11th International Conference "Environmental Engineering"
Vilnius Gediminas Technical University
Lithuania, 21–22 May 2020
Section: Environmental Protection and Water Engineering
http://enviro.vgtu.lt

eISSN 2029-7092 / eISBN 978-609-476-232-1

Article ID: enviro.2020.675
https://doi.org/10.3846/enviro.2020.675

Filtrates and Sludge Generated in the Physicochemical Treatment of Wastewater from the Lead-Acid Batteries Production

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Received 04 March 2020; accepted 12 March 2020

Abstract. Total lead (TPb) exists in Crude Wastewater (CW) from production of Lead-Acid Batteries (LABs) in water (WPb) and solid phase (SPb) as colloids and suspended solids.

Sludges produced in chemical treatment of these wastewater were dewatered in Chamber Pressure Press (CPP). Samples of dewatered sludges (Ss) were analyzed with Toxicological Characteristic Leaching Procedure (TCLP) to determine concentration of Pb in the extract (Ex(TCLP)). Selected sludges were also analyzed using Tessier's procedure for fractions. Concentration of lead in filtrates (Fs) as well as in the sludges were different, depending on the mechanisms involved in converting soluble lead to its less soluble forms. Sludges produced in chemical treatment with 10% NaOH and Lime Milk (LM), CaO or $Ca(OH)_2$, followed by coagulation with (Ixonos Na3T ®) contained lowest concentration of lead. The TPb in filtrates below 0.4 mg/L at pH > 8.90±0.3, and Ex(TCLP) of less than 5.0 mg/L were obtained in this treatment. Potential recovery of lead from sludges have been investigated.

Keywords: lead-acid batteries production, industrial wastewater treatment, sludge, TCLP leached lead, Pb fractional analysis.

Introduction

Transport vehicles are using the largest number of produced LABs (May et al., 2018; Davidson et al., 2016; Pavlov, 2017; Sladić et al., 2018). Production of LABs results in wastewater with Pb >1.0 mg/L and a wide range of concentration of sulfates, sometimes exceeding 1000 mg/L (May et al., 2018; Davidson et al., 2016; Pavlov, 2017; Sladić et al., 2018; Macchi et al., 1993; Kulkarni et al., 2018). These wastewaters are often pre-treated by neutralization combined with multi-stage precipitation of lead (Kulkarni et al., 2018; Macchi et al., 1996; Maruthamuthu et al., 2011; Matlock et al., 2002; Sadeghi et al., 2017). Alkaline solutions of NaOH, $NaHCO_3$, Na_2CO_3, Na_2S, as well as mixture of trisodium salts of 1,3,5 Triazine 2,4,6 (1H,3H,5H)- trithione (with commercial names like Ixonos Na3T® or TMT® 15), or alkaline dispersions of $Mg(OH)_2$, CaO or $Ca(OH)_2$ i.e. $CaCO_3$ are used for precipitation (Macchi et al., 1993, 1996; Patterson et al., 1977; Wallace & Singer, 1981; Matlock et al., 2001; Decostere et al., 2009; Wang et al., 2018; Zhang et al., 2016; Ye et al., 2017; Rauckyte-Żak, 2019).

Precipitation of ionic species is determined by pH. Level of pH and complexing capability with available simple anions determine the final concentration of lead in the wastewater treatment effluent (Luo & Millero, 2007; Byrne & Miller, 1984). The literature also discusses the importance of activity of species directly binding Pb^{2+} (Odyakov & Zhizhina, 2005), their molar concentration in excess of that of the metal (Matlock et al., 2002; Zhao et al., 2019) and the presence and excess of anions (Byrne et al., 2010; Packter & Sahay, 1974), especially coordination ligands, for example Cl^-, OH^-, NO_2^-, NO_3^- or HCO_3^- (Macchi et al., 1993, 1996; Peters & Ku, 1987).

Chemical addition and mixing conditions can affect the removal of lead due to changes in dynamic and kinematic viscosity (Macchi et al., 1993, 1996). It is important to note, that the type of reagent used for precipitation will affect the particular structure of the sludge formed and its dewatering ability (Decostere et al., 2009; Rao & Raju, 2010).

Objectives

The objective of this study was evaluation of lead concentrations in CPP dewatered, sludge samples (Ss) and filtrates. TCLP procedure was used to leach the lead from dewatered sludge. The sludge samples analyzed were generated in physico-chemical pre-treatment of crude wastewater (CW) from production of LABs, which included

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neutralization and one or two-stage precipitation with various reagents. The sludge samples were collected from operational, full scale industrial wastewater treatment facility (http://www.projprzemeko.pl).

1. Materials and methods

1.1. Industrial wastewater pre-treatment system

The pre-treatment system train consists of two-compartment wastewater influent equalization storage tank with grit removal filter 5×5 mm. The wastewater is mixed mechanically, to uniformily distribute the components, in the first compartment, past the grit filter. The second chamber is used as a primary settling tank with irregular inflow rate. The wastewater is then transported in a flow-through pipe-reactor to a custom designed, central reactor where main physico-chemical treatment takes place (http://www.projprzemeko.pl). Reagents are dosed in-line depending on the influent and effluent pH in the flow through pipe-reactor. The wastewater, with addition of coagulant, is flowing into the central reactor, where flocculation and sedimentation takes place. Such pre-treated wastewater is directed to a secondary-settling tank with high rate settling modules; the effluent is discharged to the sewer. Sludge from primary settling tank and/or sludges from the secondary settling are thickened with addition of flocculant (0.3% Praestol 2500 Brenntag NV), and CPP dewatered at about 12.0–12.5 atm.

1.2. Chemical reagents

Commercially available reagents are used in the treatment (NaOH, Na_2CO_3, CaO and $Ca(OH)_2$ to prepare Lime Milk (CaO) and LM ($Ca(OH)_2$)) as well as Ixonos Na3T ® Brenntag NV) (https://www.brenntag.com/pl-pl/oferta). Prior to pressure dewatering, the sedimentation sludges from the central reactor were thickened with 0.3% flocculant solution Praestol 2500.

1.3. Basic wastewater characteristics and treatment

CW (Table 1) were mixtures of two steam generated in production lines: I) (pH 7.6–12.3) preparation and lead paste fill II) (pH < 1.0–3.6) electrolyte fill and battery charging.

Table 1. Characteristics of CW

No	Parameter (Abbreviation)	Unit	Measurements$^{a)}$ (median ½)
1	pH	pH	1.73–5.39 (4.06)
2	Total suspended solid (TSS)	mg/L	7.57–56.11 (12.30)
3	Total lead (TPb)	mg/L	2.05–9.14 (3.86)
4	– lead in water phase (WPb)	mg/L	1.14–4.33 (1.70)
5	– lead in solid phase (SPb)	mg/L	1.01–7.03 (3.05)
6	Sulfates (SO_4)	mg/L	147.07–> 1000 (458.22)

Note: $^{a)}$ based on 69 measurements at full scale plant.

Dewatered sludge samples analyzed in this paper, were formed as a result of full physico-chemical pre-treatment options described in Table 2.

Table 2. CW Pre-treatment options

No	Option	Neutralization and precipitation reagents $^{a, b)}$
1	A $^{a)}$	Self-neutralization in equalization storage tank
2	A1 $^{b)}$	1^o) 10% NaOH
3	A2 $^{c)}$	2^o) 10% NaOH
4	B1 $^{b)}$	1^o) 7.5% Na_2CO_3
5	B2 $^{c)}$	2^o) 7.5% Na_2CO_3
6	C1 $^{b)}$	1^o) 5.0% $CaCO_3$
7	C2 $^{c)}$	2^o) 5.0% $CaCO_3$
6	D $^{d)}$	2^o) 5.0% LM (CaO)

End of Table 2

No	Option	Neutralization and precipitation reagents [a, b)]
7	E [d)]	2°) 5.0% LM ($Ca(OH)_2$)
8	F [e)]	2°) 10.0% NaOH (to pH ca 7.5), 3°) 5.0% LM (CaO)
9	G [e)]	2°) 10.0% NaOH (to pH ca 7.5), 3°) 5.0% LM ($Ca(OH)_2$)
10	H [f)]	1°) 10.0% NaOH (to pH ca 7.0), 3°) 5.0% LM (CaO), 4°) 15.0% Ixonos Na3T ®
11	I [f)]	1°) 10.0% NaOH (to pH ca 7.0), 3°) 5.0% LM ($Ca(OH)_2$), 4°) 15.0 % Ixonos Na3T ®

Notes: [a)] in this option sedimentation sludge is directed to thickening tank and CPP system; [b)] sludge from this option is formed in 2 stages: 1°) primary neutralization of acid wastewater (to pH about 7.0) in a separate independent train using A1) 10% NaOH or B1) 7.5% Na_2CO_3 or C1) 5.0% $CaCO_3$ and 2°) neutralization with alkaline waste from paste production in storage-equalization tank, and directing to CPP system like in option A; [c)] sludges from options A2, B2 and C2 were formed in 2-stage treatment: 1°) after CW equalization but without primary sedimentation 2°) with the same reagent dose after separation in the central reactor and transport to thickening tank in CPP system; [d)] analogical to [c)] but with LM dosing; [e)] analogical to [c)] and [d)] but dosing 2 reagents to the flow-through pipe – reactor; [f)] in these options 1°) acid wastes are initially neutralized with 10% NaOH in a separate train 2°) mixed with alkaline wastes from paste filling without primary settling 3°) dosing of 5.0% Lime Milk (5.0% LM (CaO) or 5.0% LM ($Ca(OH)_2$)), while 4°) dosing 15.0% Ixonos Na3T ® at 80 mL/m^3.

1.4. Analytical methods

1.4.1. Determination of lead concentration in filtrates

Samples of filtrates (Fs) for analysis of concentration of lead TPb were collected directly from pressure dewatering of sludge samples generated in various options listed in Table 2. Emission spectrophotometer ICP-OES (Jobin Yvon EMISSION JY 38S) and procedure (ISO 11885:2007) was used.

1.4.2. Evaluation of sludges in terms of lead leaching – TCLP procedure

Evaluation of CPP dewatered sludges from dewatered samples were conducted according to TCLP method (USEPA, 1992, Method 1311), determining leachable forms of Pb. The USEPA Method 1311 procedure was followed closely (extraction solution with 2.89±0.05 was prepared with 80% AcOH).

1.4.3. Determination of fractions

Sequential fractions were determined from dewatered sludges for selected treatment options according to the Tessier procedure (Tessier et al., 1979). 5.0 g of dewatered sludge was analyzed in duplicate.

2. Results and discussion

TPb removal efficiency and unit costs (including reagents) was considered when evaluating the pre-treatment options. The focus was on pre-treatment technologies that generated sludges that could be economically re-used. Option utilizing neutralization potential of loading line wastewater ($pH < 1.0–3.6$) with paste line wastewater (pH 7.6–12.3) for removal of TPb was given a priority due to low cost. However, the irregular wastewater flows from these two lines made it impossible to achieve continuous and optimal neutralization, with consistent precipitation of TPb.

In the filtrates (Fs) from CPP dewatered sludges measured TPb concentrations above 2.0 mg/L and Ex(TCLP) were greater than 5.0 mg/L, which categorize these sludges as toxic according to US EPA (Table 3).

Therefore, in the subsequent trials, this pre-treatment option was considered preliminary, minimizing the cost of reagent required for final treatment. Acidic wastes from loading line in a different option 1°) were neutralized (in a separate process) to about pH 7 with aqueous solutions of 10.0% NaOH or 7.5% Na_2CO_3 or 5.0% suspension of $CaCO_3$ (options A1, B1 and C1) and blended with 2°) alkaline wastewater from paste line in storage-equalization tank. Sludges formed in the treatment process were directed to CPP. Concentration of TPb in Fs were exceeding 1.2 mg/L and Ex(TCLP) > 5.0 mg/L, which again puts these sludges in the category of toxic wastes according to US EPA.

Two-stage treatment options were also tested in the full scale: 1°) CW pH adjustment (to pH 1.73–5.39) in the storage tanks followed by 2°) addition of solutions of 10.0% NaOH or 7.5% Na_2CO_3 or 5.0% $CaCO_3$ suspension. In option 2°) reagents were dosed in-line directly based on initial and final pH in the flow-through pipe reactor (options A2, B2 and C2). The formed sludges were thickened and pumped to CCP system (http://www.projprzemeko.pl). Measured concentrations of TPb and Fs as well as Ex(TCLP) indicated that additional treatments are required to improve removal of lead. Concentrations of TPb in alkaline Fs ($pH > 9.2$) were greater than 0.7 mg/L. While concentrations of lead in Ex(TCLP) from dewatered sludges were greater than 5.0 mg/L. Therefore, the methods based on initial neutralization of acidic wastewater from loading line with alkaline water from paste line or solutions of 10% NaOH or 7.5% Na_2CO_3 or 5.0% $CaCO_3$ suspension can only be considered as preliminary treatment, rather than a final one. Consistence of this preliminary treatment was significantly affected by the variable flow of wastewater into the storage tank. The variable wastewater flow made it very difficult to optimize chemical dosing to obtain consistent removal of TPb. Application of one-reagent treatment with 5.0% LM prepared from calcium oxide (LM (CaO)) or calcium hydroxide (LM ($Ca(OH)_2$)) did not result in significantly higher concentrations of TPb in Fs nor higher Ex(TCLP) in dewatered lime sludges. Introduction of suspended solids in solutions of LM significantly improved dewatering of the sludges, but also increased the sludge mass, without clear improvement in removal of TPb in Fs. The quality of Ex(TCLP) have not improved either. However, application of 3-reagents precipitation with initial neutralization of acidic wastes with 10.0% NaOH gave the lowest lead concentrations in Fs and Ex(TCLP) (options H and I). In these options, the treatment process was clearly determined by the presence of suspended-colloidal LM. This indicated the importance of control of pH, especially in the effluent of the central reactor. In these options, external OH^- anionic forms present in Ixonos Na3T ® OH were not only converting lead to its low soluble forms, but also competed for stronger binding with Pb^{2+}, improving lead removal from water. Cation lead species react with anions with different speed and effectiveness, depending on contact time, pH, and rheological properties of the wastewater, especially when LM suspension is introduced. The results from full scale wastewater treatment systems, with inconsistent parameters are difficult to compare with the literature reports obtained in controlled conditions.

Table 3. Pb concentration measured in CW, Fs and Ex(TCLP) in the tested options

Option [a]	Concentration TPb in CW, mg/L	Charge pH in Fs	Concentration TPb in Fs, mg/L	Median ($m_{1/2(Fs)}$), [a] mg/L	Concentration Pb in Ex(TCLP), mg/L	Median ($m_{1/2(Ex(TCLP))}$), [a, b] mg/L
A (one-stage)	3.90–5.04	1. 5.64±0.31	2.11–3.80	2.93	>5.0	>5.0
A1 (two-stage)	2.87–4.30 3.21–4.17	1. 7.62±0.27 2. 8.40±0.32	1.30–2.39 1.21–2.19	1.61 1.53	>5.0 >5.0	>5.0 >5.0
A2 (two-stage)	3.40–4.73 2.30–3.43	1. 7.58±0.31 2. 8.73±0.28	1.33–1.84 1.22–2.12	1.62 1.59	>5.0 4.09–> 5.0	>5.0 >5.0
B1 (two-stage)	2.60–3.92 3.02–3.95	1. 8.03±0.30 2. 9.25±0.32	1.39–2.30 1.24–1.80	1.57 1.40	4.55–> 5.0 4.20–> 5.0	>5.0 >5.0
B2 (two-stage)	3.42–4.52	1. 9.31±0.25	0.76–1.48	1.07	4.60–> 5.0	>5.0
C1 (two-stage)	2.87–4.30 2.64–3.50	1. 7.77±0.34 2. 8.58±0.30	1.40–2.10 1.26–2.15	1.72 1.46	4.96–> 5.0 3.88–> 5.0	>5.0 >5.0
C2 (two-stage)	3.38–4.50	1. 9.20±0.31	0.85–1.41	1.01	4.40–> 5.0	>5.0
D (two-stage)	2.38–3.40 2.90–4.22	1. 7.96±0.37 2. 8.90±0.35	0.44–0.69 0.30–0.71	0.60 0.49	4.66–> 5.0 4.90–> 5.0	>5.0 >5.0
E (two-stage)	3.52–4.70 3.85–4.61	1. 7.73±0.30 2. 9.08±0.28	0.36–0.85 0.27–0.61	0.53 0.42	4.02–> 5.0 3.80–> 5.0	>5.0 >5.0
F (three-stage)	3.47–4.19 2.85–3.60	1. 8.16±0.34 2. 8.96±0.25	0.29–0.93 0.28–0.60	0.53 0.46	4.14–> 5.0 3.70–> 5.0	>5.0 >5.0
G (three-stage)	3.17–4.30 3.40–4.51	1. 8.62±0.27 2. 9.11±0.20	0.37–0.88 0.22–0.59	0.49 0.38	4.97–> 5.0 3.44–> 5.0	>5.0 >5.0
H (four-stage)	2.60–3.33 2.28–3.08	1. 8.38±0.31 2. 8.91±0.30	0.23–0.57 < 0.1–0.39	0.41 0.27	2.17–> 5.0 1.90–4.88	>5.0 4.02
I (four-stage)	2.68–3.77 2.73–3.80	1. 8.20±0.32 2. 9.17±0.26	0.11–0.47 < 0.1–0.30	0.40 0.24	1.70–> 5.0 1.80–4.73	>5.0 3.90

Notes: [a] median ($m_{1/2}$) ½ calculated from 10 Fs, measurement; [b] Standard US EPA – maxim level 5.0 mg/L.

Based on the Pb in Ex(TCLP) results, sequential extraction was conducted using Tessier's procedure for two option H and I (Tessier et al., 1979). The results indicated that fraction V (i.e. residual) is dominant representing over 80%, and organic fraction represented 2.1 and 3.1%. Potential recovery of lead from sludges have been investigated. Lead recovered from the sludges can be used as a source of technical grade lead.

Conclusions

Dewatered sludges, formed in one-reagent treatment of LABs wastewater (CW) did not comply with US EPA criteria, and were considered toxic waste. Such physico-chemical treatment methods turned out to be insufficient for industrial wastewater produced in production of LABs. 3 and 4 – reagent treatment based on neutralization and precipitation with

10.0% NaOH, 5.0% LM suspension prepared with CaO or $Ca(OH)_2$ with final coagulation with 1,3,5 Triazine 2,4,6 (1H,3H,5H)- trithione salts produced sludges that after dewatering could be used to recover lead in metallurgical process. USEPA criterion can be used to select reagents that would result in the lowest lead concentration in the filtrates Fs and leached from the sludges Ex(TCLP) using TCLP procedure for their potential disposal.

Acknowledgements

The authors would like to thank Projprzemeko® Sp. z o.o., for their support and engagement in this research. Without the employees involvement, it would be impossible to collect numerous samples from the full scale pre-treatment of lead-acid battery production wastewater.

References

Byrne, R. H., & Miller, W. L. (1984). Medium composition dependence of lead(II) complexation by chloride ion. *American Journal of Science*, *284*, 79–94. https://doi.org/10.2475/ajs.284.1.79

Byrne, R. H., Yao, W., Luo, Y., & Millero, F. J. (2010). Complexation of Pb(II) by chloride ions in aqueous solutions. *Aquatic Geochemistry*, *16*(3), 325–335. https://doi.org/10.1007/s10498-010-9101-4

Davidson, A. J., Binks, S. P., & Gediga, J. (2016). Lead industry life cycle studies: Environmental impact and life cycle assessment of lead battery and architectural sheet production. *The International Journal of Life Cycle Assessment*, *21*(11), 1624–1636. https://doi.org/10.1007/s11367-015-1021-5

Decostere, B., Hogie, J., Dejans, P., & Van Hulle, S. W. H. (2009). Removal of heavy metals occurring in the washing water of flue gas purification. *Chemical Engineering Journal*, *150*, 196–203. https://doi.org/10.1016/j.cej.2008.12.025

International Organization for Standardization. (2007). *Water quality – Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES)* (ISO 11885:2007). https://www.iso.org/standard/36250.html

Kulkarni, V. V., Golder, A. K., & Ghosh, P. K. (2018). Critical analysis and valorization potential of battery industry sludge: Speciation, risk assessment and metal recovery. *Journal of Cleaner Production*, *171*, 820–830. https://doi.org/10.1016/j.jclepro.2017.10.064

Luo, Y., & Millero, F. J. (2007). Stability constants for the formation of lead chloride complexes as a function of temperature and ionic strength. *Geochimica et Cosmochimica Acta*, *71*, 326–334. https://doi.org/10.1016/j.gca.2006.09.019

Macchi, G., Marani, D., Pagano, M., & Bagnuolo, G. (1996). A bench study on lead removal from battery manufacturing wastewater by carbonate precipitation. *Water Research*, *30*(12), 3032–3036. https://doi.org/10.1016/S0043-1354(96)00089-9

Macchi, G., Pagano, M., Santori, M., & Tiravanti, G. (1993). Battery industry wastewater: Pb removal and produced sludge. *Water Research*, *27*(10), 1511–1518. https://doi.org/10.1016/0043-1354(93)90095-Y

Maruthamuthu, S., Dhanibabu, T., Veluchamy, A., Palanichamy, S., Subramanian, P., & Palaniswamy, N. (2011). Electrokinetic separation of sulphate and lead from sludge of spent lead acid battery. *Journal of Hazardous Materials*, *193*, 188–193. https://doi.org/10.1016/j.jhazmat.2011.07.050

Matlock, M. M., Henke, K. R., Atwood, D. A., & Robertson, D. (2001). Aqueous leaching properties and environmental implications of cadmium, lead and zinc Trimercaptotriazine (TMT) compounds. *Water Research*, *35*(15), 3649–3655. https://doi.org/10.1016/S0043-1354(01)00091-4

Matlock, M. M., Howerton, B. S., & Atwood, D. A. (2002). Chemical precipitation of lead from lead battery recycling plant wastewater. *Industrial & Engineering Chemistry Research*, *41*(6), 1579–1582. https://doi.org/10.1021/ie010800y

May, G. I., Davidson, A., & Monahov, B. (2018). Lead batteries for utility energy storage: A review. *Journal of Energy Storage*, *15*, 145–157. https://doi.org/10.1016/j.est.2017.11.008

Odyakov, V. F., & Zhizhina, E. G. (2005). A potentiometric study of precipitation in the (Pb^{2+} - $H_2PO_4^-$ - Cl^-) system. *Russian Journal of Inorganic Chemistry*, *50*(6), 969–973.

Packter, A., & Sahay, S. (1974). The precipitation of sparingly-soluble alkaline-earth metal and lead salts with slow development of supersaturation (slow anion and hydroxyl ion addition): Kinetics of nucleation and induction periods. *Kristall und Technik*, *9*(5), 485–496. https://doi.org/10.1002/crat.19740090505

Patterson, J., Allen, H., & Scala, J. (1977). Carbonate precipitation for heavy metals pollutants. *Journal (Water Pollution Control Federation)*, *49*(12), 2397–2410. http://www.jstor.org/stable/25039473

Pavlov, D. (2017). *Lead-acid batteries: Science and technology: A handbook of lead-acid battery technology and its influence on the product*. Elsevier.

Peters, R. W., & Ku, Y. (1987). The effect of citrate, a weak complexing agent, on the removal of heavy metals by sulfide precipitation. In J. W. Patterson, & R. Passino (Eds.), *Metals speciation, separation, and recovery* (pp. 147–169). Lewis Publishers, Inc.

Projprzemeko. (n.d.). *Technologia oczyszczania ścieków z produkcji akumulatorów – JENOX Akumulatory Sp. z o.o*. http://www.projprzemeko.pl/oczyszczenie-sciekow-przem/technologia-oczyszczania-sciekow-z-produkcji-akumulatorow-jenox-akumulatory-sp-z-o-o.html

Rao, S. M., & Raju, G. C. (2010). Comparison of alkaline treatment of lead contaminated wastewater using lime and sodium hydroxide. *Journal of Water Resource and Protection*, *2*(3), 282–290. https://doi.org/10.4236/jwarp.2010.23032

Rauckyte-Żak, T. (2019). Assessment of filtrates and sludges generated by pretreatment of wastewater from the production of lead-acid batteries. *Przemysl Chemiczny*, *98*(9), 1436–1438. https://doi.org/10.15199/62.2019.9.18

Sadeghi, M., Karimi, H., & Alijanvand, M. H. (2017). Removal of lead ions from industrial wastewater using precipitation process. *Environmental Engineering and Management Journal*, *16*(7), 1563–1568. https://doi.org/10.30638/eemj.2017.169

Sladić, S., Poljak, A., & Bulić, N. (2018). Towards the new energy storage system for conventional cars. *Engineering Review*, *38*(3), 261–267. https://doi.org/10.30765/er.38.3.2

Tessier, A., Campbell, P., & Bisson, M. (1979). Sequential extraction procedure for the speciation of particulate trace metals. *Analytical Chemistry*, *51*(7), 844–851. https://doi.org/10.1021/ac50043a017

USEPA. (1992*). Test methods for evaluating solid waste. Physical/chemical methods. Method 1311. Toxicity characteristic leaching procedure (TCLP)* (EPA Publ. SW-846, 3rd ed., Vol. 1A). Office of Solid Waste and Emergency Response, USEPA, Washington, DC.

Wallace, J. R., & Singer, P. C. (1981). Precipitation of lead from a storage battery manufacturing wastewater. In *Proceedings of the 35th Industrial Waste Conference* (pp. 702–717), Purdue University, Lafayette, Indiana. Ann Arbor Science, Ann Arbor.

Wang, D. F., Li, S. H., Wang, X. Q., Li, L. X., & Zhang, X. (2018). Synergistic passivation of fly ash and TMT on heavy metals in sewage sludge. *Sustainability*, *10*(12), 4731. https://doi.org/10.3390/su10124731

Ye, M. Y., Li, G. J., Yan, P. F., Zheng, L., Sun, S. Y., Huang, S. S., Li, H. F., Chen, Y., Yang, L. K., & Huang, J. L. (2017). Production of lead concentrate from bioleached residue tailings by brine leaching followed by sulfide precipitation. *Separation and Purification Technology*, *183*, 366–372. https://doi.org/10.1016/j.seppur.2017.04.020

Zhang, Z., Ren, J., Wang, M., Song, X., Zhang, C., Chen, J., Li, F., & Guo, G. (2016). Competitive immobilization of Pb in an aqueous ternary-metals system by soluble phosphates with varying pH. *Chemosphere*, *159*, 58–65. https://doi.org/10.1016/j.chemosphere.2016.05.082

Zhao, Y., Tian, S., Gong, Y., & Zhao, D. (2019). Efficient removal of lead from water using stabilized iron sulfide nanoparticles: effectiveness and effects of stabilizer. *Water, Air, and Soil Pollution*, *230*, 1–14. https://doi.org/10.1007/s11270-019-4157-2

Nomenclature

Abbreviations

CW – crude wastewater;
CPP – chamber pressure press;
Ex(TCLP) – extracts with TCLP procedure;
Fs – filtrates;
LM – lime milk;
LABs – lead-acid battery;

PW – pretreatment wastewater;
SPb – Pb in solid phase;
Ss – sludge samples;
TCLP – toxicological characteristic leaching procedure;
TPb – total lead;
WPb – Pb in water phase.