

Efficient removal of ammoniacal nitrogen from textile printing wastewater by electro-oxidation considering the effects of NaCl and NaOCl addition

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ABSTRACT

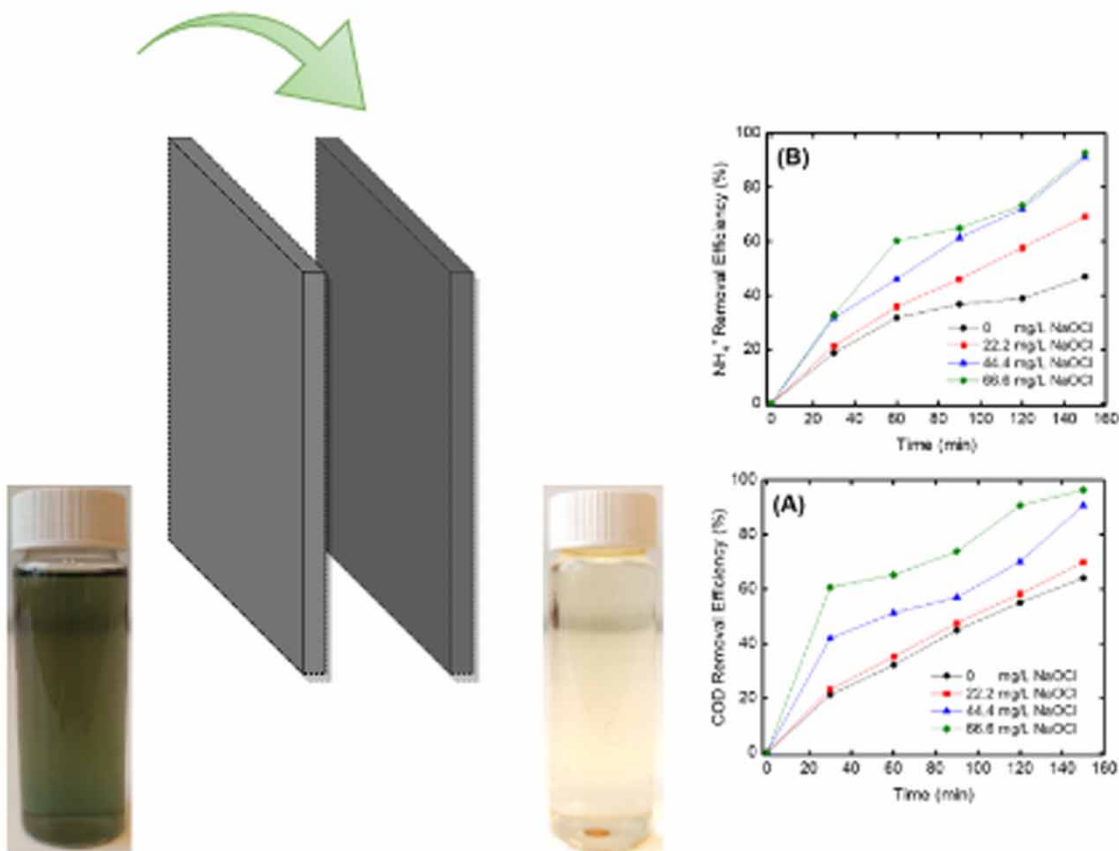
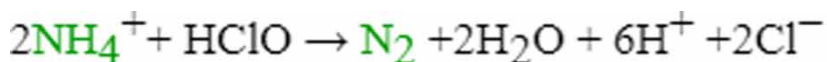
In this study, an electro-oxidation (EO) process using graphite electrodes as electrode pairs was used for the removal of chemical oxygen demand (COD), ammoniacal nitrogen ($\text{NH}_4^+\text{-N}$), and color from real textile printing wastewater. The effects of solution pH, sodium chloride (NaCl) dosage, sodium hypochlorite (NaOCl), which is the oldest and still most important chlorine-based bleach, dosage, and oxidation time were investigated on the removal efficiencies. Operating conditions for the EO reactor were applied to current density 1 mA/cm^2 , distance between the electrodes: 2 cm, 150 min operation time, and stirring speed of 500 rpm. At optimum conditions: pH 9.5, applied current density 1 mA/cm^2 , NaCl dosage of 8 g/L, NaOCl dosage of 44.4 mg/L and 150 min electro-oxidation time, the obtained removal efficiencies were 86.5% and 91.1% for chemical oxygen demand (COD) and ammoniacal nitrogen, respectively. Efficiency was increased to 91.1% for ammoniacal nitrogen from 21.7% after applying EO combined with NaOCl addition compared to individual NaOCl addition.

Key words: ammonium removal, COD removal, dye removal, electro-oxidation, textile printing wastewater

HIGHLIGHTS

- Ammonia concentrations can be high in textile dye wastewater due to the usage of nitrogen-based substances.
- Various oxidation processes are not effective for ammonium removal.
- The aim of this study was to determine the efficiency of individual EO and EO with an addition of NaCl and NaOCl.
- EO assisted NaOCl and NaCl is a promising technology for the oxidation of both carbon and nitrogen-based contaminants.

GRAPHICAL ABSTRACT



INTRODUCTION

Water resources in the world are decreasing rapidly day by day. Among these resources, potable water resources have reached the point of depletion (Lahnsteiner & Lempert 2007). Conversely, wastewater discharge to receiving environments without any treatment causes pollution of water resources, the amount of which is decreasing day by day. For this reason, wastewater should be treated to preserve existing water resources and to use water resources more efficiently (Muthukumaran *et al.* 2011).

Textile industry wastewater is one of the most important hazardous wastes threatening people and nature. Colored textile wastewater accumulation in water bodies damages the aesthetic appearance of the water and reduces the penetration of light into the water. The reduction of light penetration and dissolved oxygen concentration causes extinction of aquatic life and limits the use of water resources. Some dyes also contain toxic substances (Carmen & Daniel 2012; Yaseen & Scholz 2019). Even in very small quantities, colored textile wastewater can spread to large water bodies due to its high dispersion rate. Therefore, textile wastewater threatens the environment when it is discharged without any treatment (Pattnaik *et al.* 2018).

The textile industry involves of various processes including desizing, scouring, bleaching, and dyeing (Dos Santos *et al.* 2007). Dyeing is identified as the procedure of adding color to fibers using a solution mixture of water, dyestuff, and chemicals in these processes. Large volumes of dye containing wastewater are produced in this process (Patel & Vashi 2015). Textile wastewater is generally characterized by high chemical oxygen demand (COD) concentration, very low ratio of biochemical oxygen demand (BOD) to COD, dark color, high concentration of organic substances, alkaline pH, significant salinity, and high toxicity (Imtiazuddin *et al.* 2012; Ananthashankar 2014; Patel & Vashi 2015). The nitro-aromatics and ammonium sulphate utilized in the dyeing process are the reason for formation nitrogen-based pollutants. Urea is also widely used in

the dyeing process to improve the solubility of the dyes in water and increase the brightness and intensity of the dyes utilized. Another advantage of urea is that it retains moisture even when dried, so it increases the color yield of the dye (Sheth & Musale 2004). Hence, ammonia concentrations can be high in textile dye wastewater due to the usage of nitrogen-based substances. Discharging ammonia-containing wastewater without treatment worsens the water quality and enhances eutrophication (O'Neill *et al.* 2000).

Generally, textile wastewater treatment methods include coagulation/flocculation, biological processes, membrane processes, and advanced oxidation processes (AOPs) in various configurations have been suggested (Krull & Döpkens 2004; Vajnhandl & Valh 2014). The membrane processes and coagulation/flocculation convert contaminants from one phase to another or produce a concentrate stream, while AOPs can degrade or entirely remove pollutants (Bes-Piá *et al.* 2003; Punzi *et al.* 2015). Biological treatment consists of various types of microorganisms in a bioreactor to degrade organic and inorganic material (Khandare & Govindwar 2015; Bhatia *et al.* 2017). Biological treatment processes are a well known and cost-effective method for the treatment of textile wastewater. However, they have some limitations such as high investment cost, use for only biodegradable compounds, and contain toxic substances (Barrera-Díaz *et al.* 2014; Ganzenko *et al.* 2014; Punzi *et al.* 2015).

AOPs processes have been used to eliminate these disadvantages. AOPs are based on the production of hydroxyl radicals some of the most powerful and non-selective oxidants and are able to react with almost all materials even refractory compounds (Oturán & Aaron 2014; Miklos *et al.* 2018). Contrary to biological processes, AOPs are known as expensive due to their chemical and energy demands (Oller *et al.* 2011). Moreover, the most important advantage of AOPs is not to produce an additional waste stream (Dewil *et al.* 2017). Different techniques and more efficient reactor are being developed to make AOPs more feasible (Shang *et al.* 2006; Barkul *et al.* 2017).

Various oxidation processes including Fenton, hydrogen peroxide, ozone, ultraviolet light and their combination have proved to be efficient for the removal of non-biodegradable organic compounds, but they are not effective for ammonium removal (Cabeza *et al.* 2007). Recently, the electrochemical oxidation process has been demonstrated as a favorable option for ammonium removal from various kinds of wastewaters for example tannery wastewaters (Panizza & Cerisola 2004; Szpyrkowicz *et al.* 2005), anaerobic digestion effluent (Ihara *et al.* 2006), and domestic wastewater (Ghimire *et al.* 2019).

Ammonium degradation is performed by an indirect oxidation process where oxidants such as chlorine, hypochlorous acid, and hypochlorite are produced at electrodes by following reactions (Cabeza *et al.* 2007):



First reaction rate can be controlled with mass transfer and kinetics changing with current density, chloride concentration and reactor hydrodynamics. Formation of chlorine, hypochlorous acid, and hypochlorite is related to the pH value of the solution. Also, the higher initial chloride concentration in the electrolyte and higher current density promote the production of more chlorine (Särkkä & Sillanpää 2020).

The anodic formation of chlorine reacts with ammonium ions,



Earlier studies have stated that the current density and the initial chloride concentration are related process variables that affect the ammonium removal from wastewater (Wang *et al.* 2001; Ihara *et al.* 2006; Moraes & Bertazzoli 2005). The results proved that ammonium removal efficiency is enhanced with an increase in the current density and the chloride concentration.

The aim of this study was to determine the efficiency of individual EO and EO with the addition of NaCl and NaOCl for the removal of COD, ammoniacal nitrogen, and color from real textile printing wastewater. The research includes the evaluation of the main operational factors such as, pH, NaCl and NaOCl amount, and oxidation time on the treatment performance for the EO process. EO was performed in order to treat an ammonium-contained textile wastewater and graphite electrode was used.

MATERIALS AND METHODS

Materials

The solvents and chemicals used in COD analysis were sulfuric acid (Merck, 98% purity), ferrous ammonium sulphate (Merck), potassium dichromate (Merck), silver sulphate (Merck), mercury sulphate (Merck). Methyl red (Merck), boric acid (Merck), methylene blue (Merck), sodium tetraborate (Merck) were consumed in ammonia analysis. The pH of solution was adjusted with hydrochloric acid (HCl, Sigma-Aldrich, 38% purity). NaCl and NaOCl was supplied from Merck.

Wastewater characterization

The wastewater was obtained from the dyeing process (printing) of textile production factory in Turkey. Table 1 shows the typical characteristics of the wastewater. It shows that average COD and total organic carbon (TOC) concentrations are 1,067 mg/L and 303 mg/L, respectively. As well as the ammonia concentration in such wastewater from dyeing process is generally high, it is 1,257 mg/L in the wastewater studied. All experiments were performed in triplicate and average results are presented as the result.

Electro-oxidation reactor

The batch experiments were achieved in a 0.25 L reactor at room temperature. Schematic illustration of the experimental set-up is presented in Figure 1. The graphite electrodes were used in batch experiments for both anode and cathode. The

Table 1 | Properties of the raw wastewater

Parameter	Unit	Value for before treatment	Value for after treatment
pH (20 °C)	-	9.5 ± 0.2	10.9 ± 0.3
COD (Chemical Oxygen Demand)	mg/L	$1,067 \pm 43$	40.5 ± 3.5
TOC (Total Organic Carbon)	mg/L	303 ± 3.5	11.6 ± 2.2
NH_4^+ (Ammonia)	mg/L	$1,257 \pm 72$	91.6 ± 2.1
Cl^- (Chloride)	mg/L	$3,449 \pm 129$	$1,475 \pm 25$
Conductivity	$\mu\text{S}/\text{cm}$	$6,700 \pm 126$	$2,850 \pm 40$

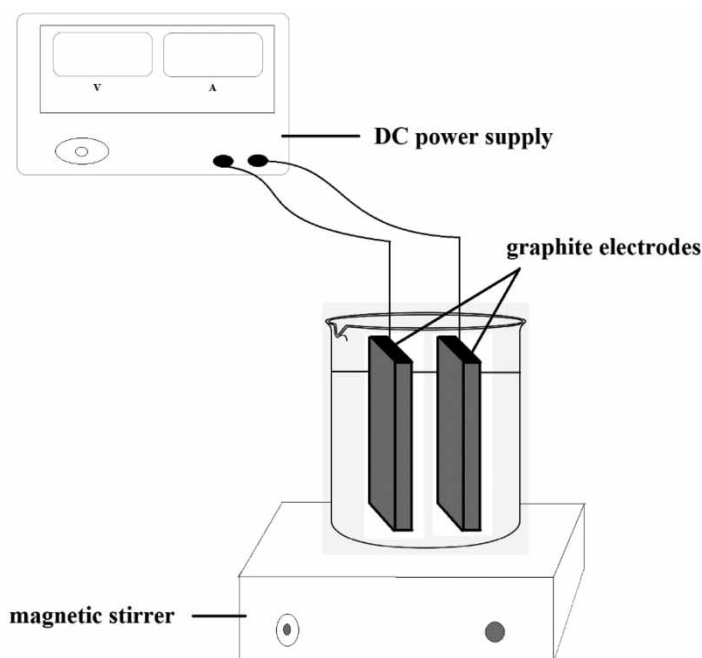


Figure 1 | Schematic diagram of the experimental set-up.

electrodes were placed in parallel at a horizontal distance of 1 cm. During preliminary experimental studies, the distance between electrodes was tested as 1, 2, or 3 cm. However, as the distance increased, the removal efficiency decreased and therefore a 1 cm distance was kept constant during the experiments. The effective wetted area of graphite electrode was 22.75 cm². The reactor was stirred with a magnetic mixer for 60 min of reaction time. All experimental sets were operated without pH control, with initial pH adjusted. The voltage through the electrodes was observed by keeping the current constant with 1.042 A in all sets. The operational conditions of the experimental sets are given in Table 2.

Experimental sets were conceived to determine the effects of pH, sodium chloride (NaCl), and sodium hypochlorite (NaOCl) on the removal of COD, ammoniacal nitrogen, and color. The effects of different NaCl concentrations (1, 8, and 15 g/L) were investigated by keeping the initial pH constant for set 1. For set 2, to determine the effect of pH, batch experiments were carried out at the original pH of the wastewater and pH 4 with the optimum amount of NaCl determined for set 1. Finally, to determine the effect of NaOCl on the removal of ammoniacal nitrogen and COD, various NaOCl concentrations (20, 40, and 60 mg/L) were added in set 3. Additionally, the effect of electrolysis time was evaluated by collecting sample for every 15 min.

Analytical procedures

The pH and the conductivity were measured using a pH/Cond. handheld multimeter (Hach-HQ40d). The chemical oxygen demand (COD) was measured using the closed reflux method-titrimetric method following Standard Method No. 5220C (APHA/AWWA/WEF 1998). The concentration of the chloride before and after the EO process was measured by the Argentometric method following Standard Method No. 4500-Cl- B (APHA/AWWA/WEF 1998). Ammonia was measured using the titrimetric method following Standard Method No. 4500-NH₃ C (APHA/AWWA/WEF 1998). Ammonia was titrated with standard sulfuric acid using a mixed indicator of methyl red and methylene blue. The color of wastewater samples was measured as absorbance at the wavelengths of 436 nm, 525 nm, and 620 nm by following the method of EN ISO 7887 using a Hach DR6000 model spectrophotometer. The TOC content of the samples was determined by using a Shimadzu 5000A model TOC analyzer. The instrument calculates the difference between TC and IC to obtain the TOC value according to Standard Method No. 5310 B (APHA/AWWA/WEF 1998). A digital DC power supply (GW Instek GPD-3303S) was used for electrolysis.

RESULTS AND DISCUSSION

Effect of NaCl addition on removal of COD, ammoniacal nitrogen, and color

First, the electrochemical reactor was operated under different NaCl amounts (0, 1, 8, and 15 g/L) to investigate salt effect on removal efficiency of COD, ammoniacal nitrogen, and color. The results showed that COD removal efficiency decreased by increasing the amount of NaCl. However, as the amount of NaCl increased, the removal efficiency of ammoniacal nitrogen and color improved. The COD and ammoniacal nitrogen removal efficiencies differed in the range of 62.5–70.0% (Figure 2(a)) and 2.4–40.6% (Figure 2(b)) for 0–15 g/L NaCl amount, respectively. The variations in color removal efficiencies for 0–15 g/L NaCl amount changed in the range 4.3–81.4%, 26.8–99.4%, and 72.1–99.7% for 436, 525, and 620 nm, respectively

Table 2 | All experimental set-up

Set		Initial pH	Initial NaCl concentration (g/L)	Initial NaOCl concentration (mg/L)
1	1.1	6.0	0	0
	1.2	6.0	1	0
	1.3	6.0	8	0
	1.4	6.0	15	0
2	2.1	4.0	8	0
	2.2	6.0	8	0
	2.3	9.5	8	0
3	3.1	9.5	8	0
	3.2	9.5	8	22.2
	3.3	9.5	8	44.4
	3.4	9.5	8	66.6

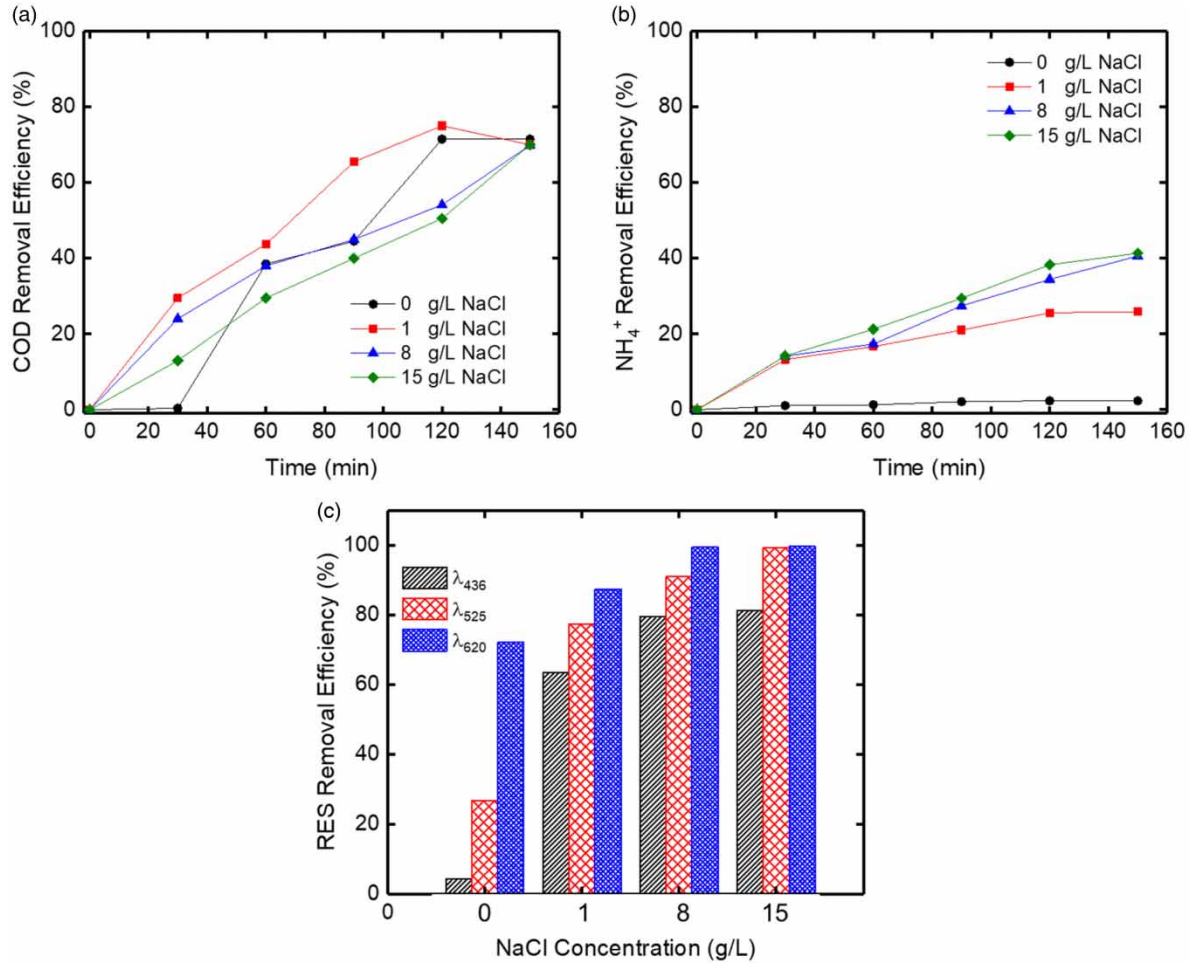


Figure 2 | (a) The effect of NaCl concentration on COD removal efficiency, (b) The effect of NaCl concentration on NH₄⁺ removal efficiency, (c) The effect of NaCl concentration on RES removal efficiency (Experimental conditions: Current density: 1 mA/cm², distance between the electrodes: 2 cm, initial pH: 6.0, stirring speed: 500 rpm).

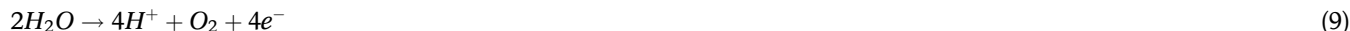
(Figure 2(c)). Ammoniacal nitrogen and color removal efficiencies improved as the amount of NaCl increased up to 15 g/L. However, 8 g/L was chosen for further experiments when considering operational cost. The increasing removal efficiency might be explained as electrogeneration degree of hypochlorite increased. Chlorides (Cl⁻) are the most well known species for mediated oxidation and chlorides in wastewater can be simply transform to chlorine gas (Cl₂) and hypochlorite (ClO⁻) in according to the reaction in Equation (5) at the anode. Additionally, the reaction occurred at the cathode is shown in Equation (6) (Li & Liu 2009).



The generated Cl₂ gas is hydrolyzed and ionized as the reaction specified in Equations (7) and (8):



Also, the side reactions take place in anode with the produced O_2 and cause the production of chlorate (ClO_3^-) according to Equations (9) and (10):



These reactive species have a tendency to react rapidly with ammoniacal nitrogen and colored substances and encourage their mineralization. Previous studies have demonstrated that the chloride concentration are significant process variables that affect the ammonium removal from wastewaters. Results showed that, as the chloride concentration was raised, the removal efficiency of ammonium was enhanced (Wang & Lau 2001; Vlyssides & Karlis 2002; Panizza & Cerisola 2004; Moraes & Bertazzoli 2005; Szpyrkowicz *et al.* 2005; Ihara *et al.* 2006). The maximum ammonium removal efficiency obtained in one study was 95.8%, for 240 min of reaction time at a current density of 30 mA/cm^2 and with an initial chloride concentration of 8570 mg/L (Cabeza *et al.* 2007).

Effect of initial pH on removal of COD, ammoniacal nitrogen, and color

It is well known that solution pH plays an important role in the removal efficiency in electrochemical processes. For this reason, the effect of initial pH between 4.0 and 9.5 was investigated for removal efficiency of COD, ammoniacal nitrogen,

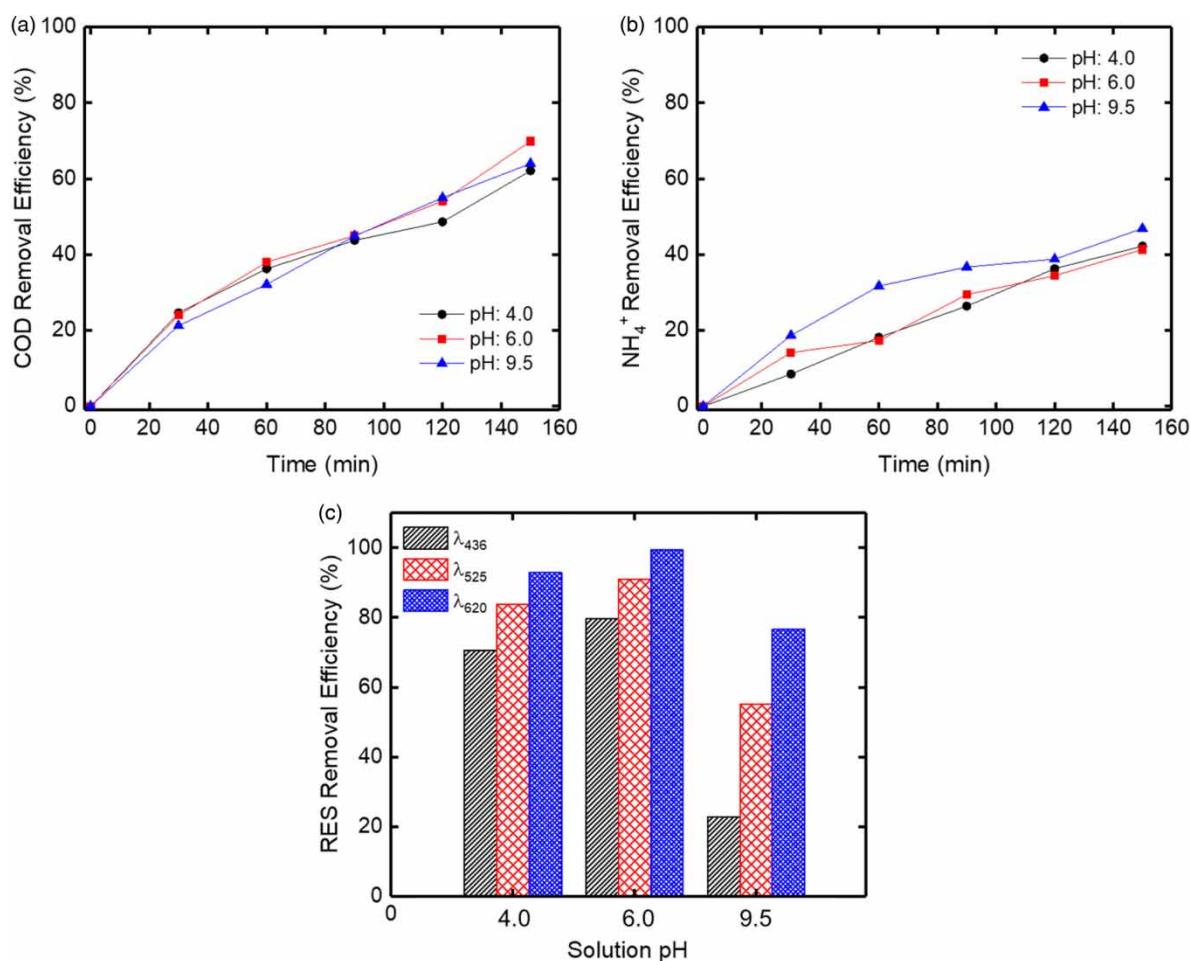


Figure 3 | (a) The effect of pH on COD removal efficiency, (b) the effect of pH on NH_4^+ removal efficiency, (c) the effect of pH on RES removal efficiency (Experimental conditions: Current density: 1 mA/cm^2 , distance between the electrodes: 2 cm, initial NaCl concentration: 8 g/L , stirring speed: 500 rpm).

and color. COD and ammoniacal nitrogen removal efficiencies changed in the range 62.1–69.9% (Figure 3(a)) and 41.3–46.9% (Figure 3(b)) for solution pH of 4.0–9.5, respectively. Ammoniacal nitrogen removal efficiencies were close under acidic conditions (pH: 4.0 and 6.0); however, they were highest for alkali conditions (pH: 9.5). The results showed that pH had the greatest effect on color removal efficiency. The variations in color removal efficiencies for pH 4.0–9.5 changed in the range 70.5–22.9%, 83.9–55.0%, and 92.8–76.6% for 436, 525, and 620 nm, respectively (Figure 3(c)). In our study, the lowest color removal efficiency was obtained for pH 9.5. This can be explained because ClO^- ion is the primary species at pH 10. HOCl ionization species reduce at lower pH values, causing a decline in the oxidation reaction. The reaction of Cl_2 in acidic and alkaline condition is specified by Equations (11) and (12) (Li & Liu 2009; Ghimire *et al.* 2019):



Acidic medium inhibits ClO^- ions as well as chlorine and they return to HOCl. Thus, the removal of chlorine reduced the available oxidant and affected negatively ammoniacal nitrogen removal efficiency. Similar results were reported by Li & Liu (2009). Ammonia concentration decreased from 32.0 mg N/L to 10.4, 1.3, and 1.8 mg N/L at pH 3.0, 7.0, and 9.3, respectively, after 2.5 h of electrolysis. The results depicted that neutral and alkaline pH were beneficial to ammonia oxidation for the EO process. In another study, the highest electrocatalytic performance for ammonia oxidation was obtained in alkaline conditions as 80.6% at pH 12, while that with a neutral condition was the lowest (Yao *et al.* 2019). $\text{Cl}_{2(\text{aq})}$ and HOCl are the predominant species in acidic conditions, whereas ClO^- mainly exists in alkaline conditions. The generated $\text{Cl}_{2(\text{aq})}$ might escape into the atmosphere, leading to a reduction in active chlorine which could oxidize ammonia in acidic conditions (Zou *et al.* 2017).

The electrochemical removal of organics is defined either as direct anodic electro-oxidation or indirect anodic electro-oxidation. In direct electrochemical oxidation, physically adsorbed hydroxyl radicals at the anode surfaces can oxidize the organics directly to carbon dioxide as a final product (Comninellis 1994). In indirect anodic electro-oxidation, generated chlorine and hypochlorite are used as the oxidizing agents for the decomposition of organics in the presence of chloride ions (Chen 2004). In our study, smaller sized dye molecules may have been adsorbed more easily on the surface of graphite electrodes in acidic conditions compared to larger organic molecules and decolorization may have occurred faster than organic molecule oxidation.

Effect of NaOCl dosage on removal of COD, ammoniacal nitrogen, and color

The effect of NaOCl dosage was investigated on removal of COD, ammoniacal nitrogen, and color. COD and ammoniacal nitrogen removal efficiencies changed in the range 64.0–96.2% (Figure 4(a)) and 46.9–91.1% (Figure 4(b)) for a NaOCl concentration of 0.0–66.6 mg/L, respectively. COD and ammoniacal nitrogen removal efficiencies increased significantly compared to without NaOCl addition. The variations in color removal efficiencies for 0.0–66.6 mg/L NaOCl concentration changed in the range 22.8–67.5%, 55.0–85.9%, and 76.6–93.6% for 436, 525, and 620 nm, respectively (Figure 4(c)). It was observed that the desired removal efficiency for COD and ammoniacal nitrogen was reached at a concentration of 44.4 mg NaOCl/L. The results showed that increasing the NaClO concentration caused an increase in COD and NH_4^+ removal efficiencies. This can be explained by a high Cl^- concentration in the wastewater (3,449 mg/L) that enhanced the production of HOCl and ClO^- ions and which increased COD and NH_4^+ removal efficiencies. Hypochlorous acid (HOCl) reacts with NH_3 or NH_4^+ through breakpoint chlorination to regenerate Cl^- ions. Thereafter, the Cl^- ions concentration was expected to remain unchanged after the reaction, serving only as a catalyst (Ghimire *et al.* 2019).

NH_4^+ removal efficiency was also investigated without electro-oxidation conditions. It can be clearly seen from Figure 5 that electro-oxidation improved the removal efficiency compared to without electro-oxidation. The more chlorine gas formed as a result of electro-oxidation caused the improved NH_4^+ removal efficiency.

CONCLUSION

In the current study, individual EO and EO with addition of NaCl and NaOCl were studied for the treatment of COD, ammoniacal nitrogen, and color from real textile printing wastewater. The effects of the pH, NaCl and NaOCl dosages, and electro-oxidation time on the treatment performance of a lab-scale batch EO reactor were examined. The graphite electrode pairs

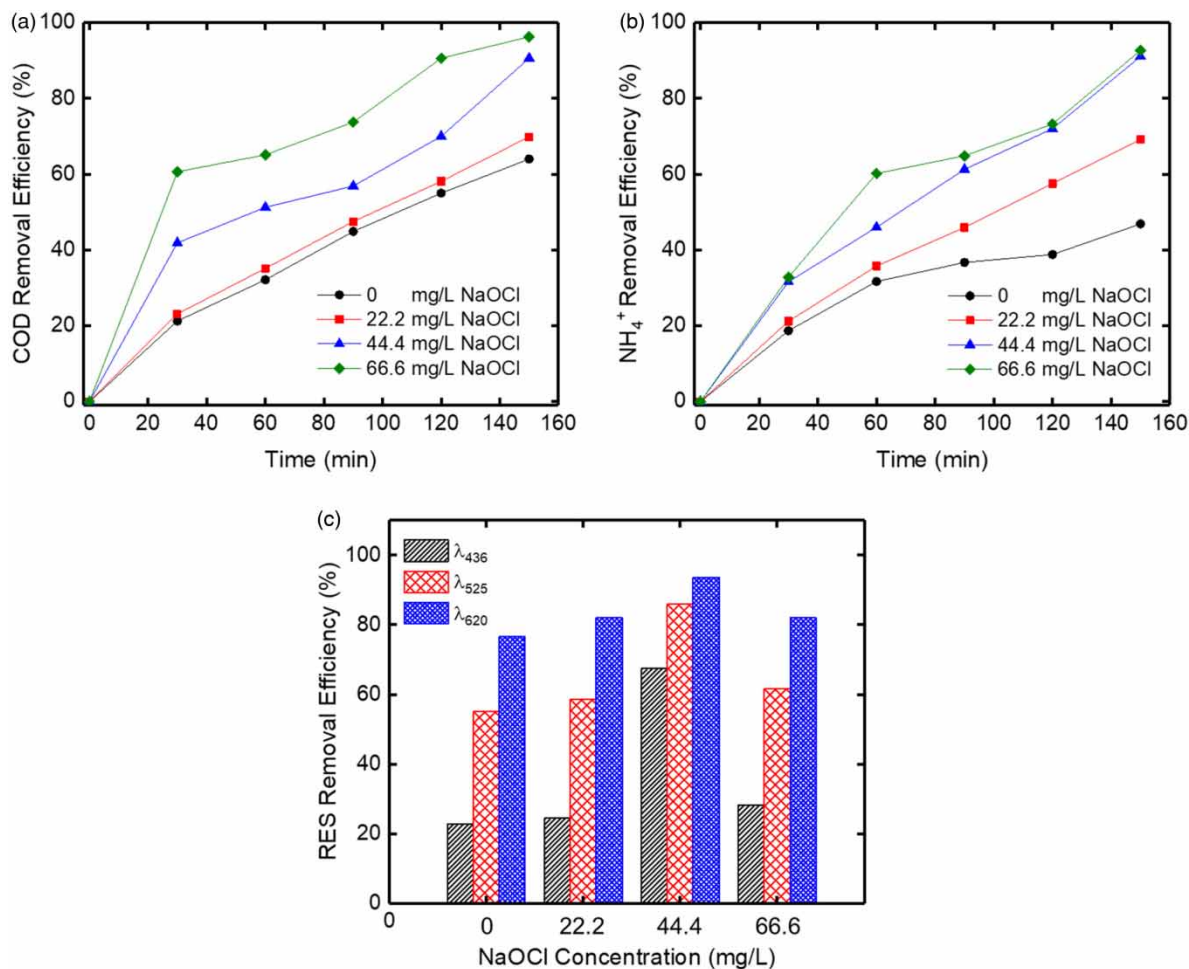


Figure 4 | (a) The effect of NaOCl concentration on COD removal efficiency, (b) the effect of NaOCl concentration on NH₄⁺ removal efficiency, (c) the effect of NaOCl concentration on RES removal efficiency (Experimental conditions: Current density: 1 mA/cm², distance between the electrodes: 2 cm, initial NaCl concentration: 8 g/L, initial pH: 9.5 (original pH), stirring speed: 500 rpm).

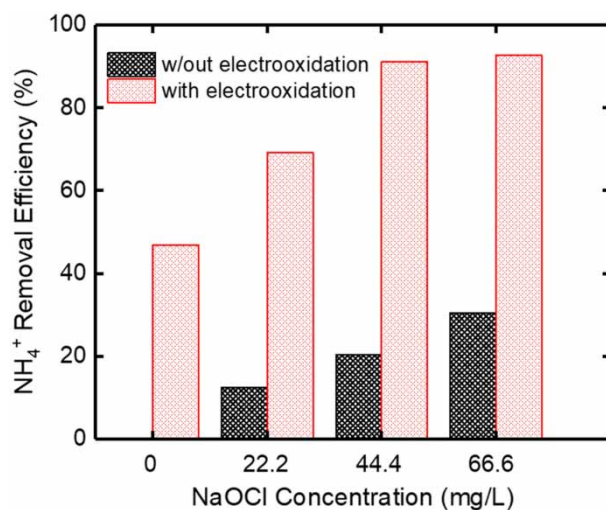


Figure 5 | The effect of NaOCl concentration on NH₄⁺ removal efficiency with or without electro-oxidation (Experimental conditions: Current density: 1 mA/cm², distance between the electrodes: 2 cm, initial NaCl concentration: 8 g/L, initial pH: 9.5 (original pH), operation time: 150 min, stirring speed: 500 rpm).

were used in the treatment process. Operational parameters of EO reactor were applied as current density 1 mA/cm², distance between the electrodes: 2 cm, 150 min operation time, and stirring speed of 500 rpm. The optimum values were obtained at pH 9.5, NaCl dosage of 8 g/L, and NaOCl dosage of 44.4 mg/L. The maximum removal efficiencies at the optimum conditions were 86.5% and 91.1% for COD and ammoniacal nitrogen, respectively. It is stated that electro-oxidation improved the removal efficiency compared to NaOCl treatment alone without electro-oxidation. The ammoniacal nitrogen removal efficiency was enhanced to 91.1% from 21.7% by applying EO combined with the addition of 44.4 mg/L NaOCl compared to only the addition of 44.4 mg/L NaOCl. The results prove that EO assisted NaOCl and NaCl is a favorable technology for the oxidation of both carbon- and nitrogen-derived pollutants.

DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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