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Green and Efficient Removal of Toxic Lead and Chromium from Synthetic Leather Tannery Wastewater using Electrocoagulation

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ABSTRACT

Most of leather tanneries in Pakistan dispose of tannery effluent untreated in sewage lines causing serious harmful environmental impact. Some of tanneries use conventional activated sludge process releasing large quantity of sludge and not effective for treating heavy metal in leather tanneries. The manuscript deals with implementation of electrocoagulation technology for removal of Cr and Pb from Leather tannery effluent, investigation of operating parameters current density, treatment time and pH on removal efficiency. Electrodes was made of Iron having dimension 9.5 cm x 0.6 cm x 0.4 cm and affective surface area of electrode was 76 cm^2. After conducting different experiments at various operating conditions, pH 3.5 and current density 18.8 milliamp/cm^2 was found to be optimized condition for treating 1 liter of synthetic leather tannery effluent. The maximum removal efficiency of chromium and lead was obtained 87.8 % and 62.7 % respectively at pH 3.5 and current density of 18.8 milliamp/cm^2. The EDX analysis of sludge shows presence of chromium, lead and iron element in sludge and FTIR analysis shows the presence of hydroxyl functional group in sludge, it is strong evidence of iron polymer complexes formation with chromium and lead during treatment of synthetic leather tannery wastewater. Both of these analysis show strong evidence of effective electrocoagulation process. It was proved that electrocoagulation can effectively treat synthetic leather tannery effluent. The SEM analysis shows production of coagulant on surface of electrode.

Keywords: Electrocoagulation; current density; fourier transform infrared radiation spectroscopy; emission dispersive x-ray spectroscopy

INTRODUCTION

Pakistan stands at 21st position in world market contribution of leather goods (Hashmi, Dastageer et al. 2017). The tanning of leather involved various processes such as soaking, fleshing, unhairing and liming, splitting, de-liming and bating, pickling, tanning, alkalizing, bleaching and finishing (Zhao, Wu et al. 2022). Leather industry known to be highly water intensive because 1 ton of skinned hide require 4000 liters of water(Ramesh and Thirumangai 2014). The tanning process used alkaline chromium sulfate solution due to this effluent of tanning industry contain large quantity of chromium ion 2-4 g/l and large quantity of organic pollutant. According to International Anti-Cancer Research Institute identified to be carcinogenic and baneful impact environment(Li, Yang et al. 2019). The effluent generated from leather tannery industry causes dark brown colored waste with high contents of COD, BOD, TDS, chromium, phenolic, high PH and pungent odor(Zhao, Wu et al. 2022). There is less attention has been given to effluent released from tanneries and causing serious effects on community living in locality, workers, crops, soils, aquatic life and drinking water. So it is need by government to focus on environment related issues so that leather products compete in international market and fulfill international environmental regulations. There are various technologies used in past to treat the industrial effluent such as precipitation (BrbootI, AbiD et al. 2011), adsorption(Vojoudi, Badiei et al. 2017), bio-adsorption(El-Sayed and Nada 2017), ion exchange(Zewail and Yousef 2015), activated sludge pond and aeration pond to treat the industrial effluent but still effluent contain some amount of chromium and lead in effluent and sludge also contain chromium it can cause serious environmental problems. Eco system and natural resources, particularly water and soil, are significantly impacted by chromium pollution, which is a serious threat to the environment. Increased levels of chromium accumulation in human and animal tissues could result from excessive exposure, which would be harmful to health (Liugė and Paliulis 2023, Raj and Das 2023). Workers in industries where they are exposed to toxic metals on aregular basis experience asthma and respiratory disorders as a result of high lead exposure, as evidenced by lead concentrations in urine and serum. Karachi is also hub of tanneries in Pakistan (Hashmi, Dastageer et al. 2017) but still most of tanneries do not have effluent treatment plant and they waste large quantity drinking water to dilute it and release into water sewage lines. It has been determined from various literature that majority of researcher have used electrochemical treatment for leather tannery effluent (Ghafoor 2015). Electrocoagulation one of the modern electrochemical techniques gives efficient results as compared with other treatment technologies. This work has shown that electrocoagulation is promising technology for treatment of leather tannery effluent (Haan, Fen et al. 2018), it is cost effective and by product generated in form of sludge can also be utilized for bricks formation and no waste has been remained after treatment through electrocoagulation process (Hasan, Hashem et al. 2022). The aim of present study was treatment of leather tannery effluent in efficient and green manner and investigating the effect of operating variable current density, treatment time and pH on removal efficiency of chromium and lead from synthetic leather tannery industry wastewater.

MATERIAL AND METHOD

COLLECTION, PRESERVATION AND CHEMICAL CHARACTERIZATION OF WASTEWATER

The wastewater was collected from Combine Effluent Treatment plant Pakistan Tannery Association (PTA) raw inlet located in Korangi industrial area Karachi Pakistan. The sample was collected in polyethylene bottle and it was added with two to three drops of nitric acid for preservation purpose. The sample was analyzed for chromium and lead using Perkin Elmer A Analyst 700, pH and electrical conductivity EC by PHS-3BW Microprocessor pH/mV/ Temperature meter, BOD,COD, TDS and TSS (material 1995, Lenori S. Clasceri 1999) by standard method. The results of characterization is shown in Table 1.

TABLE 1. Characterization of Leather Tannery effluent

S. No	Parameter	Quantity (mg/litre)	SEQS limit (mg/litre) (Sindh Environmental Gazette)
01	TDS	8500	3500
02	TSS	1352	400
03	pH	7.5	6-9
05	Chemical oxygen Demand (COD)	470	400
06	Chromium	110	1
07	Lead	20	0.5

The synthetic leather tannery wastewater was prepared by taking pure chemical potassium dichromate ($K_2Cr_2O_7$) and lead Nitrate $Pb(NO_3)_2$ of Sigma Aldrich Company. The synthetic wastewater was prepared by adding 0.6321 grams of potassium dichromate and 0.0821 Lead Nitrate in 1 liter de-ionized water. The pH of synthetic solution was adjusted by adding (1M) sodium hydroxide solution and (1M) H_2SO_4 solution in de-ionized water. The 2 % HCl solution was prepared for washing of electrodes after conducting experiment.

EXPERIMENTAL SETUP

The electrocoagulation cell was made from acrylic material rectangular in shape having (Length × Width × Height) 9.5 cm× 0.6 cm × 0.4 cm. The further details of electrocoagulation cell are presented in Table 2 and experimental is shown in figure 1. The total volume of wastewater handled in this cell was 1 dm^3. The electrodes used were made of iron materials, pH of synthetic wastewater was maintained by using 2% H_2SO_4 solution and 5% sodium hydroxide solution (NaOH). After completion of each experiment the plates of material was washed with dilute HNO_3 and dried at 105 ^oC in oven and then used for more experiments. Each batch consists of four plates in electrolytic reactor anode and cathode. The current was supplied by DC supply having range 0-30 volt and 0-5 ampere. To see the effect of electrodes after proper time interval10 ml of sample was taken out from Electrocoagulation cell and then filtered using Whatmann filter paper no.42. for separation of sludge from effluent. The filtrate concentration of was determined for chromium and lead removal by atomic absorption spectrophotometer Perkin Elmer A Analyst 700. The residue remained in filtrate was dried in oven at temperature of 105 ^oC to remove water. The remaining solid known as sludge was analyzed for chromium and lead concentration by dissolving aquarezia slow heating at about 90 °C until the entire residue get dissolved completely. After suitable dilution the residual contain of chromium and lead was determined by atomic absorption spectrophotometer.

RESULTS AND DISCUSSION

PRINCIPLE OF ELECTROCOAGULATION CELL

The electrocoagulation process involves various mechanism such as anodic oxidation, cathodic reduction, coagulation, electrophoretic migration, electrostatic attraction and adsorption. The mechanism of coagulant production in electrocoagulation is through electrode. During the electrocoagulation of iron electrode the Fe+2 ions are released in environment at anode by passing electric current through electrocoagulation reactor. Table 2 shows the detailed description of electrocoagulation experimental setup and Figure 1 shows the diagram of experimental setup of electrocoagulation cell.

$$Fe \rightarrow Fe^{+2} + 2^{e-} \qquad (1)$$

TABLE 2. Characteristics of Electrocoagulation cell

Electrodes	
Material (anode and cathode)	Iron
Shape	Rectangular plate
Size	9.5 cm× 0.6 cm× 0.4 cm
Plate arrangement	Parallel
Effective one electrode Surface area	19 cm^2
Total Effective surface area	76 cm^2
Reactor characteristics	
Material	Acrylic
Reactor Type	Batch Mode
Dimension	30 cm height and 6.5 cm diameter
Volume	1 litre
Electrode gap	0.37 cm
Power supply	DC
Voltage	0-30 volt
Current	0-5 Amp

Hydrogen gas is generated along with hydroxide formation. It has been determined that OH- production increases with an increase in pH during electrocoagulation process.

$$Fe^{+2} + 2OH^{-} \rightarrow Fe\,(OH)_2 \qquad (2)$$

$$2H_2O + 2^{e-} \rightarrow 2OH^{-} + H_2 \qquad (3)$$

In the presence of high pH various monomeric and polymeric species Fe $(OH)_2^{+}$,Fe $(OH)_2^{+2}$, Fe $(OH)_2^{+4}$, Fe $(OH)_4^{-}$·Fe $(H_2O)_2^{+}$ (Teixeira and Rosa 2006, Prajapati and Chaudhari 2014) etc. At high basic condition, the formation of insoluble Fe$(OH)_2$ can be written as follows insoluble hydroxides are settled due to its gravity.

Under basic condition

$$2H_2O + 2^{e-} \rightarrow 2OH^{-} + H_2 \qquad (3)$$

$$CrO_{4\,(aq)}^{-2} + 3\,Fe_{(aq)}^{+2} + 4H_2O_{(l)} + 4OH_{(aq)}^{-} \rightarrow 3Fe(OH)_3 \qquad (4)$$

Under acidic condition

$$Cr_2O_{7\,(aq)}^{-2} + 6Fe_{(aq)}^{+2} + 14\,H_{(aq)}^{+}$$

$$6\,Fe_{(aq)}^{3+} + 2\,Cr_{(aq)}^{3+} + 7\,H_2O_{(l)}$$

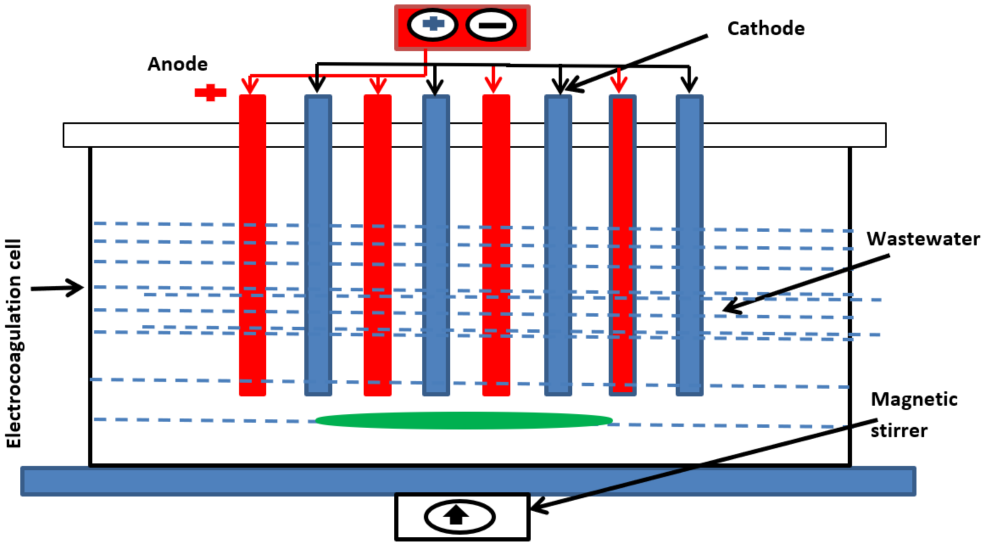


FIGURE 1. Diagram of Experimental Assembly of Electrocoagulation cell

EFFECT OF PH

The initial pH of solution has main impact on the process of electrocoagulation(Kumar Prajapati and Kumar Chaudhari 2014) . The experiment was performed at different pH range from 1.5 to 9 at current density of 6 mA/ cm^2 and treatment time 90 minutes. The initial concentration of chromium and lead was 110 mg/liter and 20 mg/liter. Chromium concentration was decrease from 110 ppm to 44.33 ppm, 13.42 ppm, 63.36 ppm, 86.45 ppm, 98.56 ppm, 97.46 ppm at PH 2, 3.5, 5, 6.5 ,8 and 9.5 respectively. At same time concentration of lead decrease from 20 ppm to 5.76 ppm 1.06 ppm, 6.21 ppm, 6.62 ppm, 6.77 ppm, 6.87 ppm at pH of 2, 3.5, 5, 6.5, 8, 9.5 respectively. The result of effect of pH on removal efficiency of chromium and lead from synthetic leather tannery wastewater is given in figure 2(a) and (b) respectively. It can be seen from result Cr remediation increase from pH below 7, consequently decreases above 7. The maximum removal percentage of chromium was 87.8 % at pH 3.5 because of presence of high hydrogen ion concentration leading to reduction of Cr(+6) to Cr(+3) than chromium (+3) will be easily precipitated by forming complex with hydroxide formation at cathode(Prajapati, Sharma et al. 2016). Lead removal concentration decreased from 20 ppm to 6.87 ppm, 6.62 ppm, 6.21 ppm, 5.76 ppm, 1.06 ppm at PH 2, 3.5, 5, 6.5,8 and 9.5 respectively. It can be seen from graph lead removal performance was higher in first 60 minutes and then decreases with slow rate. The maximum removal of lead was 94.6 % at pH 9.5 and decreases when pH drops below 7(Khosla, Venkatachalam et al. 1991). Increase in removal efficiency at pH above 7 was due to formation of ferric cations produced at anode formed polymer species of Ferric with hydroxyl ion generated at cathode than it will attract lead pollutant and after neutralizing precipitated and generation of hydroxyl ion greater at this pH range. Rate of removal efficiency was greater in first 60 minutes because of less resistance of external ions such as SO^{4-}, Na^+ for diffusion of ions to form polymer complexes of iron hydroxides(Shakir and Husein 2009). The decrease in removal efficiency of Pb at pH below 7 was attributed to formation of amphoteric nature of $Fe(OH)_3$ which lead to soluble Fe^{+3} cations when the initial pH is below 7.

Amount of energy consumed for removal of chromium and lead from leather tannery effluent is important parameter is design point of view. It can be calculated Equation (6).

$$Energy\ Consumption = \frac{VIt}{treated\ volume\ (dm3)} x0.001 \qquad (3)$$

(Nippatla and Philip 2020)

Where V is cell voltage in volts, I is current in amperes (A) t is electrolysis time (h). The amount of energy consumed was determined at different pH from 2 to 9.5 current density 15.3 A/m^2, EG= 3 cm and treatment time 90 minutes. It can be determined from result that energy consumption increases with increase in pH. The energy consumption determined from formula is given by equation 19. It was determined from results that energy consumption will be increase in pH, this may be due to production of flocks formation which covers the surface near electrode that will increase the resistance for current to flow through solution in this case the voltage will be required to increase and power consumption will also be increased. The amount of energy consumed was determined to be 0.58 KWh/m^3.

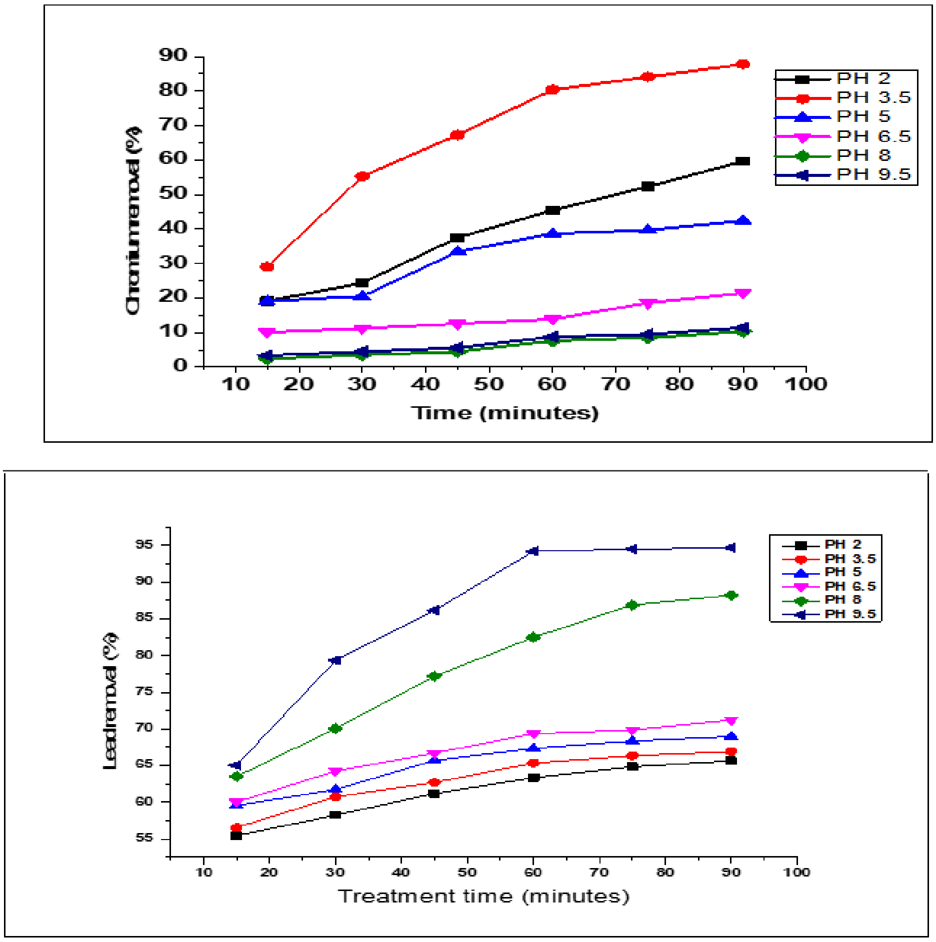


FIGURE 2. Effect of pH on (a) Cr (VI) reduction and (b) Pb reduction (Cr initial concentration = 110 ppm, Lead initial concentration = 20 mg/dm^3, current density = 18. 8 mA/cm^2, EG = 3cm

EFFECT OF CURRENT DENSITY

The current density is one of the important parameter in electrocoagulation process on which reaction rate depend (Kumar Prajapati and Kumar Chaudhari 2014, Prajapati, Sharma et al. 2016). Current density plays major role in generation of coagulant amount at anode, produce bubbles and size distribution, so the current density affect was observed at optimum pH 3.5.The concentration of Cr(Vi) and Pb as function of operating time and current density is given in figure below. The Cr (VI) concentration decreased from 110 to 1.06 mg/dm3 and Pb from 20 to 2.12 mg/dm3, when current density was 18 milliamp/cm2. The current density was changed from 12.8 to 21.0 milliamp/cm2.When current density and treatment time increases the removal of chromium and lead also increases due to production of more iron coagulant ions at anode. It was observed that increase in removal percentage was become lower after 60 minutes because initially there was more coagulant particles to remove pollutant after that there is some resistance due because of formation of layer of iron oxide on surface of iron electrodes. The figure 3(a) and (b) shows the removal of chromium and lead from synthetic wastewater at various current densities. Faraday also proves this relation stating that as current density of synthetic wastewater increased it will increase production metal-hydroxide cationic complexes and finally chromium and lead removal efficiency increases. The affinity of chromium and lead is good for ferrous hydroxide complexes(Golder, Samanta et al. 2007). However, an increase in current density beyond the optimal value has 18.3 milliamp/cm2 has no effect on pollutant removal efficiency because of side reaction and fouling on metal surface.

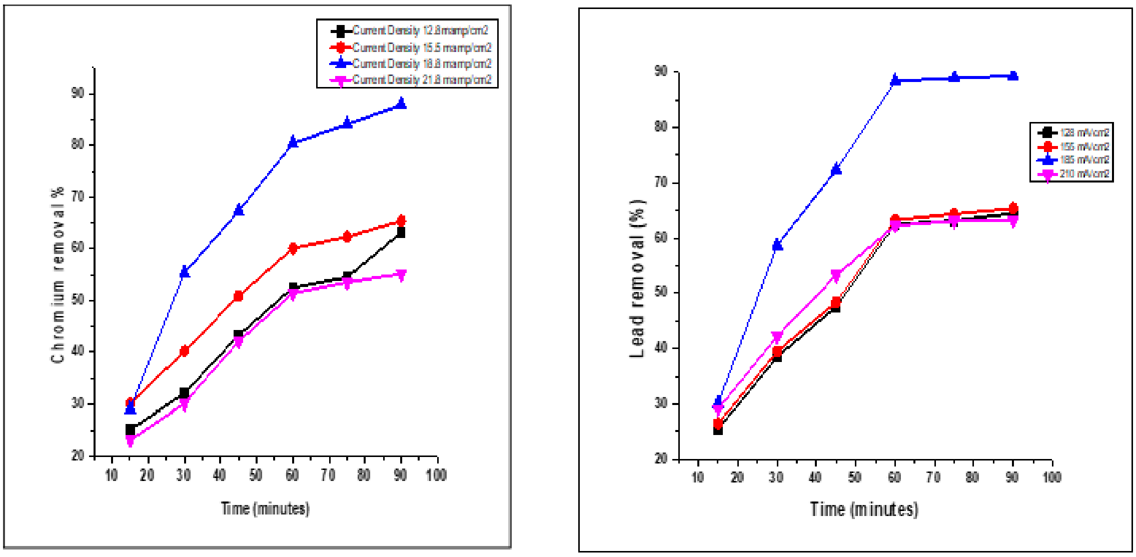


FIGURE 3. Effect of current density on (a) Cr (VI) reduction and (b) Pb reduction (operating conditions Cr initial concentration = 110 ppm, lead initial concentration = 20 ppm, pH= 3.5, EG = 3 cm)

MATERIAL BALANCE FOR PB AND CR (VI)

After completion of electrocoagulation experiment the concentration of chromium and lead was determined in residual synthetic wastewater and sludge. The sludge was separated from residual synthetic wastewater by filtration. The filtrate was first dried in oven at a temperature of 105 ^{0}C until sludge has been completely dried. Dried sludge was analyzed for EDX analysis to determine concentration of chromium and lead. The FTIR analysis was used to see the evidence of electrocoagulation process. The table 3(a) and 3(b) shows there is no more than 2.5 % error in material balance of chromium and lead from synthetic wastewater. It can be shown from material balance at pH 3.5 chromium and lead in sludge is 95.9 mg/dm^3 and 13.1 respectively.

SLUDGE ANALYSIS

The difference between SEM image before treatment is shown in figure 4(a) and SEM image after treatment is shown in figure 4(b) after experiment shows there is coagulant particles on surface of iron electrode after experiment. It shows evidence of metal electro-dissolution in synthetic wastewater, same behavior was observed by golder at al (2007). The figure 4(c) shows EDX (Emission dispersive X-Ray Spectroscopy) shows the presence of lead and chromium in sludge. It is evidence of adsorption of chromium and lead on surface of polymer complex of metal hydroxide. Figure 4(d) shows FTIR spectrum of chromium-lead iron hydroxide. The sharp and strong peak at 3182.24 shows cm^{-1} shows the starching vibration in Iron hydroxide structure. It is evidence for removal of chromium and lead by Iron hydroxide polymer complexes.

TABLE 3(a). Material balance for chromium (vi) and lead at different pH

pH	Initial Cr concentration in Tannery effluent (mg/dm^3)	Cr in sludge (mg/dm^3)	Cr in solution (mg/dm^3)	Total Cr in solution and sludge (mg/dm^3)	% Error
Material balance for chromium (vi) at different pH					
2	110	64.8	44.33	109.13	0.79
3.5	110	95.9	13.42	108.58	1.29

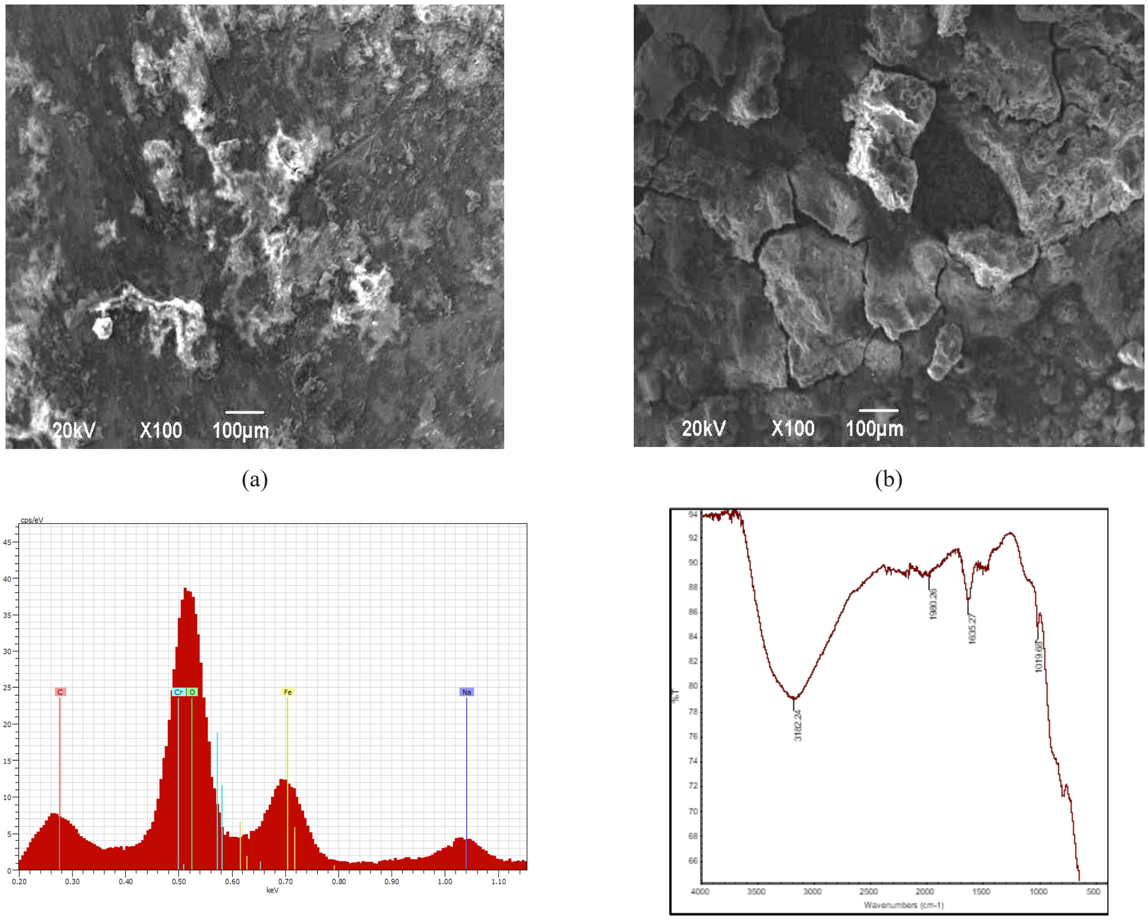
continue ...

... cont.

5	110	46.1	63.63	109.73	0.24
6.5	110	23.1	86.36	109.46	0.24
8	110	11.1	98.56	109.66	0.49
9.5	110	12.1	97.46	109.56	0.4

TABLE 3 (b). Material balance of lead removal of synthetic wastewater at different pH

pH	Initial Lead concentration Tannery effluent(mg/dm^3)	Lead in sludge (mg/dm^3)	Lead in solution (mg/dm^3)	Total Lead in solution and sludge (mg/dm^3)	% Error
2	20	13.04	6.68	19.72	1.4
3.5	20	13.1	6.62	19.72	1.4
5	20	13.4	6.2	19.60	2
6.5	20	13.74	5.76	19.50	2.5
8	20	17.45	2.35	19.80	1
9.5	20	18.84	1.06	19.9	0.5



(a) (b)

(c) (d)

FIGURE 4(a). SEM analysis of electrode before experiment (b) SEM analysis of electrode after treatment (c) EDX analysis of sludge (d) FTIR analysis of sludge

TABLE 4. Comparison of current work with other researcher work

Type of sample	Electrode	Current Density (milliamp/ cm^2)	pH	Initial concentration in ppm	Treated volume in ml	Time minutes	% Removal	ENC (KWH/ m^3)	Ref
Electroplating Effluent	Fe	73.5	3.5	Cr=55.3, Pb=3.5	800 ml	90	Cr=91.7, Pb=91.3	17.14	(Sharma, Chaudhari et al. 2020)
Aqueous solution	Al	9.14 volt	4.2	Cr=200	5000	30	91	3.536	(Zaroual, Chaair et al. 2009)
Ground water	Iron	7.94	8	Cr=0.2	2651	5	100	0.6	(Hamdan and El-Naas 2014)
Synthetic wastewater	Aluminum	48.78	5.1	Cr=8064	1000	60	90	3.906	(Golder, Samanta et al. 2007)
Tannery wastewater	Aluminum	200	3.8	Cr=6000	2000	360	94	35	(Elabbas, Ouazzani et al. 2016)
Synthetic Tannery wastewater	Iron	188	3.5	Cr=110, Pb=20	1000	90	Cr=87.8 Pb=89.7	0.58	Present work

CONCLUSION AND FUTURE RECOMMENDATIONS

This research work determines that electrocoagulation is one of effective, economical and eco environment process for treatment of leather tannery effluent. Best removal efficiency obtained was chromium 87.8 % and lead 62.7 % at operating conditions of pH 3.5, current density 18.8 milliamp/cm^2, initial concentration of chromium 110 ppm and lead 20 ppm. The consumption of energy was 0.58 KWH/m^3 and proves that the process was economical and alignment with various researchers work as shown in table 4. The research suggest that electrocoagulation can be used as primary treatment for real tannery effluent followed by other organic pollutant degradation technology such as aeration pond. Electrocoagulation technology has been proven to be efficient and economical option because of various mechanism for treatment of leather tannery effluent have been carried during such as electrostatic attraction, chemical precipitation, oxidation and reduction.

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DECLARATION OF COMPETING INTEREST

None.

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