Optimizing the Conditions for Leaching Lead from Solid Waste Produced by Pyrometallurgical Process of Recycling Automobile Used Batteries

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Abstract. Over 90 % of automobile used lead-acid batteries are recycled worldwide. The typical new car battery contains 60-80 % recycled lead. The current method of disposing lead-contaminated solid waste, generated by lead-acid battery recycling industries, is to bury it in a remote waste management site. This practice of disposing such hazardous solid waste (~22 % in lead) is not a positive approach to solve the environmental problem. In this work we determined the optimal conditions for leaching lead from solid waste using EDTA. Our experimental results indicate that at pH 4.8, a leaching temperature of 55°C and a 30 min leaching time, 96.8 % of lead present was leached from the solid having particle size <325 mesh (0.051 mm).
Keywords: Automobile battery recycling; lead leaching time; pH; temperature; particle size

Resumen. En todo el mundo más del 90 % de las baterías de automóvil plomo-ácido son recicladas. Una batería nueva para automóvil contiene de 60 a 80 % de plomo reciclado. El método común de confinamiento de los desechos sólidos contaminados con plomo, generados por la industria recicladora de baterías de plomo-ácido, es enterrarlos en un sitio lejano de manejo de desechos. Esta práctica de confinar tales desechos sólidos (~22 % de plomo) no es adecuada para resolver el problema ambiental. En este trabajo encontramos las condiciones óptimas de lixiviación de plomo de desechos sólidos usando EDTA. Nuestros resultados experimentales indican que se lixivió el 96.8 % del plomo presente en el sólido que tiene un tamaño de partícula < 325 mesh (0.051 mm) a pH 4.8, temperatura de 55 °C y 30 min de tiempo de lixiviación.
Palabras clave: Reciclamiento de baterías de automóvil, tiempo de lixiviación del plomo; pH; temperatura; tamaño de partícula.

Introduction

Recyclable lead-acid batteries are the principal source of power for automobiles, trucks, motorcycles, boats, forklifts, golf carts, lawn and garden tractors and wheel chairs. These batteries also support a variety of back-up systems in computers and rechargeable battery- operated products, such as handheld power tools. Some of the little known yet significant applications of lead batteries include maintaining power to operating rooms when a storm knocks out electricity, power electric vehicles and providing power for important military missions. Needless to say the lead-acid battery makes our lives easier; its usefulness is taken for granted because it works "behind the scenes".

Salamon [1] reported that a typical automobile lead-acid battery contains 18 – 20 pounds of lead. The average life of a domestic car battery is about five years. Used batteries end up at battery recycling sites. The current major recycling method for lead-acid batteries employs a pyrometallurgical process; the used battery is first crushed to drain off the sulfuric acid, the lead plates of the battery are heated in a tilting rotary high temperature furnace, liquid lead drains into a mold to produce ingots of lead after they are cooled. Besides producing metallic lead, this thermal process also oxidizes some lead metal and converts other lead compounds to lead oxides; the lead oxides mix with the rest of the solid waste left behind in the furnace. The lead content in this solid waste is about 22 wt-%.

Up to now, there is no existing economical technology to recover this lead and to reduce the lead content of the treated

solid to a Maximum Contaminant Allowed Level (MCAL) < 5 ppm as required by Mexican Environmental Protective Agency stated in Diario Oficial de Federacion [2]. In order to keep the battery recycling business going, the owner of the battery-recycling site usually transports the lead contaminated solid waste to a remote solid waste management site to have it buried. This method of disposing a highly lead-contaminated solid is not a positive approach to solve the environmental problem of this waste. This practice only postpones the problem; eventually lead will be leached out to poison the environment. Therefore we ought to develop an efficient and economical process to recover the lead from the lead-contaminated solid waste produced by lead-acid battery-recycling industries.

Using Ethylenediaminetetraacetic acid (EDTA) to extract heavy metals such as lead from lead-contaminated solid has proven to be an effective method due to the strong complexing ability of EDTA for heavy metal ions. Heil [3] and Monika [4] reported their works on remediation of lead contaminated soil by EDTA. Hong [5] investigated the interference of other heavy metals on the chemical extractability of lead by EDTA. The purpose of this investigation was to optimize the leaching parameters that include the temperature, the pH of the leaching solution and the particle size of the lead-contaminated solid. The actual solid waste, produced by the pyrometallurgical lead-acid battery-recycling process taking place in Tijuana, Baja California, Mexico, was used in this work. We aim at developing moderate leaching conditions that are technically doable and economically feasible for the battery-recycling industries.

Experimental

Materials, Equipments and Methods

Materials

The automobile battery recycling company, Metales y Derivados de México of Tijuana, Mexico provided the lead-contaminated solid waste. The chemical analysis of a typical solid waste from this company is given in Table 1. Reference solutions of Pb^{2+} for atomic absorption were prepared from a 1000-ppm standard aqueous solution, purchased from Polarchem. Stock solutions of Pb^{2+} were prepared from ACS Analytical Grade chemicals supplied by Aldrich Chemicals, U.S.A.; the concentrations of Pb^{2+} in each of the stock solutions were determined by atomic absorption. EDTA of 99.4% purity was purchased from Productos Químicos Monterrey, Mexico.

Equipments

The concentrations of Pb^{2+} in the test samples were determined with a VARIAN SpectrAA-5 Atomic Spectrophotometer. A & D weighing precision analytical balance Model HR-120 was used to weigh all solid reagents and samples in this work. The apparatus (Figure 1) used for leaching lead from the solid waste at constant temperature water bath was fabricated in our laboratory. Sieves of 70, 200 and 325 mesh (0.21, 0.074 and 0.051 mm) were obtained from McMaster-Carr Supply Company, Los Angeles, California, U.S.A. BAS 100B Electrochemical Analyzer was purchased from Bioanalytical Systems Inc., West Lafayette, Indiana, U.S.A.
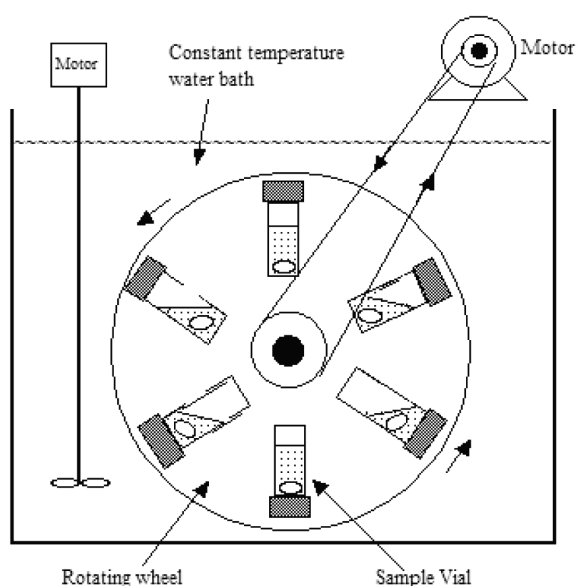

Fig. 1. Experimental set-up for leaching lead from solid waste generated by automobile battery recycling company located at Tijuana, Baja California, México.

Methods

1. Separation of lead-contaminated solid waste into its size range

The lead-contaminant solid waste from lead-acid battery recycling operation via pyrometallugical process was provided by Metales y Derivados de México, Tijuana, Baja California, México. The solid was first thoroughly grinded with a porcelain mortar and pestle set for 10 minutes for each batch of solid sample, and then the grinded solid was placed at the top of a stack of sieving trays having sizes of 70, 200 and 325 mesh; the sieving trays were shaken until no more solid passing through to the next sieving tray. The solid does not go through the 70 mesh sieve is labeled as Particle Size > 70 mesh (0.21 mm), the solid going through 70 mesh sieve and retained by the 200 mesh sieve is named as Particle Size < 70 and > 200 mesh (0.074 mm); finally the solid passing through the 325 mesh sieve is classified as the Particle Size < 325 mesh (0.051 mm). There were no visually observable amount of metallic lead present in each particle size solid; granulated metallic lead may tend to be > 70 mesh in size.

2. Determination of total lead content in each solid size range

An accurately weighed amount of a solid sample was placed in an Erlenmeyer flask, 32 mL of concentrated nitric acid was poured slowly into the flask, the flask was plugged with a rubber stopper having a 12-inch long glass tubing going through it. After the mixture was heated at 50°C and stirred with a magnetic stirring bar for 24 hours inside the flame hood, a small amount of the final leaching solution was collected through a 0.2 mm micron filter, the lead content in this filtered solution was determined by Atomic Absorption against a standard lead solution. The total lead content in each size range of solid is tabulated in Table 2.

Table 1. Results of analysis of a typical waste solid produced by an automobile used battery recycling plant operated in Tijuana, Baja California, México.

Component	Weight % from Dried Sample
Lead	22.3
Carbon	26.8
Silicone	9.0
Sulfur	8.1
Iron	6.4
Copper	1.7
Aluminum	1.5
Manganese	1.4
Tin	1.1
Water soluble compounds	21.7 (mainly due to sodium salt)

3. Sample loading for the lead leaching process

Sample loading for the lead leaching experiment was carried out by placing an accurately weighed 1.0000 grams of lead-contaminated solid into a 15 mL-green-cap-vial containing a ¼-inch Teflon-coated magnetic bar and 7 g of distilled water, the vial was closed tight, the mixture was shaken well for 3 min, then 2 g of EDTA and a predetermined amount of solid NaOH were added to the sample to make the resultant mixture to be at a desired pH level. The sample vial was closed tightly and was attached to the rotating wheel immersed in a constant temperature water bath (Figure 1). Up to six sample vials could be attached to the rotating wheel for each experiment.

4. Lead leaching experiment and collection of leaching solution for lead analysis

The lead leaching experiment was initiated at the time when the EDTA and NaOH were added to the vial containing the sample mixture. When the rotating wheel was turned on at 10 rpm, the tumbling of the Teflon-coated magnetic bar agitated the solid-solution mixture inside the sample vial. A small amount of liquid sample (0.2 g) of leaching solution was collected with a syringe equipped with a 0.2 mm micron filter at time intervals up to 60 min of total leaching time. The water content in each of the collected sample liquids was determined by evaporating the solution slowly on a thin glass plate to dryness in an oven at 80°C. Knowing the lead content in this sample solution through Atomic Absorption analysis and the quantity of water in the collected sample solution, the total lead contained in this leaching solution was calculated. This enables us to determine the % of lead leached from the lead-contaminated solid sample at each time interval of the leaching process. The experimental results from the lead leaching process at various leaching times, temperature, pH and particle size are shown in Figures 2 and 3 and Tables 3 and 4.

5. Recovering lead from the EDTA leaching solution via cathodic reduction

In order to establish the condition for recovering lead from the EDTA leaching solution via electrodeposition, cyclic voltammograms of synthetic solutions containing lead with/without addition of EDTA and the actual sample solution from the lead leaching process were recorded with the BAS 100B Electrochemical Analyzer. The experimental conditions and the cyclic voltammetric traces of these three solutions are shown in Figure 5.
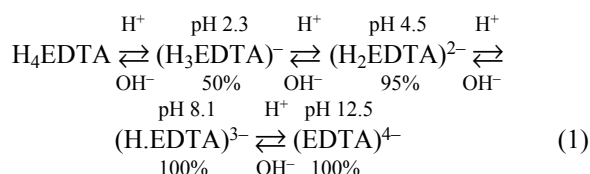
Results and Discussion

We chose EDTA as the leaching agent because of its high thermal and chemical stabilities; it is stable up to 240 °C and immune to bacteria or mold breakdown and to high acidity or

alkalinity. Also H_4EDTA is practically insoluble in water at its isoelectric point, therefore it can be easily recovered by adjusting the pH of the leaching solution after the lead is recovered by electrodeposition at cathode surface. Our experimental results indicated that up to 85% of EDTA was recovered from the leaching solution at pH~2.

In general, increased temperatures result in lower stability constant. This effect is usually quite negligible, on the order of one log unit for each 55 °C of temperature change. However, increase in the temperature decreases the viscosity of the leaching solution. Also the thermal agitation becomes more vigorous at higher temperature. These two factors increase the diffusion rates of the leaching agent into the porous solid and the chelates moving out from the core of the solid into the bulk of the leaching solution.

Beside temperature, pH is considered to be an important factor that affects the leaching rate. EDTA is a polycarboxylic acid; the pH determines the particular ionic species that is present in the system. Pecsok [6] had shown in Equation (1) that the species of the acid present in solution is greatly pH-dependent.

$$
\begin{array}{c}
\overset{H^+ \qquad pH\ 2.3 \qquad H^+ \qquad pH\ 4.5 \qquad H^+}{H_4EDTA \underset{OH^- \quad 50\%}{\rightleftarrows} (H_3EDTA)^- \underset{OH^-}{\rightleftarrows} (H_2EDTA)^{2-} \underset{95\% \qquad OH^-}{\rightleftarrows}} \\[2mm]
\overset{pH\ 8.1 \qquad H^+ \quad pH\ 12.5}{(H.EDTA)^{3-} \underset{100\% \qquad OH^- \quad 100\%}{\rightleftarrows} (EDTA)^{4-}} \qquad (1)
\end{array}
$$

The primary chelating species are the tetra-negative EDTA ions. Increasing the acid concentration (lowering the pH) therefore favors formation of the less highly ionized, weaker chelating species. The competition seen in Equation (2) is between the metal ion (M^{2+}) and the hydrogen ion for the chelating anion.

$$MEDTA^{2-} + 4\ H^+ \rightleftarrows H_4EDTA + M^{2+} \qquad (2)$$

Increasing the pH stabilizes the completely ionized chelating species ($EDTA^{4-}$), but this advantage is balanced by the tendency of the metal ion to interact with the hydroxide ion. In Equation (3), the competition is between the chelating agent and the hydroxide ions for the metal.

$$MEDTA^{2-} + 2\ OH^- \rightleftarrows M(OH)_2 + EDTA^{4-} \qquad (3)$$
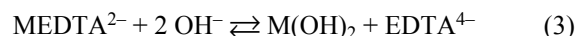
Heavy metal hydroxides are quite insoluble materials. When the hydroxide concentration is great enough, metal hydroxide precipitates from the leaching solution. Therefore the favorable pH range for leaching the lead from the lead-contaminated solid has to be experimentally determined.

The sole objective in this work was to determine the optimum conditions for leaching lead from the actual industrial waste solid. The detailed mechanism and the kinetic aspects of the leaching process of metal or metal oxide ores that have been investigated extensively by Wadsworth [7], Dixon [8] and Samani [9] are beyond the scope of this investigation.

The experimental results of this work are grouped into three major areas: 1) dependence of % lead leached from solid

Table 2. Total lead weight % in solid waste of three ranges of particle size.

Particle Size Range (Mesh)	Pb wt-%
<325 (0.051 mm)	$22.40^+ 0.17$
<70 (0.21 mm) and >200 (0.074 mm)	21.30 ± 0.30
>70 (0.21 mm)	20.13 ± 0.10

Table 3. Dependence of % Pb leached from solid of particle size < 325 mesh on temperature and pH of the leaching solution for a 30 minutes leaching time.

pH	4.8	6.0	8.0	10.0
T (°C)		% Pb leached		
25	82.3	69.9	72.3	69.7
40	89.5	80.7	73.0	70.1
55	96.8	90.9	75.3	74.4
75	97.5	92.7	86.8	78.1
90	96.9	~100.0	87.3	84.3

of particle size < 325 mesh (0.051 mm) on time, temperature and pH of the leaching solution, and 2) dependence of % lead leached on particle size, leaching time, temperature and pH of the leaching solution. 3) Recovering lead and EDTA from the leaching solutions. The results of each of these three areas of experimental work are presented separately in the following.

1. Dependence of the % lead leached from solid of particle size < 325 mesh (0.051 mm) on time, temperature and pH of the leaching solution

For a given set of leaching conditions, the % lead leached from the solid sample was calculated from the amount of lead present in the leaching solution at the time the sample solution was collected for analysis and from the total lead content in the original solid sample (22.4 wt %, Table 2). The plot of the % lead leached versus the time at various temperatures and pHs for particle size < 325 mesh is shown in Figure 2. It clearly indicates that lead can be leached out more from the solid waste at pH = 4.8 than pH = 6.0, 8.0 or 10.0 for the leaching temperature ranges of 25 to 75 °C. For example, at 55 °C and a 30 minute leaching time, the lead leached was found to be 96.8, 90.9, 75.3 and 74.4 % at pH 4.8, 6.0, 8.0 and 10.0 (Table 3), respectively. Further increase in the leaching time and temperature did not increase significantly the amount of lead leached from the solid (Figure 2). Figure 3 revealed the significance of the dependence of the percentage of lead leached on temperature and pH of the leaching media at a 30 min leaching time. For a given temperature of the leaching solution, the % lead leached was decreased as the pH of the leaching medium was increased.

For the purpose of energy conservation, the lead leaching bath temperature at about 55 °C seems to be a reasonable choice for industry to adopt for recovering lead from lead contaminated solid waste using EDTA as the leachant. The pH of 4.8 would be a better choice for the leaching medium based on the observation that after complexed lead is recovered by the electrodeposition process, 85% of EDTA can be recovered from this solution as solid EDTA at pH~2.
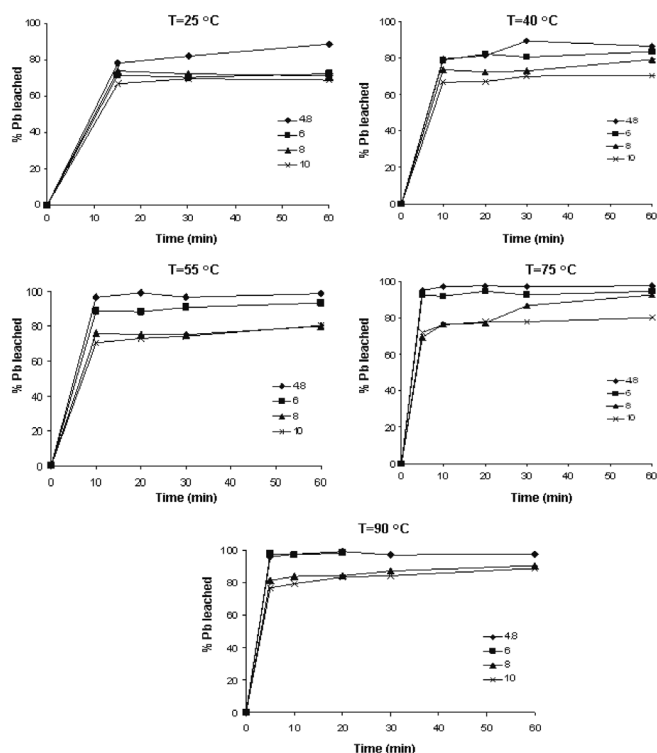
Fig. 2. Dependence of % Pb leached on time, temperature and pH (particle size < 325 mesh (0.051 mm)).
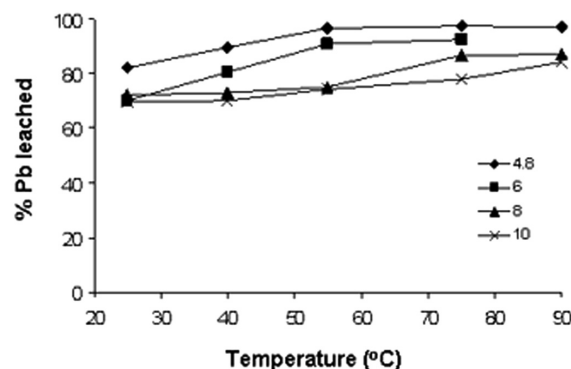
Fig. 3. Dependence of % Pb leached on temperature and pH (particle size < 325 mesh (0.051 mm) for a 30 min leaching time.

2. Dependence of % lead leached on particle size, time, temperature and pH of the leaching solution

Three different size ranges of solid waste were chosen for this investigation, namely particles of sizes >70 mesh, >200 mesh and <325 mesh. Particle size >200 mesh means that it contains all particles having sizes between <70 and >200 mesh. Extensive lead leaching experiments were performed under various parameters that affect the rate of the lead leaching process. The parameters investigated were leaching time and the pH of the leaching medium for each of these three ranges of particle size. Table 4 show the dependence of lead leaching velocity on the particle size and the leaching time at pH 4.8, 6.0, 8.0 and 10.0 at temperature range 25 to 90 °C at a 30 min leaching time. Generally speaking, leaching processes proceed faster for porous solid having smaller particle size. Table 4 illustrates this trend for our test sample solids. At a 30 min leaching time, the leaching velocities for particle size <325 mesh were observed to be out-performing those of particle size ranges of >70 and >200 mesh at all temperatures at pH 4.8. This effect may be attributed by the fact that the diffusion of leaching agent (EDTA) into the core of the porous solid particle and the removal of the leached species lead-EDTA complexes to the bulk of the leaching medium is faster for porous solid having smaller size.

3. Recovering lead and EDTA from the leaching solutions

In order to obtain the optimal electrodeposition potential for the reduction of the lead at a given cathode, cyclic voltammograms were performed for three different lead containing sample solutions; namely (a) synthetic sample solution without EDTA containing 5.00 g/L $Pb(CH_3CO_2)_2$, 0.33 g/L $Cu(CH_3CO_2)_2$, 1.80 g/L $FeCl_3$ and 0.10 M $NaBF_4$ (b) synthetic sample solution with EDTA containing 5.00 g/L $Pb(CH_3CO_2)_2$, 0.33 g/L $Cu(CH_3CO_2)_2$, 1.80 g/L $FeCl_3$ and 26.00 g/L at pH 4.8 adjusted with NaOH and (c) real sample solution obtained from the leaching the solid waste with EDTA at pH 4.8.

Figure 4 illustrates two important diagrams obtained by using MEDUSA program (Make Equilibrium Diagrams Using Sophisticated Algorithms, version 25 mar 1998); this program requires only a list of metallic ion, ligand and concentrations of both. The plot of Figure 4(a) shows the distribution of the species Pb^{2+}, $Pb(CH_3CO_2)^+$, $Pb(CH_3CO_2)_2$, $Pb(CH_3CO_2)_3^-$ and $Pb(OH)_2$ as a function of pH when the concentration of Pb^{2+} is kept constant at 13.20 mM. The plot of Figure 4(b) shows the distribution of species Pb^{2+}, $PbH_3(EDTA)^+$, $PbH_2(EDTA)$, $PbH(EDTA)^-$, $Pb(EDTA)^{2-}$ and $Pb(OH)_2$ as a function of pH when the concentrations of Pb^{2+} and $CH_3CO_2^-$ are kept constant at 13.20 mM and 26.00 mM respectively. In this diagram one can see that at EDTA concentration higher than 15.00 mM, the predominant specie is $Pb(EDTA)^{2-}$ which is not affected by the presence of acetate ion.

The cyclic voltammograms of the three lead containing solutions are shown in Figure 5(a), (b) and (c). In all measure-

Table 4. Dependence of % Pb leached on particle size and temperature of the leaching media for a 30 min leaching time and pH of the leaching media of 4.8.

Particle size (mesh) T (°C)	>70	>200 % Pb leached	<325
25	78.1	82.2	82.2
40	85.0	85.6	89.5
55	93.2	93.2	96.8
75	95.8	96.2	97.5
90	95.9	96.7	96.9

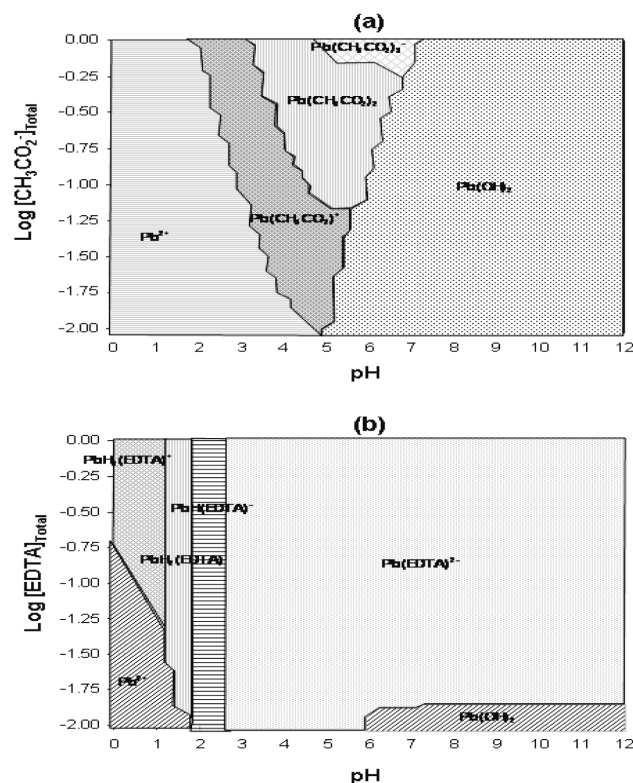**Fig. 4.** Predominance diagrams as a function of pH for (a) species lead and acetate ($[Pb^{2+}]_{Total}$ = 13.20 mM) and for (b) species lead and EDTA ($[Pb^{2+}]_{Total}$ = 13.20 mM, $[CH_3CO_2]_{Total}$ = 26.00 mM) calculated by the MEDUSA program.

ments carbon paste was used as the working electrode, Ag/AgCl and platinum wire were used as reference electrode and counter electrode respectively. For the synthetic solution containing no EDTA, the cyclic voltamogram trace from +0.10 to −1.40 and −1.40 to +1.0 volts is shown in Figure 5(a); the cathodic scan reveals that the reduction of the uncomplexed copper and lead ions occurred at about -0.50 and −0.80 volts, respectively. The dissolution potentials of deposited lead and copper from the working electrode took place at about −0.18 and +0.10 volts correspondingly. For sample solution containing EDTA, due to higher stability of the complex
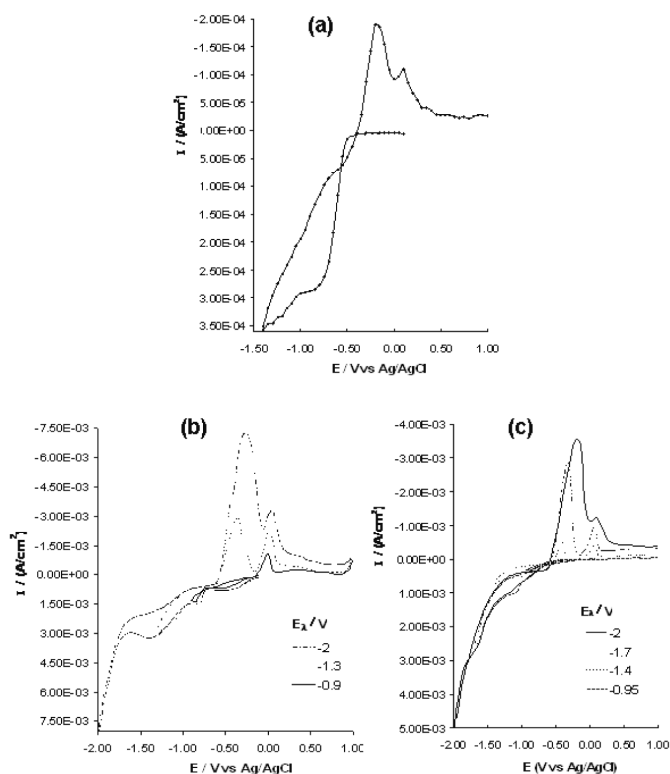
Fig. 5. Cyclic voltammagrams for different solutions. (a) Synthetic sample without EDTA prepared with 5.0 g/L Pb(CH$_3$CO$_2$)$_2$, 0.33 g/L Cu(CH$_3$CO$_2$)$_2$, 1.8 g/L FeCl$_3$ and 0.1 M NaBF$_4$. (b) Synthetic samples with EDTA prepared with 5.0 g/L Pb(CH$_3$CO$_2$)$_2$, 0.33 g/L Cu(CH$_3$CO$_2$)$_2$, 1.8 g/L FeCl$_3$ and 26.0 g/L EDTA at pH 4.8 adjusted with NaOH. (c) Real samples solution obtained from leaching at pH 4.8. Scan rate: 100 mV/s.

metal ion, we expect that the electrodeposition potentials of the complexed metal ions would shift to more negative potential than the free metal ions, so does the redissolution potential of the metal to form complex ion with EDTA. As shown in Figure 5(b), the reductions of copper and lead ion complexes occur at about −0.9 and −1.30 volts, respectively. In fact i$_{peak}$ of redissolution of lead was absent when the reverse potential of the cathodic scan was set at −0.90 volt, at this reverse potential the redissolution of copper took place at about +0.08 volt. When the cathodic scan reverse potential was at -1.30 volts, the redissolution potentials of lead and copper were observed to take place at −0.37 and +0.08 volts correspondingly. There cyclic voltammograms of the real sample solution shown in Figure 5(c) were obtained for three different reverse potentials, they were +0.10 to −0.95 and −0.95 to +1.0, +0.10 to -1.4 and −1.40 to +1.00 and +0.10 to −2.0 and −2.0 to +1.0 volts. The feature of these cyclic voltamagrams appeared to be similar to that of Figure 5(b); i$_{peak}$ related to the redissolution of lead was absent when the reversed potential was −0.95 volt, the redissolution of copper was observed to take place at +0.08 volt. When the reverse potential was set at −1.40 volts, the i$_{peak}$ of redissolution of lead was observed to occur at about

−0.38 volt. Based on our cyclic voltamagram results, for a practical rate of electrodeposition of lead at a graphite cathode, the potential applied should be in the range of −1.30 to −2.0 volts in reference to Ag/AgCl reference electrode.

For environmental and economical considerations, EDTA should be recovered from the lead leaching solution after lead is reclaimed. After 99% lead was recovered from our leaching solution, the pH of the real sample solution was lowered to ~2 by addition of hydrochloric acid, about 85% of EDTA used for the leaching process was recovered as EDTA solid.

Conclusions

Even though over 90 % of used automobile batteries are being recycled each year, the lead content in these used batteries has never been 100 % reclaimed. The current major recycling method for lead-acid batteries employs a pyrometallurgical process; besides producing metallic lead, this thermal process also oxidizes some lead metal and converts other lead compounds to lead oxides, these lead oxides mix with the rest of the solid waste left behind in the furnace. No economical technology has been developed to clean up this highly lead-contaminated solid waste (~ 22 % in lead). The current method of disposing this lead solid waste is to bury it in a designated remote area. This method of disposing such solid waste is not a positive approach to solve the environmental problem. Our experimental work on optimizing the conditions for leaching lead from the actual lead-laden industrial solid waste by EDTA at various leaching times, temperatures, pH of the leaching medium and different sizes of the solid waste may provide valuable technical information to the battery recycling industries for reclaiming the lead from this solid waste. Our experimental results indicate that, at pH 4.8, a leaching temperature of 55 °C and a 30 min leaching time, 96.8% of lead present was recovered from the solid waste having particle size < 325 mesh. Lead and solid EDTA recovered from the leaching solution can be reused. We believe that the lead-acid battery recycling industries can easily adopt these manageable leaching conditions; recovering lead from lead-contaminated solid waste generated by the pyrometallurgical lead-acid battery recycling process is technically doable and economically feasible. The success of optimization of the process for recovering lead from the waste solid generated during the pyrometallurgical process of recycling used automobile batteries may open a profitable and environmental friendly business for the private sector's participation in clean-up of this solid waste.

Acknowledgment

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