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electrolysis system of
 $\text{Fe(s)}/\text{NaCl(aq)}, \text{Pb}(\text{NO}_3)_2$
 $(\text{aq})//\text{H}_2\text{O(aq)}/\text{C(s)}$

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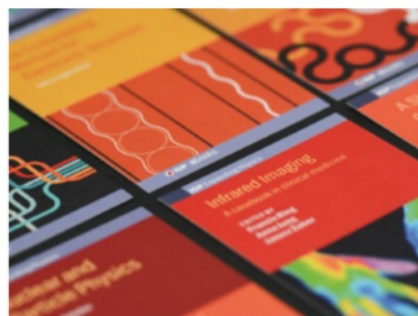
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Removal of Pb^{2+} metal ion using electrolysis system of $Fe(s)/NaCl(aq)$, $Pb(NO_3)_2(aq)/H_2O(aq)/C(s)$

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Abstract. Lead²⁺ is very dangerous heavy metal for the environment due to its toxicity and it requires a serious handling. A more efficient and effective method for handling is electrolysis using electrocoagulation. This method provides an electric current to the system, a redox reaction occurs followed by a coagulation process, which then absorbs Pb^{2+} metal ions to form flocculants. The study covered electrocoagulation of artificial waste containing Pb^{2+} , characterization of functional groups and compounds contained in flocculants. The two compartment reactor was used. It consisted of an anode (iron) and cathode (graphite) parts separated by a salt bridge. The anode was filled with 0.1M NaCl electrolyte solution, artificial waste of $Pb(NO_3)_2$. The cathode contained H_2O . Potential was set in the range of 1-6 V. Then the filtrate at the anode was analyzed by AAS and the precipitate was characterized by FTIR and SEM EDX. The results showed Pb metal waste was removed 43.2% by electrocoagulation method at a maximum voltage of 6 V. FTIR result shows Fe-O in the wave numbers of 1117 and 111.99 cm^{-1} and Pb-O in the wave numbers of 482.22 and 474.82 cm^{-1} . SEM analysis shows that the morphology has slightly changes. Elements contained by EDX analysis of the floc are iron, lead, oxygen, silica and sodium.

1. Introduction

Lead (II) is an inorganic compound, is toxic [1], many are ⁵ water [2] so that their presence are harmful to the environment. Lead metal poisoning can cause damage to the kidneys, nervous system, reproductive system, cardiovascular system, liver and brain [3]. Therefore it is necessary to do water treatment to reduce lead heavy metal level.

Effective and efficient design systems have been widely studied including ion exchange, precipitation, adsorption and electrocoagulation [4]. The electrocoagulation approach is chosen because it is a combination of electrochemical process and flocculation process. The coagulation process is proposed as an alternative handling of this metal waste that has been carried out by [5-6].

Factors that influence the success of this electrocoagulation technique are variables of electrodes, voltage, electrolytes and adequate energy applications. Meanwhile, considering the coagulation system that is the deposition of colloidal particles due to the electric current given ⁸ successful in forming a coagulant that is capable of absorbing nearby heavy metal ions. Coagulant $Al(OH)_3$ and $Fe(OH)_3$ are very good coagulation agents [7]. The electrodes used are graphite, aluminum and iron as the anode, while at the cathode graphite is used. The supporting electrolyte used is NaCl by Mansour et al [1].

This research focuses on the electrocoagulation system by prioritizing environmentally friendly, fast and efficient processes. The sample in this study was chosen in the form of an artificial lead²⁺ sample and examined the efficiency of the process and the reliability of the approach from the

standpoint of environmental indicators and selecting the right electrodes and the functional groups, compounds contained in the precipitated electrolysis.

2. Experimental section

2.1. Materials

Materials used include: $\text{Pb}(\text{NO}_3)_2$ (p.a, Merck), NaCl (pa, Merck), carbon (Graphite), Iron Plate, distilled water, filter paper.

2.2. Instrumentation

The tools used include: a set of pirex glassware, electrolysis reactors, analytical balance (S / NP 1 835442), filter paper (Brand MN 640d, 10mm), Power supply (DC), Digital Multimeter (Sanwa CD800a), pH meter, Spectrophotometer of Atomic Absorption (PinAAcle 900F Model), Perkin-Elmer Spectrum Version (FT-IR) and SEM (Phenom Pro X Desktop SEM with EDX).

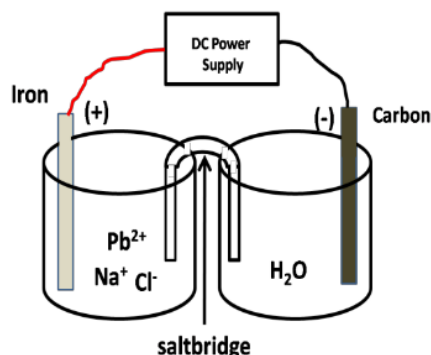


Figure 1. Schematic diagram of electrocoagulation system.

2.3. Procedures

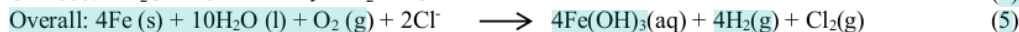
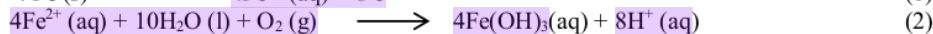
Electrolysis consists of two compartments, the anode and the cathode connected by a salt bridge. The concentration of waste is 10 ppm, NaCl 0.1M and the time is 1 hour. Voltage varies from 1-6 V. The schematic diagram of electrocoagulation system is shown in Figure 1. The electrolysis solution after filtration was analyzed with AAS and the sediment was analyzed by FTIR and EDX.

3. Results and discussion

In this study the removal process of Pb^{2+} was carried out by electrocoagulation using iron metal as an anode and carbon as a cathode. The use of iron aims to produce $\text{Fe}(\text{OH})_3$ coagulants. The process of forming $\text{Fe}(\text{OH})_3$ is shown in equations (1) to (5). $\text{Fe}(\text{OH})_3$ can take Pb (II) in solution through adsorption followed by coagulation [8]. The colloidal $\text{Fe}(\text{OH})_3$ particle has a positively charged colloidal core and a negatively charged surface because it is surrounded by many OH^- ions. A colloidal particle has an electric charge on the colloidal surface. With this charge, ions with opposite charge will gather to the colloidal surface, and the ion atmosphere is formed [9]. The positively charged Pb metal ion will be adsorbed very strongly on the surface stern layer of $\text{Fe}(\text{OH})_3$ due to electrostatic attraction, this ionic layer does not move. Between the stern layers with its outer atmosphere is called the electrical double layer or the diffusion layer. When high ionic strength diffuses to the surface an electrostatic repulsion will occur, to obstruct the approach between two colloidal particles. As a result, there is an immediate coagulation-flocculation, which is the clumping of colloidal particles to form macro fase which are easily deposited [9]. Pb metal ions that are absorbed on the colloidal surface will also settle together. The process of coagulation of colloidal particles is also caused by the neutralization of the colloidal particle charge due to the large number of Pb metal ions that are

absorbed on its surface, so that the stability of the colloid becomes disrupted and allows clumping and precipitation.

The electrolysis process produces reddish orange deposits of $\text{Fe}(\text{OH})_3$ at the anode and H_2 gas bubbles at the carbon cathode. The reactions that occur are:



3.1. Voltage variations

The higher the voltage the more Pb^{2+} was electrocoagulated. This is in accordance with the ohm law $V = I.R$ where V = voltage (Volt), I = current (ampere and R = resistance) so that the higher the voltage the higher the current produced. The more the current produced the more $\text{Fe}(\text{OH})_3$ obtained that functions as coagulation for Pb^{2+} ions.

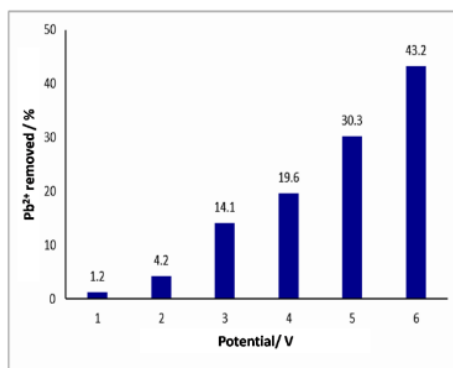


Figure 2. Pb^{2+} recovered (%) Vs potential (V)

3.2. Floc characterization with FTIR instrument

To find out the clusters contained in floc, analysis was carried out before and after being given a solution of $\text{Pb}(\text{NO}_3)_2$. FTIR spectra results can be seen in Figure 3. This data show that the compound $\text{Fe}(\text{OH})_3$ has been successfully synthesized in the anode compartment. As the bonding vibrational peaks in $\text{Fe}(\text{OH})_3$ were observed in the spectra as shown in table 1.

Table 1 and figure 3 can be seen that before and after insertion of Pb^{2+} ion there are almost the same wave numbers of OH, namely 3445.97 and 3436 cm^{-1} , N-O groups in wave numbers of 1636.58 and 1631.24 cm^{-1} . The nitro group arises at wave numbers $1300 - 1700 \text{ cm}^{-1}$, while Fe-O groups are at 1117 and 1111.99 cm^{-1} . Meanwhile Pb-O appears in wave numbers of 482.22 cm^{-1} and 474.82 cm^{-1} .

Table 1. Data on the peaks of FTIR coagulant spectra before and after the presence of Pb^{2+} waste in the electrocoagulation process.

Vibration	before (cm^{-1})	after (cm^{-1})
OH stretching	3445.97	3436
Fe-O stretching	1117.51	1111.99
	-	1039
	616.82	-

N-O stretching	1636.58	1631.24
Pb-O	-	474.82, 482.22

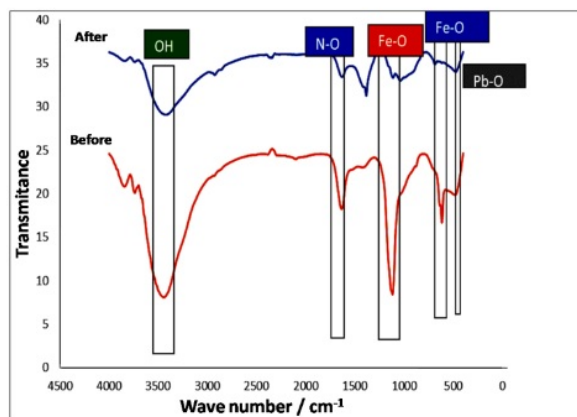
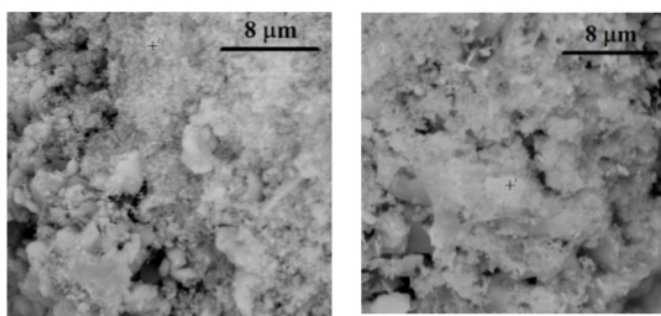


Figure 3. FT-IR Spectra of the floc before and after Pb^{2+} insertion in the electrode Fe/C.

3.3. Floc characterization with SEM EDX instrument

SEM results show fine depositions of $Fe(OH)_3$ that the shape of the sediment does not produce differences in the surface morphology of the floc, but the presence of the floc constituent can be confirmed on EDX data.

Characterization of electrocoagulation floc before and after coagulating Pb metal using EDX instrument to find out the composition of elements contained in each floc produced at the anode. The results of the characterization can be seen in Figure 4. The floc constituent consists of Fe, O, and Pb elements. Pb arises before the presence of artificial waste; it can be from the iron used. After adding artificial Pb waste its composition increased until 71.6% of the coagulation process. The presence of Si comes from silica present in the glass used as a compartment. While Na and Cl atoms derived from NaCl compounds. More complete composition can be seen in table 2.



Before $[Fe(OH)_3]$ After $[Fe(OH)_3]-Pb$
Figure 4. SEM images of floc before and after recovered of Pb^{2+} .

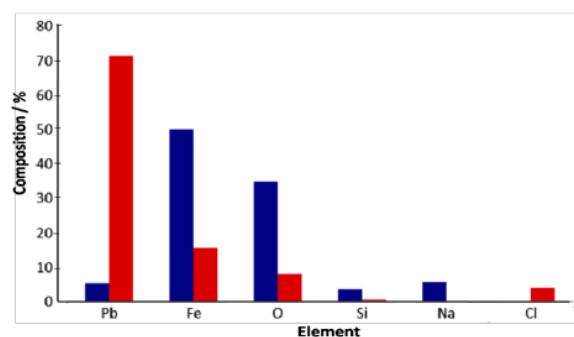


Figure 5. Elemental composition of floc before (blue color) and after (red color) Pb^{2+} introduction measured by EDX.

Table 2. EDX of floc before and after coagulated Fe.

Condition	Pb	Fe	O	Si	Na	Cl
Before	5.5	50.2	34.9	3.8	5.6	0
After	71.6	15.6	8.1	0.7	0	4

4. Conclusion

Pb metal waste was successfully conducted by electrocoagulation method with a maximum voltage of 6 V and a yield of 43.2%. The FITR results show that Fe-O is in wave numbers of 1117 and 111.99 cm^{-1} and Pb-O in wave numbers 482.22 and 474.82 cm^{-1} . SEM shows that morphology does not show significant changes. The elements contained in floc are elements of iron, lead, oxygen, silica and sodium.

Acknowledgements

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