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Performance evaluation of electrocoagulation process using aluminum and titanium electrodes for removal of urea

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ABSTRACT

In this research, the efficacy of electrocoagulation has been investigated as a potential treatment process for the removal of urea from synthetic and real wastewaters. The purpose of this study is to evaluate the ability of the electrocoagulation process to remove urea using aluminum and titanium electrodes under varying conditions. The impact of electrode material type, electrode voltage, electrolyte type, and electrode spacing has been investigated. The maximum removal efficiencies for urea occurred after 90 min and its values were 59% at a 9 V for titanium and 40.16% at 12 V for the aluminum electrode. Comparative analysis of urea uptake by chemical coagulation and electrocoagulation has been conducted. The most efficient removal was observed using titanium electrodes and this comparison demonstrates that the urea removal efficiency when using the electrocoagulation process is superior to that which is obtained from conventional chemical techniques. The maximum urea removal efficiency using chemical coagulation at a coagulant dose equivalent to the amount of metal dissolution in electrocoagulation was 35%.

Keywords: Electrochemistry; Chemical coagulation; Urea; Voltage; Wastewater

1. Introduction

Urea contaminants have a significant impact on the ecosystem [1]. While urea is not directly toxic in wastewater, its hydrolysis into ammonia remediation and hydrogen production causes toxicity for marine life and animals. Removal of urea from wastewater can be performed using stabilization ponds, physical-chemical techniques including air stripping and ion exchange, and biological methods including trickling filters activated sludge and oxidation ditches. Electrocoagulation of urea has several advantages over these techniques as the technique does not require adding large volumes of chemicals to wastewater or the supply of oxygen to the cathode. Also, secondary pollution is unlikely and the setup is relatively simple. Because of these advantages, electrocoagulation is more attractive than other oxidation processes [2].

The ideas behind electrocoagulation and electroflotation are relatively similar [3–6]. Both techniques depend on destabilizing repulsive forces, which keep the particles in suspension, to remove these particles from wastewater [3,7,8]. Larger particles, or flocs, will be formed as a result of the destabilization of repulsive forces and when this has occurred these flocs made up of suspended particles will settle out of the solution [3,8,9].

During electrocoagulation, a direct voltage is applied to the submerged electrodes and an electrolyte is used as conductive media in the wastewater [3,10]. Three main phases occur during the electrocoagulation/electro flotation process which is: (i) anode oxidation, (ii) cathode gas bubble

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formation, and (iii) sedimentation and flotation of the generated flocs. Once a current is passed through the anodes, oxidation reactions on the anode generate cations and rebate reactions take place on the cathode [3]. Metal hydroxides are formed as results of those cations responsible for effective disruption of suspended solids. On the cathode, the generation of hydrogen gas is a continuous process. The removal of pollutants is achieved by floating or washing out the resulting flocculates [3,7,11].

Removal techniques can include charge neutralization, adsorption, or sweep coagulation [12,13]. In the electrocoagulation process, the reactions applied to the electrochemical process with metal M as electrodes are as follows:

At the anode: $M \rightarrow M^{n+} + ne^-$ for coagulation

At the cathode: $2H_2O + 2e^- \rightarrow H_2 + 2OH^-$ for flotation

Similarly, the urea electro-oxidation reactions occur as follows [14,15]:

At the anode:

$$CO\,(NH_2)_{2(aq)} + 6OH^-_{(aq)} \rightarrow N_{2(g)} + 5H_2O_{(1)} + CO_{2(g)} + 6e^- \quad (1)$$

At the cathode:

$$6H_2O_{(l)} + 6e^- \rightarrow 3H_{2(g)} + 6OH^-_{(aq)} \quad (2)$$

Overall:

$$CO\,(NH_2)_{2(aq)} + H_2O_{(l)} \rightarrow N_{2(g)} + 3H_{2(g)} + CO_{2(g)} \quad (3)$$

Some literature reviews state that the majority of electrocoagulation studies examine the use of nickel, platinum, ruthenium-titanium oxide or boron-doped electrodes in the electrochemical treatment of urea [14,16–18]. There are advantages when using these electrodes, however, with regards to cost, and due to the formation of high volumes of by-products and sludge. Searching for, and finding a new material in the electrochemical creation of coagulants, therefore, is a must. In this regard, this research gives special attention to using titanium and aluminum materials to treat organic polluted liquid effluents such as urea. Indeed, to the best of our knowledge, this is the first case of investigating electrocoagulation treatment of water streams contaminated with urea using titanium and aluminum anodes. In this study, many of the tests and experiments were performed using a bench-scale electrochemical electrodes cell to study the decomposition of urea. The effects of voltage, the composition of the electrolyte, types of the anode, spacing between electrodes, electrolyte type, and the impact of synthetic and real wastewater on the removal rates and pH were investigated.

2. Materials and methods

2.1. Characteristics of wastewater

In this experiment, two different types of wastewaters were used. The first type is synthetic and was composed of urea with 99% purity, and sodium chloride with 99% purity. These chemicals were all of the commercial-grade. The investigated solutions were created by mixing and dissolving urea and sodium chloride salt into distilled water to achieve the required concentration. These chemicals were used without any further purification. The concentration of the urea and sodium chloride salt was 1 and 0.40 g/L, respectively. The second type consisted of real wastewater with the measured characteristics of the wastewater shown in Table 1. Batch experiments were conducted at ambient temperatures ranging from 22°C±2°C.

2.2. Electrocoagulation system setup

The electrocoagulation system was processed in a beaker batch-stirred reactor for 90 min at atmospheric pressure. In each experiment, the beaker was filled with 1 L of the synthetic wastewater containing urea. The cathode and anode were placed parallel to each other and at a spacing of 4.50 cm and connected to a DC power supply (3–12 V, 2 A) in which electrical current was controlled using a variable transformer. A voltmeter and ammeter were used to measure the voltage and the current intensity during the electrocoagulation process. The initial surface area for the titanium, aluminum, and stainless steel (SS) electrodes was 30 cm². The cathode material was SS, and the anode material was either titanium (Ti) or aluminum (Al). The Two electrodes were placed 4.50 cm apart and a urea solution of 1,000 mg/L urea was added. The immersed surface area was 21 cm² for each electrode. The stirring rapidity was maintained at low levels of 100 rpm to prohibit the shearing of the flocs. The electrolysis duration was kept in the range of 5–90 min. The electrodes were washed and cleaned with distilled water. Samples were periodically withdrawn every 5 min for the first 20 min, every 10 min until 40 min, at 60 min, and finally at 90 min. Samples were then filtered to remove any sludge formed during electrolysis. Experiments were performed for each anode's type to examine the effect of decreasing the gap distance between electrodes from 4.5 to 3 cm, electrolyte type, an increase in the surface area of the anode, and finally the effect of using the system with real wastewater.

2.3. Analysis

In order to assess the performance of the treatment process over time, several analytical measurements were performed for Influent and effluent samples. The urea removal efficiency (%) after the process of electrocoagulation was estimated based on the formula: removal efficiency % = $(C_0 - C_e)/C_0 \times 100$ where C_0 represents the influent concentration of urea, while C_e represents the effluent concentration of urea. The sludge formed as a result of the electrocoagulation process was analyzed using Fourier-transform infrared (Nicolet 6700 FT-IR Spectrometer) spectrometer. The anode electrode morphologies were inspected using a scanning

Table 1
Characteristics of real wastewater

Parameter	Unit	Value
pH	–	7.68
COD	mg/L	150
Total dissolved solids (TDS)	mg/L	570

electron microscopy (SEM Quanta FEG 250). Conventional chemical coagulation was examined using jar tests. Titanium tetrachloride and aluminum sulfate were used in chemical coagulation to simulate titanium and aluminum electrodes. Chemical coagulation involved rapid mixing for 90 s at 100 rpm followed by a slow mixing for 20 min at 30 rpm, then a 20 min settling [19]. After than samples were collected for additional analysis [3]. The analysis of the experimental conditions employed is discussed in the results and discussion. Fig. 1 shows the electrocoagulation setup.

3. Results and discussion

3.1. Electrocoagulation voltage dependence

In the electrocoagulation process, a voltage that controls hydrogen bubble generation and the progression of flocs was applied between the anode and cathode electrodes to support the electrodes decay to generate coagulant species [20]. The performance of the titanium and aluminum electrodes was analyzed at 3, 6, 9 and 12 V [21,22]. The removal efficiencies of urea over time for each electrode and the comparison between the two electrodes at the most effective voltage over time are shown in Fig. 2. As shown in these figures, the urea removal rate for all voltages rapidly increased during the first 15 min. After this, the urea removal efficiency reduced as a result of the desorption phenomenon [23]. The oxidation reactions that encourage erosion phenomena may also cause the stabilization of the oxide layers

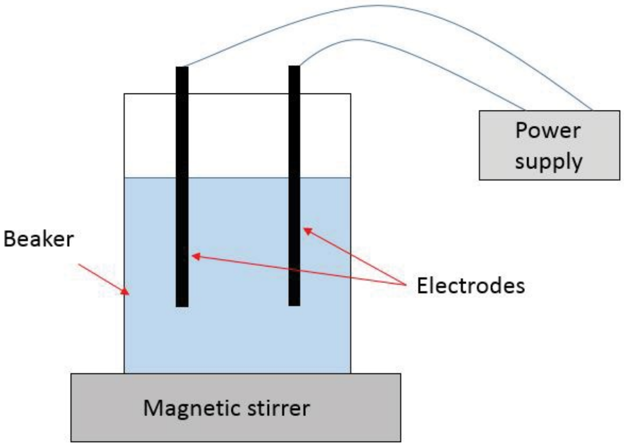

Fig. 1. Schematic diagram of the electrocoagulation unit.

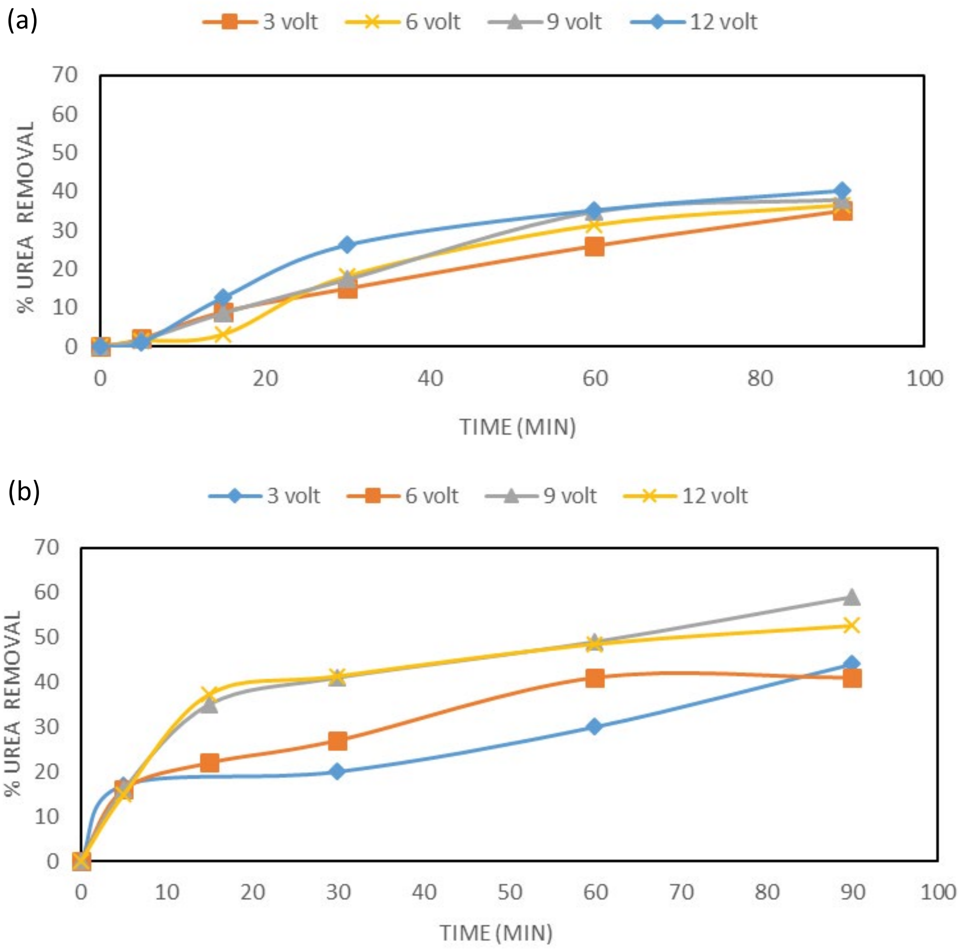

Fig. 2. Efficiencies of urea uptake overtime at various voltage values: (a) Al electrode and (b) Ti electrode.

on the surface of anode electrodes. The presence of these oxide layers cause passivation effects, and therefore reduce the efficiency of electrocoagulation cell [3]. The maximum removal efficiencies for urea occurred after 90 min and its values were 59% at a 9 V for titanium and 40.16% at 12 V for the aluminum electrode. The titanium electrode performed better than the aluminum electrode for all voltages. The number of ions that are released in the form of Ti^{2+} or Al^{3+} is related to the applied voltage and these effects the resulting coagulant rate. Accordingly, there will be an increase in the generation of metal hydroxide as the dissolved metal ions rate in the wastewater is increased leading to an increase in urea removal efficiencies [24]. Adsorption phenomena for pollutants will also occur on metal hydroxides' surface, oxides' surface, and oxyhydroxides' surface [6]. A decrease in pollutants may also occur as a result of the destabilization mechanism which consists of three procedures: compression of the double layer, charge neutralization, and floc formation [19]. Increasing the voltage causes an increase in the number of hydrogen bubbles into solution while reducing their size. This will lead to the removal of the pollutants via the flotation process. In the case of the titanium electrode as an anode, the highest urea removal efficiency value has not been observed at the maximum voltage. The reason behind this phenomenon might be that as a result of the effect of the higher voltage, system turbulence increases. If the turbulence is too high this will lead to an instability of the coagulation process and caused by the fact that particles in the system will not have sufficient time to agglomerate and remove contaminants. Fig. 3 shows the variation in pH values vs. time. In the electrocoagulation process, a minor increase has been observed in pH for all values with the different values of voltage vs. the time. This phenomenon may occur as a result of the reactions that occur at the cathode. During the electrocoagulation process, water molecules collect electrons and transform into hydrogen bubbles and hydroxyl ions, increasing pH or the creation of hydrogen gas at the cathode [20,25]. The increase in pH values while using the titanium electrode as an anode was much higher than those for the aluminum electrode at all voltages. This shows that the aggregate separation of the molecules of water in the case of the titanium electrode was much higher than that for the aluminum electrode. The greater pH values were achieved for both kinds of anodes when the higher values of voltages were used (9 and 12 V) and this may explain the erosion of the cathode which occurred at high voltage

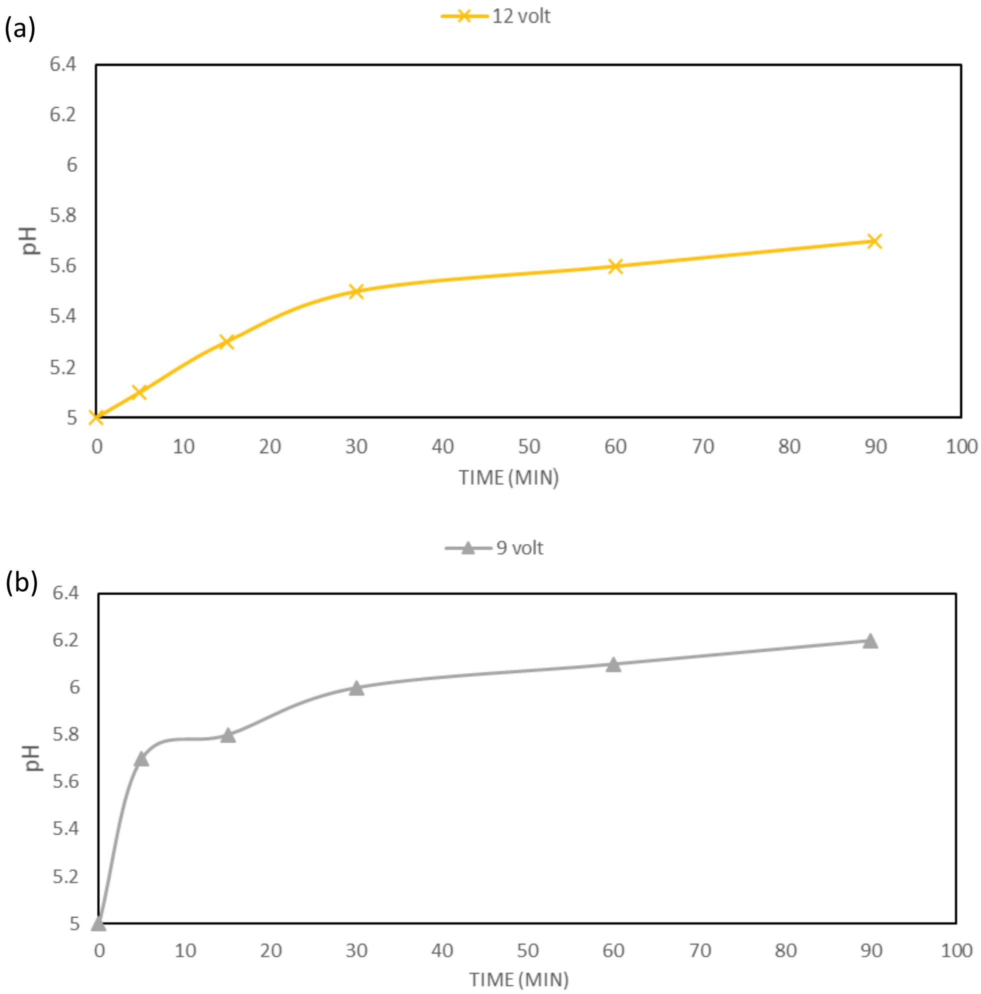

Fig. 3. Variation of pH overtime for the optimum voltage for urea removal: (a) Al electrode and (b) Ti electrode.

due to the severe creation of hydroxide. The pH values for both anodes were observed to be <7. As shown in Fig. 3 there is no major effect of pH on urea removal. It seems that this parameter does not have a significant effect on the treatment process. After 90 min, the color of the solution became white in the case of the aluminum electrode and remained colorless for the titanium electrode.

3.2. Effect of electrode spacing

The spacing between electrodes plays a major role that affects the ohmic potential related to the electrocoagulation cell and the energy depletion [3]. The effects of electrode spacing were investigated at 12 V for the aluminum and 9 V for the titanium electrodes. Fig. 4 shows the removal efficiencies of urea at different gap distances (3.0 and 4.5 cm). The 3.0 cm spacing resulted in removal efficiencies higher than that obtained with 4.50 cm. This phenomenon may happen as a result of the system configuration. As the cross-section of the reactor was circular, the distances between both the electrodes and the distance between the electrodes and the beaker edge was almost the same value when the gap distance was 3.0 cm. This case might achieve the following two results: (i) flocs inside the beaker will have a uniform distribution (ii) floc disruption that may occur during mixing will be minimized. When the electrode spacing is very small, there will be an increase in the electrostatic effect that obstructs particle collision. Additional electrochemically produced gas bubbles lead to turbulence, while the large spacing showed a strong decrease in floc formation [26,27]. The system performance during the pollutant removal was almost identical for all spacing. The progress of pollutants removal was high from for the first 10 min of the experiment, and after that, the removal rate decreased until the experiment had ended. The highest removal efficiencies observed for urea removal at the spacing of 3 cm were 66% for the titanium electrode and 57% for the aluminum electrode. The removal efficiencies when using the titanium electrode were greater than those values obtained from the aluminum electrode. This could be related to the fact that the amount of floc generation when using the titanium electrode was much higher than the amount generated from the aluminum electrode. This demonstrates that the electrocoagulation process with the titanium electrode is better than that with the aluminum electrode.

3.3. Effect of the variation of the surface-area-to-volume ratio

The ratio of the electrode surface area to the solution volume, S/V, is a very important reactor design consideration in electrocoagulation (EC) [25]. Indeed, the percentage of the active surface area of the electrodes with respect to the volume of the treated solution is considered to be a key metric for the measurement of the EC process. It is reported that the current density consumption is reduced when the ratio S/V is increased [25]. This parameter is therefore of great importance as it has a direct effect on the electrocoagulation process performance. The variation of S/V and its effect on the efficiency of urea removal was studied by increasing the surface area of the anode electrode. EC tests were performed using surface areas of 30 and 60 cm^2. Fig. 5 shows the variation in the urea removal efficiency over time for titanium and aluminum electrodes. The removal efficiencies were 63%, and 44% for the titanium and aluminum electrodes, respectively, when the surface area was increased to 60 cm^2. It is observed that the larger was the surface area, the better the removal efficiency. This may motivate the electrical transport, better chemical dissolved of urea and the resistance of the electrochemical cell will be increased causing an increase in the removal efficiency of urea as a benefit of better chemical dissolved.

3.4. Effect of electrolyte type on the electrochemical process

The electrolyte material has a major impact on the treatment process efficiency. Suitable selection and choice of the material, therefore, is very important. Here, the electrolyte materials shall be also non-toxic for human health. $CaCl_2$ and NaCl were selected as electrolyte materials as they are both non-toxic, and regularly available. The electrolyte has an important supporting role during the electrocoagulation

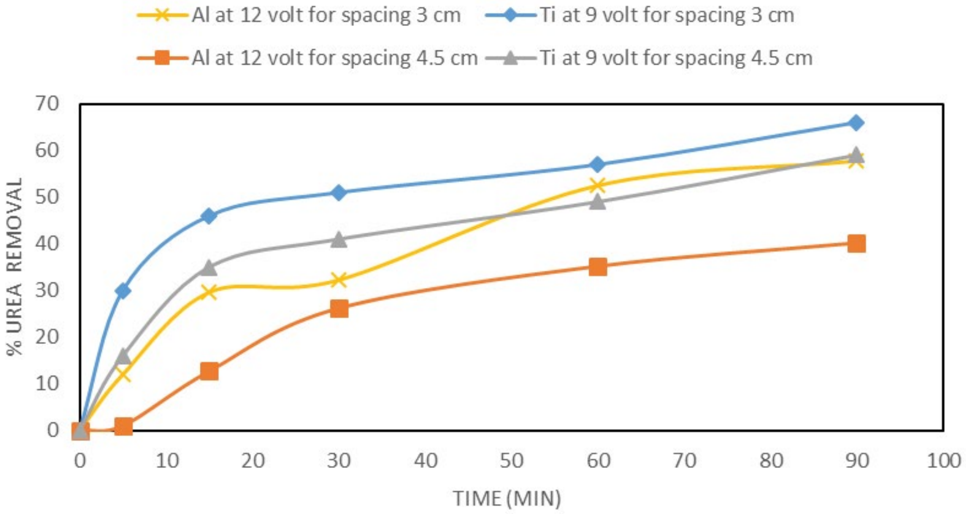

Fig. 4. Urea removal efficiencies overtime at various voltages with a spacing of 3 cm.

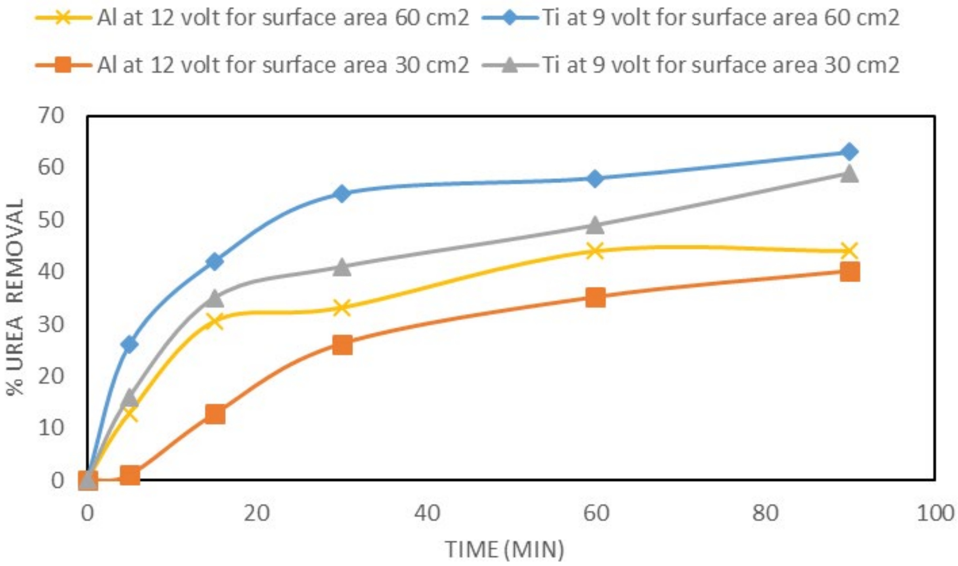

Fig. 5. Efficiencies of urea uptake overtime at various voltages with a surface area of 60 cm^2.

process as it significantly increases the solution conductivity. The type of electrolyte also affects the production of some oxidants in the solution [28]. In this experiment two types of electrolytes have been used, the first electrolyte was sodium chloride (NaCl) and the second electrolyte was calcium chloride ($CaCl_2$). The concentration for both electrolytes was 0.40 g/L. The intention behind changing the type of the electrolyte is to examine the effect of electrolyte type on urea removal during the electrocoagulation process. Fig. 6 shows the removal of urea for time with $CaCl_2$ and NaCl. It was observed that when using $CaCl_2$, the efficiency increased which may be a result of the fact that $CaCl_2$ has a higher conductivity when compared with NaCl. This leads to an enhancement of the chemical dissolution of urea, which in turn encourages electrical transport. The resistance of the electrochemical cell will be increased causing an increase in the removal efficiency of urea as a result of higher dissolution. It is suggested that chloride ions may be responsible for the corrosion of the electrodes [29]. The removal efficiency was 67% and 46% for titanium and aluminum electrodes, respectively.

3.5. Characterization of the generated by-products from the electrocoagulation using FTIR

To investigate and clarify the by-products generated from the electrocoagulation process, FTIR spectral analysis was conducted to analyze and characterize those by-products.

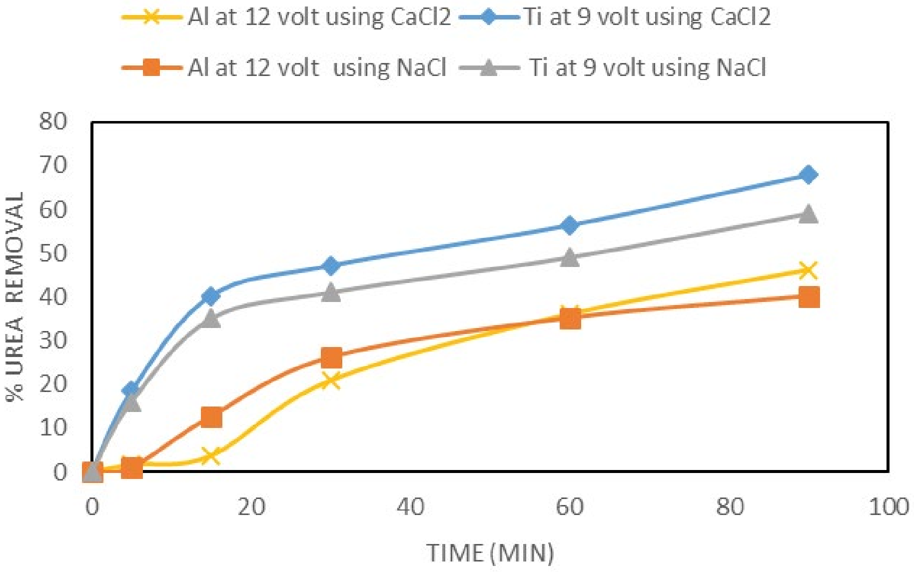

Fig. 6. Efficiencies of urea uptake overtime at various voltages using different electrolytes.

The analysis of two sludge samples for each electrode was carried out with one reference sample which contained only the electrolyte and a second sample which contained both the electrolyte and urea. Figs. 7 and 8 show the FTIR spectra for the generated sludge samples at 9 and 12 V using titanium and aluminum electrodes, without and with urea, respectively. Spectroscopic changes were observed for the sludge samples. The FTIR spectra show that the fingerprints of these two samples were not identical between 350 and 4,000 cm^{-1}, providing evidence for the existence of diverse compounds. These diverse compounds occurred as a result of the dissolution of either titanium or aluminum electrodes throughout the electrocoagulation progression [3]. The FTIR spectral analysis for two generated sludge samples shows a

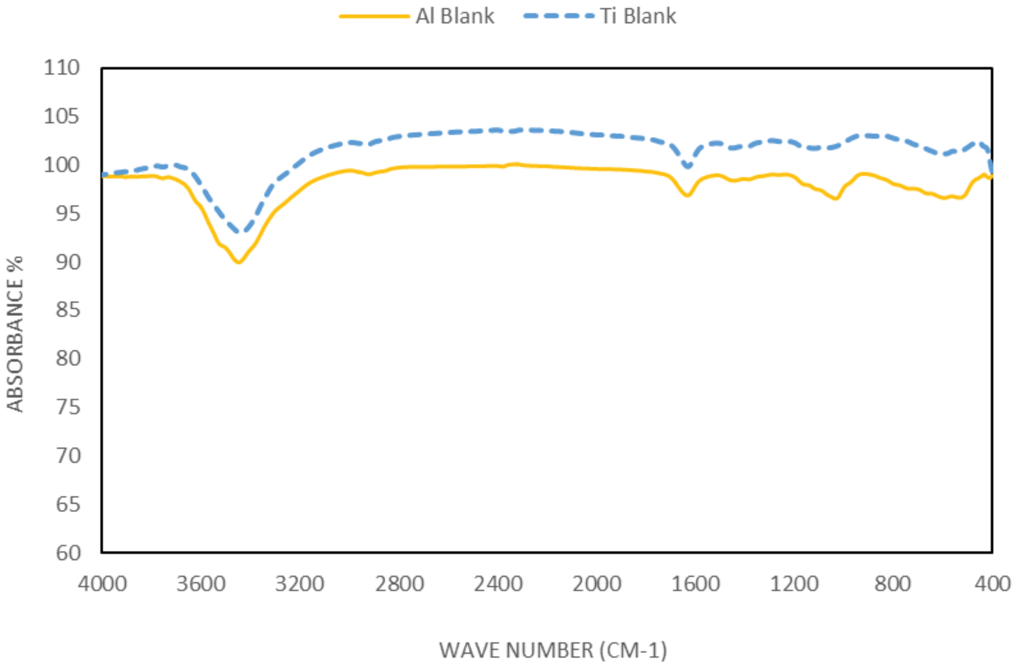

Fig. 7. FTIR spectra of the by-products without urea.

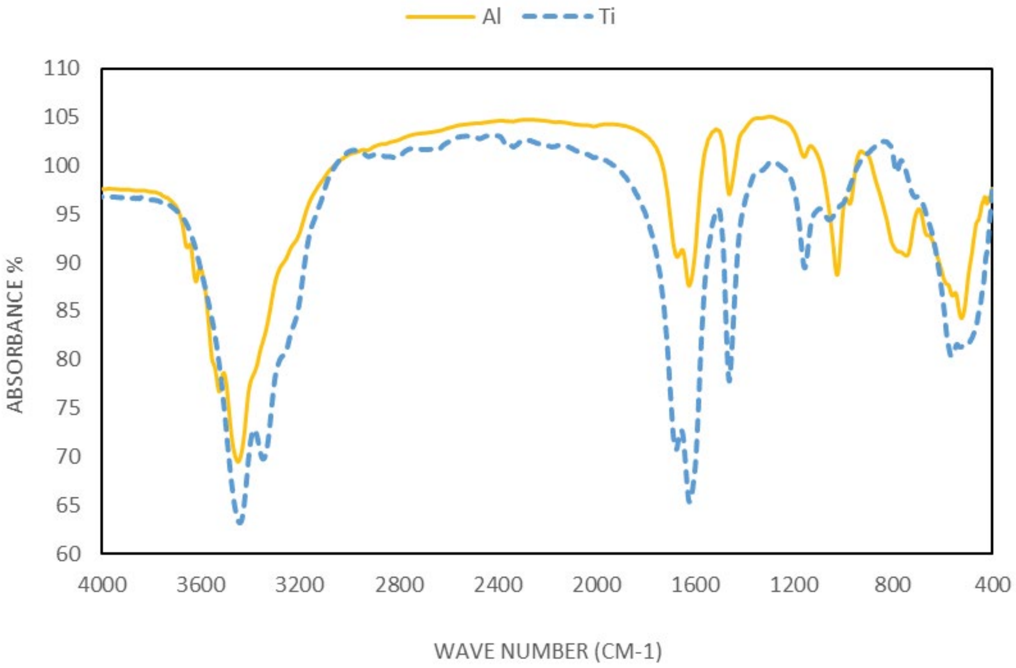

Fig. 8. FTIR spectra of by-products with urea.

wide-ranging and extreme band between 1,350 and 1,850 cm^{-1} which is repeated between 2,850 and 3,850 cm^{-1}, indicating the existence of an OH group. The existence of the OH group improved the adsorption of the counter ions throughout the settling process. This demonstrates that adsorption is considered as one of the removal techniques during the electrocoagulation process.

3.6. Electrode morphologies

The morphology of the titanium and aluminum electrodes was inspected before and after the electrocoagulation at a voltage of 9 V for titanium and 12 V for aluminum. Fig. 9 shows the SEM image of the electrodes before and after the treatment process. For each of the titanium and aluminum electrodes, erosion has occurred on the anodes as a result of the electrocoagulation experiment, demonstrating that the treatment has occurred. The external surface was inspected using SEM and it was found that the surface of the titanium electrode consisted of cracks and large cavities. However, in the case of the aluminum electrode, the surface was coarse and consisted of small cavities. The presence of a large number of cracks and cavities on the surface of anodes results from the exhaustion of minerals on the active side of anodes resulting from the formation of oxygen at electrodes' surfaces [30]. The erosion seen on the titanium electrode can be considered to be regular erosion, while the erosion in the case of the aluminum electrode is irregular due to the surface pitting. Regular erosion is considered better than irregular erosion here as it is considered easier to be expected.

3.7. Performance of chemical coagulation

Experimental work looking at the impact of different dosages (10–160 g/L) of titanium tetrachloride and aluminum sulfate on the performance of urea removal by conventional coagulation methods using titanium tetrachloride and aluminum sulfate salts has been carried out [19]. Fig. 10 shows the jar tests using aluminum sulfate and titanium tetrachloride. Fig. 11 shows the performance of chemical coagulation for urea removal at different coagulant doses. The urea removal efficiencies were found to increase as the doses of aluminum sulfate were increased demonstrating better performance than titanium tetrachloride. The maximum urea removal efficiency was 60% for both salts when using a high coagulant amount of 160 g/L. The obtained values are

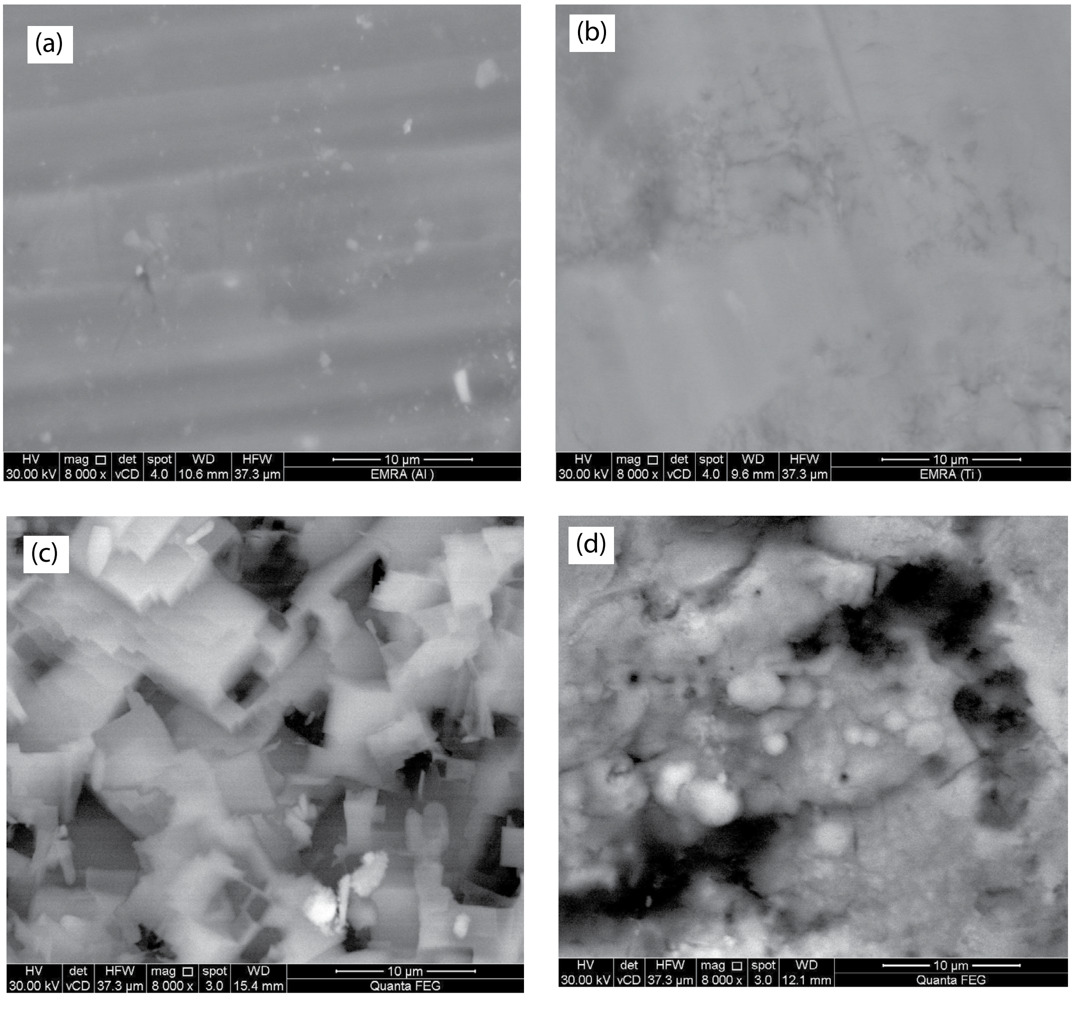

Fig. 9. SEM images: (a) Al electrode prior to the treatment, (b) Ti electrode prior to the treatment, (c) Al electrode following the treatment, and (d) Ti electrode following the treatment.

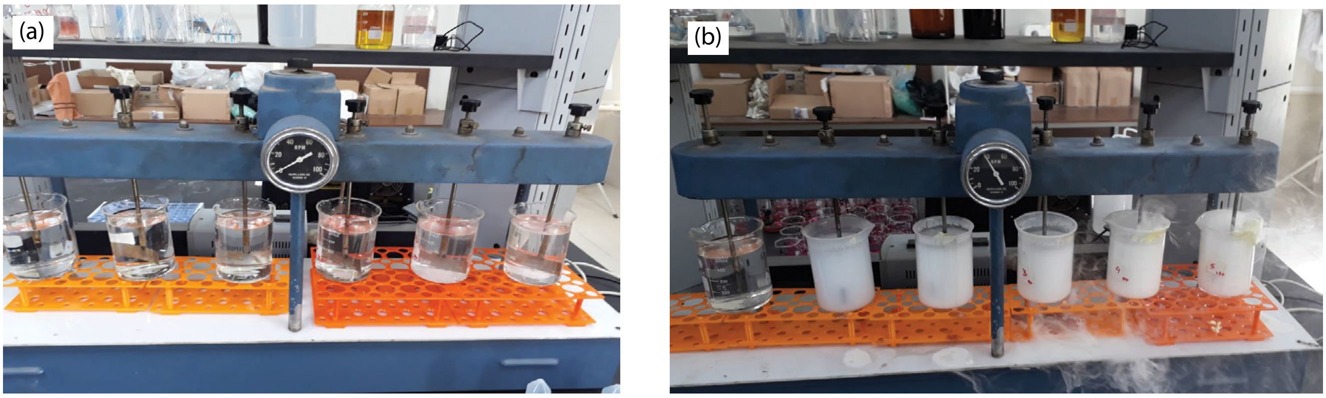

Fig. 10. Jar test using: (a) aluminum sulfate and (b) titanium tetrachloride.

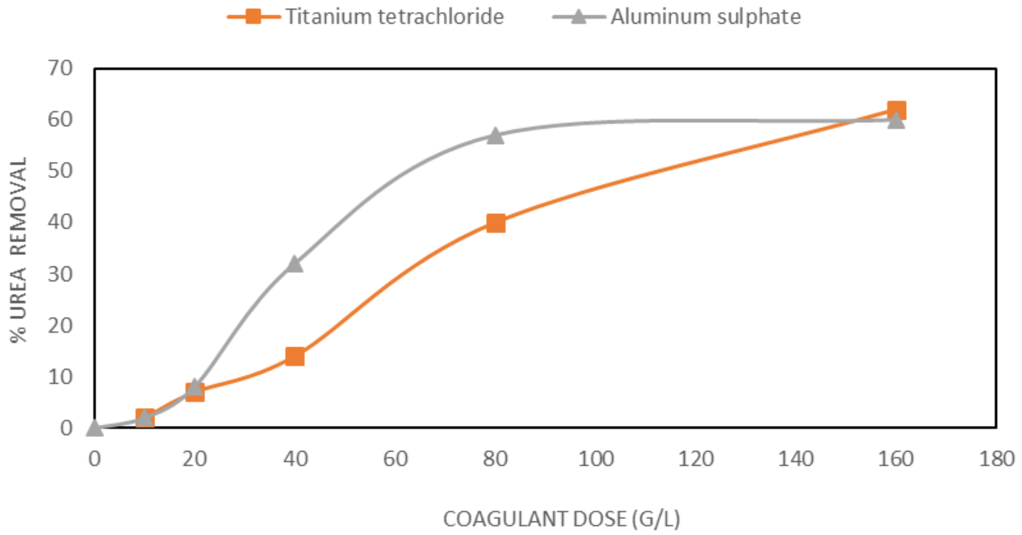

Fig. 11. Urea removal efficiencies for varying coagulant doses.

less than those obtained from the electrocoagulation process, demonstrating that the electrocoagulation process is more efficient than the chemical coagulation with respect to urea treatment with a titanium anode. In the case of the aluminum anode, however, the chemical coagulation is better than electrocoagulation for urea treatment.

3.8. Performance of EC with real wastewater

The effect of electrocoagulation was analyzed using real wastewater for both the titanium electrode at 9 V and the aluminum electrode at 12 V. The maximum removal efficiencies of chemical oxygen demand (COD) after 90 min were 30% and 22% for titanium and aluminum electrodes, respectively. When using the titanium electrode, the removal efficiency of the COD was higher in comparison to the aluminum electrode for the entire duration of the process. Fig. 12 shows urea removal efficiencies over time for Al and Ti electrodes. The titanium electrode performed better than that of the aluminum electrode in urea removal with the maximum rate of urea removal efficiency after 90 min observed to be 30% in the case of titanium and 27% in the case of aluminum. As the generated amount of metal hydroxide increased, the rate of metal ions that settled into the wastewater increased and therefore, the COD removal efficiency increased. On the metal oxides' surface, hydroxides' surface, and oxyhydroxides' surface, the adsorption process of pollutants also occurs [24,31]. The electrocoagulation process causes an increase in the formation of hydrogen bubbles with a reduction in the bubble sizes. Over time pH values increased during the electrocoagulation process which may be a result of many reactions that are occurring at the cathode during the electrocoagulation process. Water molecules withdraw electrons and can then be divided to form hydroxyl ions and hydrogen bubbles which has the effect of increasing pH values. The pH value for both titanium and aluminum electrodes is similar after 90 min which may be attributed to the equal rate of water molecule separation for both materials. The dissociation of titanium produces divalent Ti^{2+} ions which form titanium hydroxide that is thermodynamically favored at a pH of more than 7. The dissociation of the aluminum produces $Al(OH)_3(s)$ at pH ranges between 4 and 9.5. This hydroxide formation is favored at higher pH values and acts to trap the colloids/contaminants, sweeping them away from

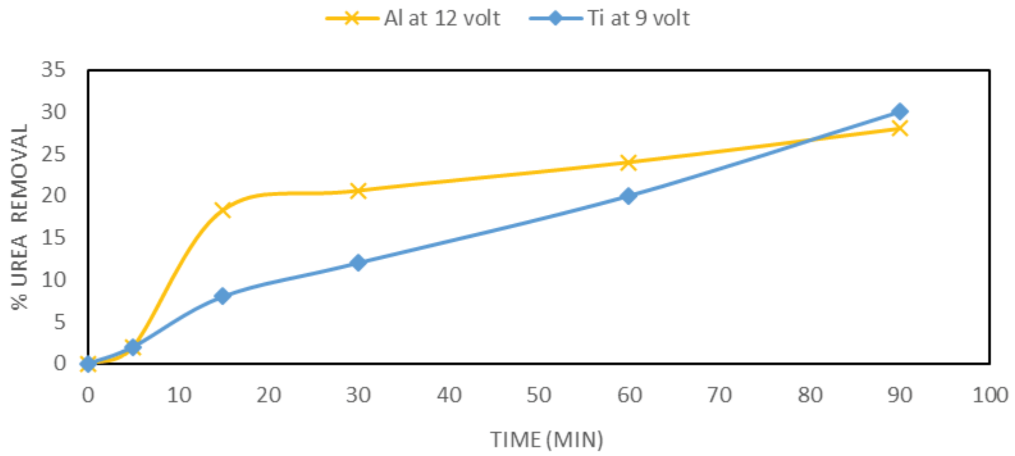

Fig. 12. Urea removal efficiencies over time for Al and Ti electrodes.

the solution like the coagulation process via settling and resulting in a higher COD removal. The rate of urea removal efficiency was less than that observed when using synthetic wastewater which may be due to the presence of other pollutants in the real wastewater.

4. Conclusions

This research was conducted to study the removal of urea using the electrocoagulation technique. Two types of electrodes (titanium and aluminum) were studied. The results presented indicate that urea removal efficiencies are much higher when using the titanium electrode than that found when using the aluminum electrode. The maximum urea removal efficiency when using a titanium electrode was around 60% at 9 V, while the maximum removal efficiency for the aluminum electrode was 40%, obtained at a 12 V. Solution pH was in the range of 6 to 8 for all voltages over time. Higher removal efficiencies were observed with an electrode spacing of 3 cm. The experiment shows that when using $CaCl_2$ as an electrolyte, removal efficiencies were greater than those obtained when using NaCl as an electrolyte. FTIR spectral analysis was conducted to analyze and characterize the by-products of the sludge produced at 9 V for titanium and 12 V for aluminum. FTIR measurements showed that sample spectral finger-prints were not similar, proving the existence of diverse compounds. The morphology of both the titanium and aluminum electrodes were inspected before and following electrocoagulation using the SEM. Erosion occurs on the anodes as a result of the electrocoagulation process, demonstrating that the treatment has occurred. The erosion in the titanium electrode was seen to be regular erosion, while the erosion in the case of the aluminum electrode was irregular, with pitting on the surface. Electrocoagulation was seen to be more efficient than the chemical coagulation process for urea treatment in the case of the titanium anode but for aluminum, this trend was reversed. The rate of urea removal efficiency with real wastewater was less than that observed when using synthetic wastewater. The generation of oxide layers that settled on the surface of anode electrodes from the oxidation reactions that encourage the erosion produced passivation effects which have a negative impact on the efficiency of the electrocoagulation cell.

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