of dyes was studied and mentioned in detail in Nippatla & Philip (2019) using EC-F and PPT processes. A plausible reason for the better performance of rotating electrodes is the high activity of electrodes by proper mass transfer of generated coagulants to the bulk solution during rotation. The results are also confirmed by velocity gradient (s^{-1}) as given in Table S1 (Supplementary Information). The obtained faster and complete decolourization (100%) of MO and RB19 was due to the reduction of chromophore bonds by the abundant production of Fe^{2+} ions during rotation. Also, during rotation of electrodes in dye wastewater, the generated coagulants and dye molecules uniformly distributed in the reactor. This ultimately enhanced the adsorption and precipitation processes and thus the removal efficiency. Moreover, the continuous contact of electrodes with NaCl-assisted electrolytic solution enhanced the coagulant generation due to pitting corrosion of electrodes. According to Garcia-Segura *et al.* (2018) electrodes with high anode potential directly oxidize the dye molecules on the electrode surface. Furthermore, the chloride ions converted to reactive chlorine species that act as strong oxidizing species. This could be one of the added reasons for high COD reduction with rotating electrodes.

On the other hand, in the case of non-rotating electrodes, poor mass transfer of generated coagulants and dye

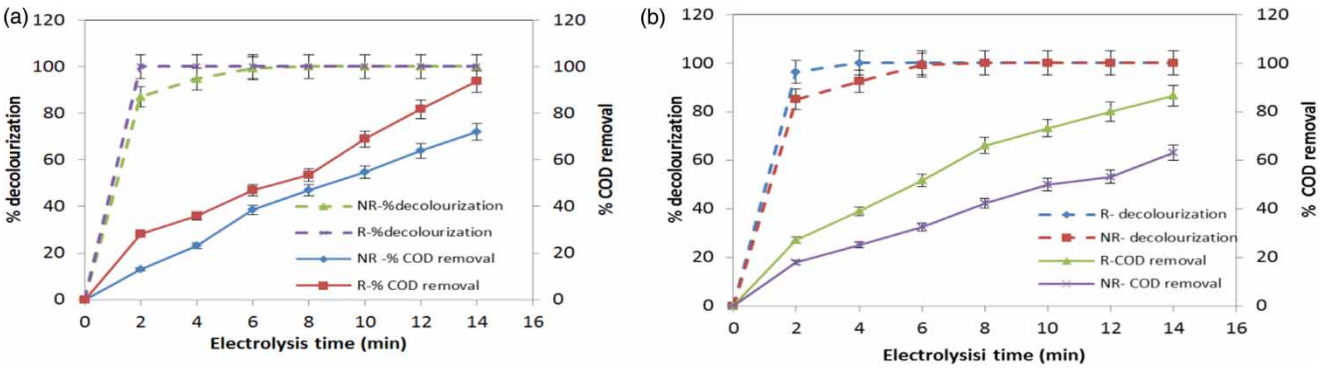

Figure 2 | Comparison of rotating and static electrodes: (a) MO removal and (b) RB19 removal. R: rotation; NR: non-rotation (static).

molecules results in the formation of a thin passivation layer on the surface of the electrodes (Nassar *et al.* 1983). This highly inhibits the activity of electrodes, i.e., reduction in electrochemical reactions (oxidation and reduction of electrodes). In such cases, the electrodes quickly get deteriorated in case of the static electrode EC-F process (Palani *et al.* 2017). Less availability of metal coagulants leads to the reduction in removal efficiency. Moreover, the measured current efficiency (ϕ) also reveals that rotating electrodes ($\phi = 96.92\%$) have more potential than static electrodes ($\phi = 81.63\%$). In addition, the energy consumption was highly reduced in the rotating case (0.56 kWh/kg dye) compared to non-rotating case (0.63 kWh/kg dye). Even though external energy was used for motor rotation in the EC-F process, less ENC was obtained due to reduction in electrolysis time and increase in removal efficiency. Therefore, it can be concluded that the designed rotating bipolar electrode reactor is highly efficient in minimizing the limitations of a conventional EC-F process. Hence, further experiments were carried out with rotating electrodes and various operating parameters were optimized.

Effect of current density and electrical conductivity

In the EC-F process, current density (CD) is an important parameter as it decides the amount of coagulant generation. The effect of varying CD (4.76 mA/cm^2–11.9 mA/cm^2) was studied for RB19 dye as shown in Figure 3(a). The results clearly show that at low CDs of 4.76 mA/cm^2 and 7.14 mA/cm^2, above 90% of decolourization was obtained within 4 min of electrolysis time. Also, the electrolysis time was reduced by 50% (4 min–2 min) at high CDs of 9.52 mA/cm^2 and 11.9 mA/cm^2 to achieve complete decolourization (100% decolourization). Moreover, the initial COD concentration was effectively reduced from 55.3% (128 mg/L–61.75 mg/L) to 96.39% (128 mg/L–8.1 mg/L) with increase in CD (4.76 mA/cm^2–11.9 mA/cm^2) (Figure 3(a)). A plausible reason is the higher electrolytic dissolution of anode as per Faraday's law in which anode oxidation depends on the charge acting on the surface of the electrodes, which increases the removal efficiency with increase in CD. Hence, increase in CD eventually increases the amount of charge on the electrode surface, which results in massive production of mono- and poly-metal hydroxide coagulants. This ultimately boosts the adsorption and precipitation processes of dye removal, as also reported by Nandi & Patel (2017) and Turkanu & Bechtold (2017). However, to achieve such a high COD reduction is not simple with the conventional EC-F process within 14 min of electrolysis time. For instance, Ashtoukhy & Amin (2010) reported 87.3% of COD (initial concentration 100 mg/L) removal in 21 min of electrolysis time at CD of 3.8 mA/cm^2 for the removal of Acid Green 50 dye. Similarly, Alizadeh *et al.* (2014) reported 66% (128.6 mg/L initial COD) removal in 28 min at CD 20 mA/cm^2 using a conventional EC-F process. This reveals that conventional EC-F processes demand either longer electrolysis time or high CD even for the removal of lower concentration of pollutants. In the present study, both anode and cathode underwent self-cleansing through rotation in the electrolytic solution and accelerated the electrochemical reactions. It ultimately resulted in faster decolourization (100% in 2 min) of dyes along with significant COD removal. But it was observed that only 1.8% of removal efficiency was increased with increase in CD from 9.05 to 11.9 mA/cm^2. This may be due to the fact that sufficient coagulants to adsorb and precipitate the given 50 mg/L of RB19 dye might have been generated at a CD of 9.05 mA/cm^2. Therefore, to reduce the overall energy consumption, the optimal CD for RB19 removal was selected as 9.05 mA/cm^2.

Next, the effect of varying electrical conductivity (EC) (4–8 mS/cm) on removal of RB19 in the EC-F process was evaluated and the results are presented in Figure 3(b). The maximum EC was selected based on the actual EC of a

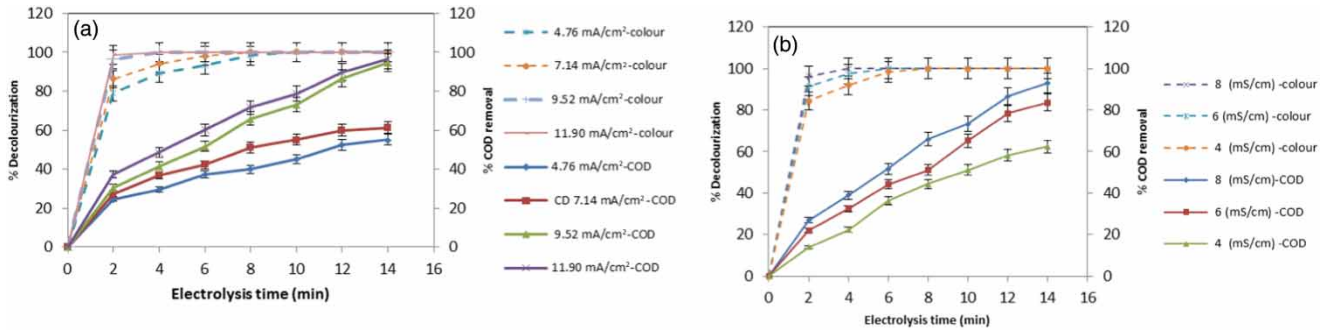

Figure 3 | Effect of (a) current density and (b) electrical conductivity on RB19 removal in RBDE EC-F process.

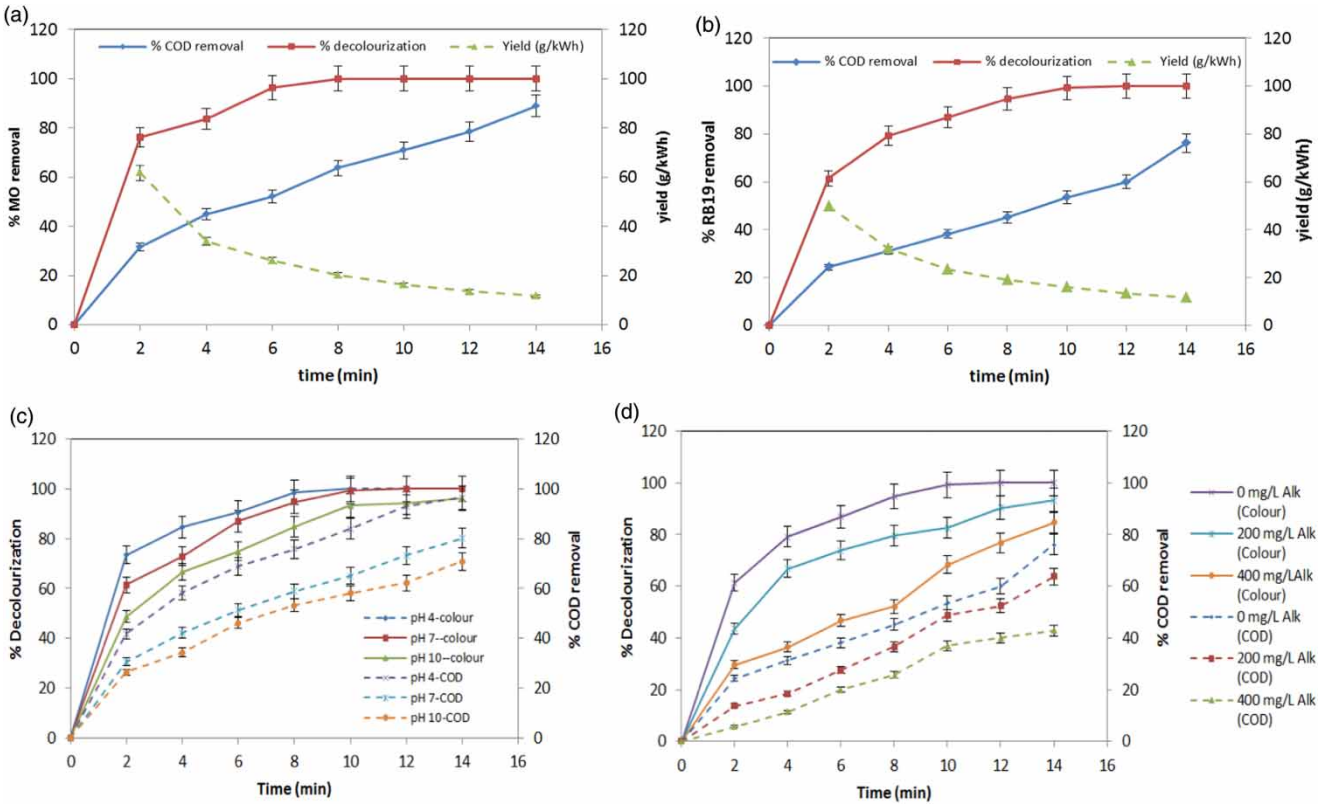

Figure 4 | Effect of pulsed corona (PPT) discharge on (a) MO removal and (b) RB19 and its yield. Effect of (c) pH and (d) alkalinity on RB19 removal in PPT process.

Tirupur real textile wastewater, which was 8.6 mS/cm (Nippatla & Philip 2019). From the results it can be seen that colour removal was not much influenced with increase in EC from 4 mS/cm to 8 mS/cm and complete decolourization (100%) was achieved within 6 min of electrolysis time. However, increase in EC significantly increased the COD removal from 62.33% to 92.9% as shown in Figure 3(b). This is because high conductivity induces more consumption of the Fe electrodes due to corrosion pitting. A similar observation was also reported by Wang *et al.* (2016). Increase in conductivity also decreased the intake voltage (32.3 V–24.2 V) that notably decreases the energy consumption.

Performance evaluation of pulsed corona discharge for the removal of dyes (RB19 and MO)

Figure 4(a) and 4(b) show the degradation of dyes MO and RB19 under pulsed corona discharge. The result shows that the removal efficiency was increased with increase in corona discharge time. It can be seen that the complete decolourization of reactive dye MO and azo dye RB19 was obtained at 8 min and 10 min of corona discharge, respectively. Also, significant COD removal of 89.3% in the case of MO (Figure 4(a)) was achieved, which indicates its faster degradation compared to RB19 (76.18%) (Figure 4(b)). Increase in corona discharge time increased the removal efficiency because of significant increase in the amount of various radicals generated. For instance $OH^{\cdot}$ radical concentration was linearly increased from 0.8×10^{-6} M to 2×10^{-4} M with increase in corona discharge time from 5 min to 30 min (Sahni & Locke 2006) and Singh *et al.* (2016) reported 18 mg/L–60 mg/L of OH and 12–23 mg/L of H_2O_2 with increase in corona discharge from 2 to 12 min. Hence, the removal efficiency is faster due to the group effect of all the high potential ROS generated simultaneously with increase in time. On the other hand, the results show that MO removal was higher than RB19 at the same operating conditions of 23 kV, 25 Hz frequency and 14 min of corona discharge time. A plausible reason is the chemical nature of dyes. The log K_{ow} values of RB19 and MO are −1.85 and 3.29, respectively. The high interfacial concentration of MO over RB19 increased the removal efficiency of MO. In the case of RB19 removal, it involves a series of reactions to achieve its complete degradation, which demands more ROS species. A previous

study (Singh *et al.* 2017) also reported similar behaviour during the removal of various organic compounds by pulsed plasma process. The yield (amount of dye degradation per unit of energy consumed, g/kWh) was higher in the case of MO (61.7 g/kWh–20.26 g/kWh) as compared to RB19 (49.3 g/kWh–19.18 g/kWh) at a voltage of 23 kV and 25 Hz frequency. The higher yield indicates the faster degradation of dye. Pulsed corona discharge showed remarkable results in complete degradation of RB19 and MO, respectively without sludge production.

Effect of pH and alkalinity on dye removal

In the PPT process, pH is an important parameter as the production of ROS mainly depends on the pH of the solution (Liu *et al.* 2018). The influence of pH on the removal of RB19 was studied from initial pH 4–9 as shown in Figure 4(c). As mentioned, it can be seen that highest removal of RB19 was obtained at pH 4. Complete decolourization (100%) was obtained in 8 min of corona discharge along with a COD reduction from 128 mg/L to 4.96 mg/L at pH 4. A plausible reason is that maximum concentration of the highly potential $OH^{\cdot}$ and H_2O_2 radicals was obtained at pH 4 as also reported by Singh *et al.* (2016). With increase in pH, most of the ROS such as H_2O_2 undergo self-decomposition and some of the radicals ($OH^{\cdot}$) get scavenged by the hydroxide ions. This can be explained as $OH^{\cdot}$ radicals play a major role in pulsed discharge systems for the decolourization process. In basic conditions most of the generated $OH^{\cdot}$ radicals immediately react with the carbonate ions but do not react with the dye molecules or its intermediates during the oxidation process. Thus, the availability of oxidizing species required for the degradation of dye molecules decreases at high pH. Hence, at pH 10 the maximum decolourization of 96.1% was obtained in 14 min of corona discharge whereas at pH 4 the complete decolourization was obtained in 10 min of corona discharge. Also, COD was effectively reduced to permissible limits within 14 min of corona discharge at acidic pH (pH 4). This is explained by the effective utilization of ROS at pH 4 as also reported by Koutahzadeh *et al.* (2016). However, 100% decolourization was obtained at all pH values with increase in treatment time due to production of sufficient quantity of ROS with time. Therefore, to achieve maximum removal efficiency in less treatment time and to reduce energy consumption significantly, the optimum pH (4) is important.

The effect of alkalinity (0–400 mg/L) on the removal of RB19 was also studied (Figure 4(d)). Alkalinity showed a negative effect on dye degradation and the removal efficiency decreased with increase in alkalinity as also reported in the previous studies by Faria *et al.* (2009). The COD removal efficiency was decreased from 76.18% to 42.3% with increase in alkalinity from 0 mg/L to 400 mg/L. It can also be seen that colour removal was decreased to 84.6% from 100% when alkalinity was increased from 0 mg/L to 400 mg/L. This could be due to the scavenging of ROS by carbonates and bicarbonates. As aforementioned, $OH^{\cdot}$ radicals play a major role in dye degradation and it is the species that gets generated in greater amount compared to other ROS in pulsed corona discharge. The presence of inorganic anionic species (carbonates and bicarbonates) affects the destruction of dye molecules in wastewater (Wang *et al.* 2019) by scavenging mainly the available $OH^{\cdot}$ radicals. Hence, most of the ROS may not be utilized properly for dye degradation alone and some will be scavenged by the inorganic ions. This ultimately inhibits the mineralization process of dyes. The results are in good agreement with Bian *et al.* (2009).

CONCLUSION

The present study focussed on the design and performance evaluation of a novel RBDE reactor for the treatment of textile wastewater. The reactor performed far better than conventional electrocoagulation systems. Complete decolourization of two different classes of dyes, with an initial concentration of 50 mg/L, of was achieved in 2–4 min of electrolysis time. The faster pollutant removal with less ENC (KWh/kg dye) illustrates the superiority of the RBDE reactor over existing EC-F reactors. On the other hand, plasma treatment showed complete degradation of dyes without any sludge production. The production of various ROS ($OH^{\cdot}$, H_2O_2, O_3, $O^{\cdot}$) species directly degraded the pollutant. EC-F with rotating electrodes is more economical and effective as a pre-treatment process whereas PPT is suitable as a tertiary treatment process that can eliminate the pollutants completely from secondary treated textile industry wastewater. The RBDE EC-F process followed by plasma treatment can generate effluent that can be reused in the industry for various purposes.

SUPPLEMENTARY MATERIAL

The Supplementary Material for this paper is available online at https://dx.doi.org/10.2166/wst.2020.137.

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