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RESEARCH ARTICLE

REMOVAL OF CADMIUM WITH THE SACRIFICIAL IRON ELECTRODES

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ABSTRACT

In this article, we demonstrate the effect of initial p^H (2-10), charge loading (1.83-18.25 Fm^{-3}), current density (0.4-3.2 Adm^{-2}), distance between two electrodes (1-5 cm), time of electrolysis (5-50 min), and metal ion concentration (2-25 mgl^{-1}) on the removal of cadmium (Cd) from industrial wastewater using electro-coagulation (EC) technique. In particular, we find that when the initial p^H of 8, charge loading of 18.25 Fm^{-3}, current density of 3.2 Adm^{-2}, and distance between electrodes is 3 cm; Cd percentage of the industrial wastewater is reduced to 99.9 % after 15 minutes of electro-coagulation process. In spite of this result, for 1 kg of Cd removal produces 2.8 kg of sludge; meanwhile, the 32 kg of sludge is generated from the iron sulphate precipitation method. These results suggest that the proposed EC technique has improved industrial wastewater quality by controlling various physico-chemical characteristics.

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INTRODUCTION

Removal of toxic metals from drinking water and industrial effluents has long been identified as an important field of research (Henry *et al*., 1963**;** Borgono *et al*., 1971). This theme of research has been attracting more and more attention to prevent the severe problems in both environmental water pollution and human health (Chowdhury *et al*., 1999; Mata *et al*., 2007; Huang *et al*., 2004). Among several purported toxic metals, cadmium (Cd), arsenic (As), chromium (Cr), and lead (Pb) are considered as the most environmental toxicants which can easily enter into the human body through drinking water and crop irrigation. Of those, Cd has become one of the highly toxic elements and diffused into drinking water. For examples, it has reported that the vegetables grown by the drinking water consist of Cd complexes in the presence of superphosphate (7.25 parts of Cd per million) accumulate severe problems in human kidney and liver (Skubal *et al*., 2002; Gupta *et al*., 2003). Since then, environmentalists have paid much attention to removing the Cd from drinking water to prevent acute and chronic disorders, renal damage, emphysema, hypertension, and testicular atrophy (WHO, 1984; Leyva-Ramos, 1997). In addition to that, the rapid growth of world population is rapidly developing, hence wastewater has been produced largely, and removing of toxicants from large volumes of water is challenging and most contests. Few methods were used for removing the toxicants is technically friendly, but are very expensive. Thus, it is necessary to develop an innovative, simplistic, generic and inexpensive method to purify wastewater from the toxicants. In the recent past, several attempts have made on the removal of toxic metals from wastewater using chemical precipitation, nano-filtration and ultra-filtration, reverse osmosis, adsorption (Mansour *et al*., 2011), solvent extraction, and ion exchange respectively (Holt *et al*., 2002; Boddu *et al*., 2003; ZHU Zhi-liang *et al*., 2007; Maryam Kazemipour *et al*., 2008). Despite the most recent developments in this field, electrocoagulation (EC) approach is still a preferred option because of its simple procedure, less sledge production, a clean technique, and low cost treatment method (Daniel *et al*., 2012). Recently, we purported that the electro-coagulation batch reactor method is an efficient approach in the removal of Arsenic from drinking water and

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industrial effluents, and the removal percentage has reached to 99 % (Daniel *et al*., 2012; Daniel *et al*., 2008). Moreover, the production of sludge is considerably less compared to all known standard industrial facilities. With the motivation of our earlier results, here we made a special effort to remove the Cd toxicants from industrial effluent using standard electro-coagulation method. We also investigate the removal percentage of Cd from industrial effluents by changing various novel physico-chemical parameters like initial p^H, charge loading, current density, and Electrolysis time in electro-coagulation process. Characterization of sludge produced in the EC process has been analyzed by Scanning Electron Microscope (SEM).

Experimental Section

Typical solution of 1000 mgl^{-1} was prepared by dissolving 1.63 g of Cadmium Chloride ($CdCl_2$) into 1000 ml double distilled water. The hydrochloric acid and sodium hydroxide subsequently added drop wise to adjust the p^H of the solution. Combinations of Al and Fe plates (100 mm×50 mm×0.5 mm with purity of 99.9 %) are used as electrodes. The conventional electro-coagulation experimental setup is shown in Fig.1 (a); this experimental setup is used for the present study. The typical synthetic solution is taken in 1000 ml beaker and the electrodes were carefully dipped into the solution, and electrodes were connected to radiometer potentiostat/ galvanometer (Model: RPS-302 D). During the EC process, iron complexes formed by the polyvalent Fe (II) cat ions which are electrolytically produced from the sacrificial anode and this phenomenon helped to enhance the Cd coagulation process. By means of Faraday's Law, these cat ions facilitate coagulation by neutralizing the negatively charged particles, and these charged particles drawn towards the anode by electrophoretic motion. The electrolysis gases produced at cathode could enhance the flocculation of the coagulants. The enhancement of flocculation could cause to carry the pollutants to the top of the solution. After the completion of the whole treatment, pollutants were collected from the reactor, and filtered to eliminate sludge. From Fig. 1(b), a clear distinction is identified on the electrode surface before and after the experiment commenced. The impurities on the electrode surfaces were removed by dipping in a freshly prepared 100 ml HCl solution for 5 minutes. Soon after, the electrodes washed thoroughly with water, the electrodes were dried and weighed at the end of each run. The amount of the wet sludge has been estimated, and the residual amount of sludge has been determined after drying at 105 ^oC for 24 h and ignited at 550 ^oC for 2 h, respectively. The TCLP have been carried out to understand leach-ability of Cd from the sediment. The residual concentration of metal ions is estimated by using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP - OES) (Model: Thermo Electron Corporation model IRIS Intrepid II XSP). Cd analyzed by AAS and the statistical data has given in Table 1.

RESULTS AND DISCUSSION

Influence of p^H

Fig. 2 presents the removal efficiency of Cd as a function of p^H of the solution with different combinations of electrodes. From the figure, it is obvious that the removal efficiency of Cd ions increases with the increase of the initial p^H in the range of 2 to 10. Notably, the removal efficiency of Cd increased from 84.2 to 94.6 % and 86.4 to 99.0 % with an increase of the initial pH of the solution, when we used Al-Al and Al-Fe electrodes respectively. Interestingly, when we use Fe-Fe electrodes, the removal efficiency of Cd has reached a maximum of 99.9 % at initial p^H =8.0. It is interesting to note that after p^H =6.0, the Cd removal percentage has slightly increased about 0.8 % with Fe electrodes. After the EC process, the removal percentage of Cd lies in between 84.2 and 96.8 with the final p^H value of 4.9 to 8.3 when we used Al electrodes. Despite of this, the removal percentage of Cd lies in between 89.8 to 99.9 with the final p^H values of 5.0 to 8.5 with Fe electrodes. It is very interesting to notice that the final pH values are less compared to initial pH values in EC Process. This is due to the formation of $Fe (OH)_4$ and $Al (OH)_4$ complexes; are in the aqueous stream as a gelatinous suspension, which can remove the pollutants from wastewater, either by complexation or by electrostatic attraction followed by coagulation and flotation during electro-chemical process. During the formation, all the aluminum cat ions produced at the anode and formed polymeric species Al^{3+}, precipitated as $Al(OH)_3$ leading to a more effective treatment in the removal of Cd. The slight increases in pH values are due to the formation of Hydrogen gas at the cathode and hydroxyl ions (OH^-) produced at the cathode electrode. These OH- ions compensate the acidic buffer and make the solution alkaline. This is due to the excess of hydroxyl ions generated at the cathode and the exchange of Cl^{-1} with OH^- in $A1 (OH)_3$ and $Fe (OH)_3$ in acidic conditions. The initial p^H is above 8, slight decrease in the final p^H could be due to the formation of $Al (OH)_4$ and $Fe (OH)_4$. Hence, the Cd removal efficiencies were better at p^H 6 for Fe and p^H 7 for Al electrodes.

Influence of Charge Loading

Fig. 3 shows the influence of charge loading on the removal percentage of Cd with different electrodes. It can be seen that the removal percentage increases from 96.8 to 99.9 with increasing of charge loading from 0.316 Fm^{-3} to 0.916 Fm^{-3} when we used Fe electrodes. Meanwhile, the removal percentage of Cd is increased from 94.5 to 99.4 with charge loading changes from 0.222 Fm^{-3} to 0.711 Fm^{-3} by Al electrodes can be seen in Fig 3. This is ascribed to the fact that, at high charge loading, the amount of iron oxide increased due to increasing of oxidation at anode, and resulting in a greater amount of precipitate for the removal of cadmium (Chen *et al*., 2002). This is due to the purity of electrodes causes for the formation of more amounts of metal-hydrous ferric complexes during electrocoagulation process. From the results it could be understood that Fe electrodes are identified as best electrodes in the removal of Cd from wastewater. Charge loading depends on Al^{3+} and/or Fe^{2+} ions produced in solution during the electro-chemical process. Hence, the amount of aluminum and iron adsorption increased significantly with an increase in adsorbent concentration, which indicates that the adsorption depends on the availability of binding sites for iron or aluminum hydroxides flocs. In the case of Al and Fe electrodes used in electro-coagulation, where the expected Cd removal is governed by the formation of metal-hydrous ferric or aluminum oxide complexes and the simultaneous flotation of $Al(OH)_3$ or $Fe(OH)_3$.

Table 1. Statistical data for trace element analysis by AAS

Element	Taken μg g^{-1}	Mean μg g^{-1}	SD μg g^{-1}	CV (%)	RE (%)
Cd	10.00	10.30	0.75	7.50	-3.00

Table 2. The Effect of electrolysis time on removal of cadmium

Contact time (min)	Residual concentration (in ppm)			
	0.4 A dm^{-2}	0.8 A dm^{-2}	1.6 A dm^{-2}	3.2 A dm^{-2}
5	0.58	0.48	0.39	0.24
10	0.42	0.37	0.26	0.16
15	0.28	0.17	0.07	0.01
20	0.18	0.09	0.03	0.006
30	0.10	0.03	0.007	0.001
40	0.07	0.009	0.003	0.001
50	0.007	0.003	0.001	0.001

Surface area 50 cm^2, electrodes Fe/Fe, pH=6.

Table 3. Operating conditions for removal of Cd from industrial effluents using EC

Environmental Parameter	Initial concentration	Effluents before primary treatment treated with EC		
		5 min.	20 min	30 min
pH	6.8±0.2	7.2±0.1	7.3±0.1	7.2±0.2
TDS, mg/l	1240±12.6	236±8.3	54±4.29	17±1.67
COD, mg/l	396±4.8	38±2.1	26±1.2	7±0.83
BOD, mg/l	31±1.4	11±0.94	7±0.81	4±0.58
Cd, mg/l	18±0.97	1.16±0.03	0.09±0.01	0.01±0.005
Environmental Parameter	Initial concentration	Effluents before primary treatment treated with EC		
		5 min.	20 min	30 min
pH	5.2±0.2	7.0±0.1	7.3±0.2	7.2±0.2
TDS, mg/l	5460±42	1200±7	970±6.1	885±3.8
COD, mg/l	2150±9	310±4	275±2.6	242±1.2
BOD, mg/l	106±6	51±2.8	35±1.1	19±0.9
Cd, mg/l	89±9	10.2±0.6	5.63±0.2	4.02±0.11
Environmental Parameter	Initial concentration	Effluents before primary treatment treated with EC		
		5 min	20 min	30 min
pH	6.9±0.2	7.3±0.1	7.2±0.2	7.5±0.2
TDS, mg/l	92±2.68	60±1.26	28±0.92	20±0.81
COD, mg/l	71±2.43	46±1.08	11±0.84	8±0.61
BOD, mg/l	27±1.23	13±0.93	5±0.09	ND
Cd, mg/l	6±0.91	0.9±0.01	0.01±0.001	0.006±0.001

Table 4. The sludge formed after Electro-coagulation and Operating cost

Electrodes	pH	Electrolysis Time (min)	% Removal of Cadmium	Sludge formed (kgm^{-3})	Operating cost per \$ m^{-3}
Fe-Fe	2	30	98.4	0.034	0.0222
	4	25	98.5	0.041	0.0220
	6	20	99.6	0.048	0.0196
	8	20	99.9	0.056	0.0184
	10	20	99.9	0.054	0.0168
Al-Fe	2	30	95.2	0.025	0.0198
	4	25	96.8	0.028	0.0218
	6	20	98.2	0.032	0.0229
	8	20	98.6	0.036	0.0237
	10	20	98.4	0.035	0.0179
Al-Al	2	40	95.4	0.019	0.0226
	4	30	96.2	0.021	0.0232
	6	30	97.8	0.027	0.0246
	8	25	98.4	0.034	0.0252
	10	25	98.4	0.032	0.0198

It is obvious that these parameters are kept at low level to achieve a low cost treatment. The bubble density and upward flux increases at high charge loading thus faster removal of the coagulant by flotation is possible. Cd reduction is due to the collision between the coagulant and targeted pollutants.

Influence of current density

We have been examining the influence of the current density variation between 0.4 Adm^{-2} - 3.2 Adm^{-2} on the removal of cadmium using Al and Fe combinations of electrodes which is shown in Fig. 4.

Figure 1(a). Laboratory bench-scale Electro coagulation set-up

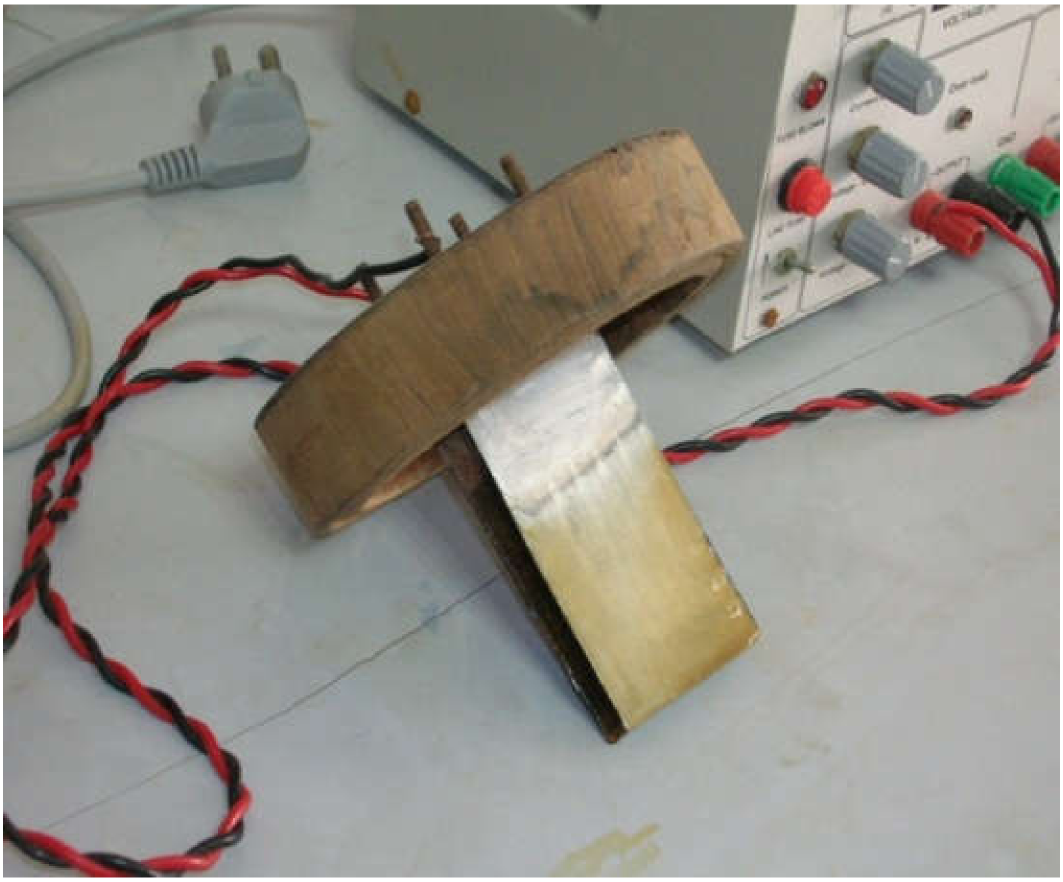

Figure 1(b). Electrodes after Electocoagulation

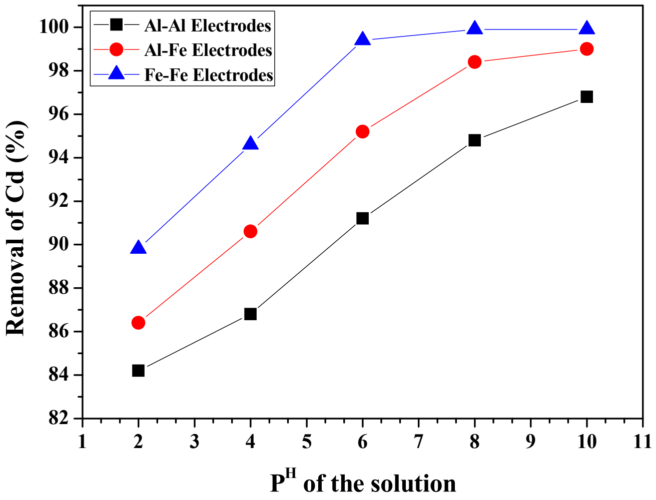

Figure 2. Removal percentage of cadmium as function of initial pH

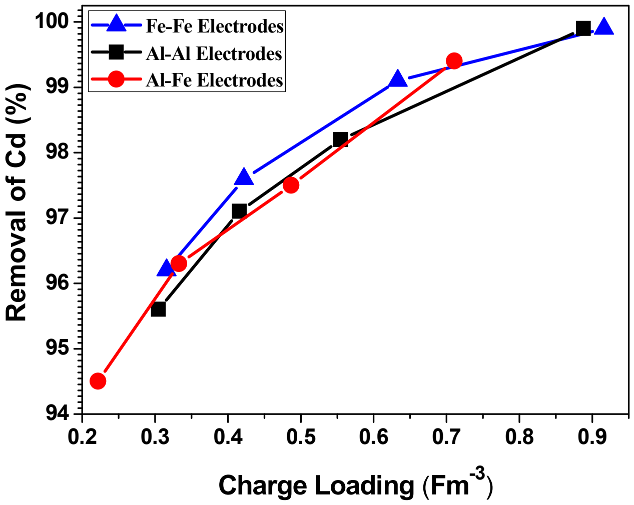

Figure 3. Removal percentage of cadmium as function of various charge loadings

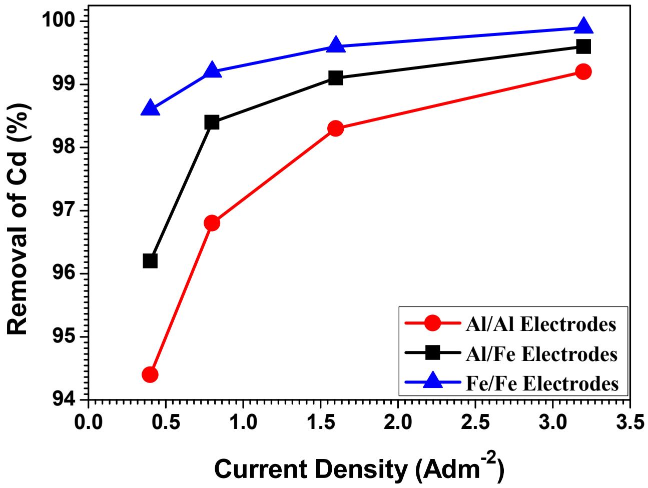

Figure 4. Removal percentage of cadmium at various current densities

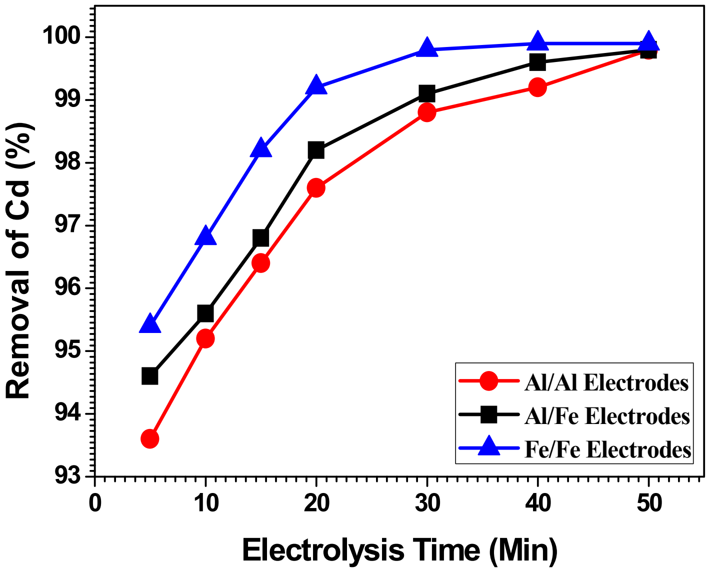

Figure 5. Removal percentage of cadmium at various Electrolysis time

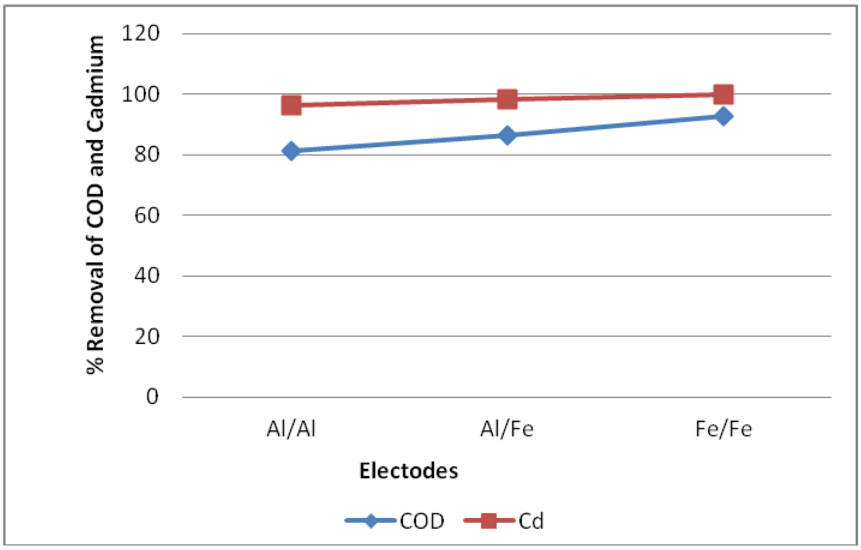

Figure 6. Removal of Cadmium from Pharmaceutical Effluents by Electrocoagulation

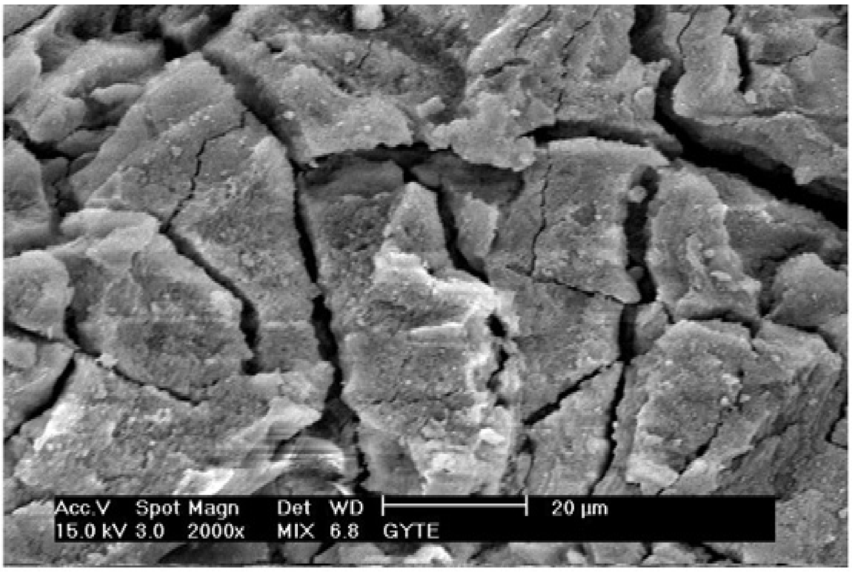

Figure 7(a) SEM image of Sludge at 20 µm size

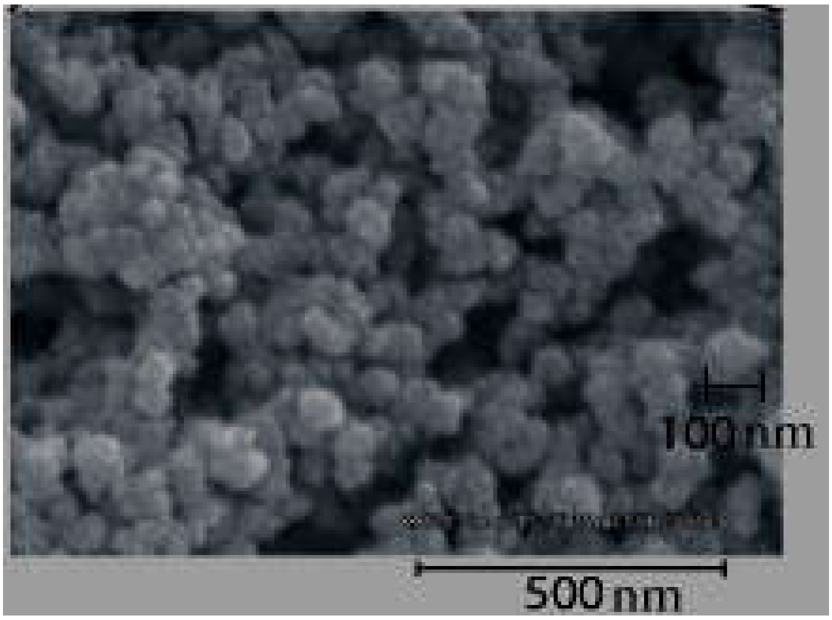

Figure 7(b) SEM images of Sludge at 100 and 500 nm size

From the measurements, the removal percentage of Cd increases from 94.4 to 99.2 and from 98.6 to 99.9 with Al and Fe electrodes respectively. The highest removal percentage of Cd has reached of 99.9 at 3.2 Adm^{-2} with Fe electrodes. Interestingly, it was noticed that the higher the current density led more turbidity in the solution. This can be explained by considering the fact that an increase in current density lead to increase in the rate of anode dissolution and hydrogen formation consequently favours flocculation in EC process. Hence affects the growth of flocs in the EC reactor with different electrode configurations (Kobya *et al*., 2010). It is interesting to know that the bubble sizes reduced with increase of current density, this is beneficial in the separation process. This startling behavior could be due to increase of coagulant; bubbles generation rate and distribution which result more efficient and faster removal which is possible with increased current density.

Influence of Electrolysis time

The removal percentage of Cd at a various electrolysis time using different electrodes has been investigated. The residual concentration of Cd is 0.58 ppm to 0.007 ppm at 0.4 Adm^{-2} Current density with electrolysis time of 5 - 50 min respectively. The residual concentration of Cd is 0.24 ppm to 0.001 ppm at 3.2 Adm^{-2} Current density. The highest removal of Cd reaches 99.9% has been observed at 3.2 A dm^{-2} with Fe electrodes with a 15 min electrolysis time. The current density and electrolysis time are directly proportional to the removal percentage of Cd shows in Fig. 5. The residual concentration of Cd decreases with increasing of Electrolysis time from 5 – 50 min at various current densities mentioned in Table 2.

EC for treatment of pharmaceutical wastewaters

We have also measured the removal percentage of Cd from pharmaceutical wastewater using different combination of electrodes. Figure 6 shows the results and it can be found that the removal percentage of Cd is 96.4% with Al electrodes and 99.8 % with Fe electrodes. It has found that the samples having initial concentrations of 6mgl^{-1}, 18mgl^{-1} and 89mgl^{-1} after the treatment of EC reached to 0.9mgl^{-1}, 1.16mgl^{-1} and 10.2mgl^{-1} within 5 min respectively. From the Table 3, it is found that the residual concentration of Cd decreases slowly and finally reaches to 0.01mgl^{-1} within the electrolysis time of 20 minutes, and the sample having an initial concentration of 6mgl^{-1}. The percentage of Cd removal has reached a maximum value of 99.9. The rate of removal percentage of Cd from the industrial effluents seems to be relatively slow as compared with the removal of Cd from synthetic wastewater. This could be caused by the presence of organic compounds in the industrial effluents, and the organic compounds might adsorb by Fe (OH)$_3$ and/or Al (OH)$_3$ coagulants which lead to as a substantial reduction of metal ions.

Characteristics of EC sludge

In the EC process sludge formation is primarily due to formation of denser fine metallic particulates, further separate from liquid to settle at the bottom of the reaction vessel due to gravity and magnetic separation. The sludge produced from the effluents is listed in the Table 4. The sludge was formed after the EC from 0.034 Kgm^{-3} to 0.054 Kgm^{-3} with Fe electrodes and from 0.019 Kgm^{-3} to 0.034 Kgm^{-3} with Al electrodes. The removal percentage of Cd is from 98.4 to 99.9 with Fe electrodes and from 95.4 to 98.4 with Al electrodes respectively. The amount of sludge formation increased with an increasing removal efficiency of Cd from the water. As the current density increased the anodic dissolution was favoured so that the metallic sludge residues increased and Cd ions were effectively removed from the water by co-precipitation or adsorption on metallic sludge. Values of sludge for each electrode were compared with respect to molar electrode consumptions based on molar water chemically and physically. Settable solids and matter destabilized by coagulation and the concentration of flocculent is also proportional to current density and electrolysis time (15, 19, 20, 21, and 17). The sludge was analyzed by SEM has been observed in Fig 7 (a) and 7 (b). The SEM image in Fig 7 (a) and 7 (b) indicates that the presence of mostly amorphous structure which has an aggregate size of 20µm. The sludge at 2000 x magnification appeared to be a relatively uniform cake (showing cracks due to drying). Fig 7 (b) shows particles which have a size of 100nm to 500nm.

Operating cost of cadmium removal

One of the most important parameters that affect the application of any method of water and wastewater treatment greatly is the operating cost. The operating cost includes material (mainly electrodes) cost, utility (mainly electrical energy) cost, as well as labor, maintenance and other fixed costs. The latter cost items are largely independent of the type of the electrode material. Thus, energy electrode material and chemicals costs for the treated water were taken into account as the major cost items in the calculation of the operating cost as of

Operating cost = a C_{energy} + b $C_{\text{electrodes}}$ + c $C_{\text{chemicals}}$

When C_{energy} (consumption in kWh energy per m^3), $C_{\text{electrode}}$ (consumption in kg chemicals per m^3) of potable water treated. Unit prices, a, b and c given from the American market, September 2011, are as follows: a is electrical energy prices as 0.04 /kWh, b is electrode material price as 0.89 /kg for aluminum and 0.46 /kg for iron, respectively, c is the prices of NaOH and H_2SO_4 as 0.23 /kg and 0.09 /kg, respectively. The operating cost for different electrode connection modes were calculated for the removal of Cd from potable water at 10µg/L or lower. The results from Table 4 were, in order of Al/Al, Al/Fe and Fe/Fe electrodes. The lowest operating costs for Al electrodes is from 0.0226 $\$m^{-3}$ to 0.0198 $\$m^{-3}$ with respect to the removal percentage of Cd is 95.4 to 98.8 for potable water at 10 mgl^{-1} or lower concentrations. When using Fe electrodes operating costs are 0.0222 $\$m^{-3}$ to 0.0168 $\$m^{-3}$ with respect the removal percentage of Cd is 98.4 to 99.9.

Conclusion

In summary, we conclude that this study showed the applicability of electrocoagulation to treat water contaminated by Cd. More than 99.9% of the pollutants were eliminated by using iron electrodes. The Cd removal efficiencies were better at p^H 6 for Fe and p^H 7 for Al electrodes. The optimum charge loading of 0.633 Fm^{-3} is required to remove Cd with 15 min for Fe electrodes. Al electrodes remove Cd at optimum charge loading of 0.711 Fm^{-3} with 20 min electrolysis time is necessary to achieve the best efficiency. It is inferred that the quickest removal rate is prevailed at highest charge loading of 0.916 Fm^{-3} with Fe electrodes. Increasing the current density from 0.4 Adm^{-2} to 3.2 Adm^{-2} resulted in a high binding capacity for Iron (III) hydroxide compared to that of aluminum hydroxide. The optimal current density retained in the case of iron was 1.6 Adm^{-2}. Charge loading examination also showed a correlation between the increase of this parameter current density and the removal efficiency. This study confirmed that iron electrodes are found to be ideal; the treatment was greener, faster and better than aluminum as well as no secondary pollutants release with this technique. The sludge formed during the electro-coagulation is tested for TCLP and found that Cd is not leaching from the sludge. The surface of the sludge was characterized by SEM and the image was mainly composed of irregular and porous particles having amorphous structure.

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