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Morphological and Elemental analysis of the effluent of Lead-acid battery manufacturing

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ABSTRACT

Excessive release of Lead (Pb) contaminated effluent into the water bodies become the lead poisonous. This waste water is the main source for the transfer of Lead (Pb) into the living organisms. Lead (Pb) is a poisonous heavy metal that accumulates in the tissues of organisms when it is taken excessively through drinking water. This bioaccumulation disturbs functional activity of the tissues in living organisms. In this research the morphology of the lead (Pb) compound precipitate powder was analyzed using Scanning electron microscope (SEM). This precipitate was also analyzed for the elemental composition using Energy dispersive X-ray (EDX), which is coupled with SEM. From the EDX results, Weight and Atomic percentages of Lead (Pb) in the precipitate were 62.81 and 13.05 respectively in spectrum 1 and 69.32 and 17.51 respectively in the spectrum 2. And also the concentration of lead (Pb) was determined as 1.21 mg/L using Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES). Temperature, pH, Chemical oxygen demand (COD), Biochemical oxygen demand (BOD) and turbidity of the effluent were also measured as 14^0C, 2.34, 448 mg/L, 173 mg/L and 163.4 NTU respectively.

1. INTRODUCTION

Heavy metals such as arsenic, cadmium, chromium, copper, lead, mercury and zinc are not degradable by living organisms and hence they are accumulated in the cells of living organisms especially humans through various mechanisms including complexation, adsorption, precipitation and active transport. This causes damage to the tissues [1, 2, 15, 16]. There are two ways for heavy metals to reach humans through water bodies and food derived from water bodies [6]. There are permissible limits to these heavy metals release into the water bodies given by the different organizations like EPA, WHO and Indian standards, which indicate exceeding of that permissible limits causes severe hazardous to the environment. Therefore they should not be exceeded more than those permissible limits. But heavy metals are released into the water bodies from accidental discharges of industries such as alloy manufacturing, cosmetics, electroplating, chemical fertilizers and pesticides, lead batteries, glass operations, metal plating, mining, paint and pigments [3, 4 & 7]. Since the permissible limits of heavy metals are being exceeded in the drinking water and wastewater, pollution from heavy metals has become a serious problem all over the world [5]. There are five routes to exhibit the toxicity of heavy metals such as binding and blocking the essential functional groups biomolecules such as carbohydrates, lipids, proteins and specially enzymes; removing essential metal ions from their native binding sites; disturbing the conformational structure of biomolecules; degrading essential metabolites; and changing the ion transport across the cells [12]. In this manner pressures from these toxic metal polluted environment disturbs the metabolic activities of living organisms [14]. So the survival ability of the organism in the presence of metal toxicity depends on its intrinsic properties [15 & 16]. Lead (Pb) is one of the heavy metals used in various manufacturing units such as mining, smelting and battery manufacturing [8, 9, 17 & 18]. Lead is discharged in to the nature by anthropogenic activities and entered into the food chain [10 & 11]. Pb is not useful element to the plant and animals. The toxic effects of Pb depend on its concentration and the age of person exposed. Low concentration of Pb affects the mental and physical growth of children. Exposure of high concentrations of Pb leads to the anemia, brain damage, hyper tension, muscle weakness and kidney problems finally reach to death in children [17, 18].

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Lead pollution affects metabolic activity, respiration, membrane transport and ribosome and protein synthesis and activities, which results in the cell death [13]. According to the Indian Standard (IS 10500 1992), EPA and WHO the maximum allowable limits of Pb (II) ion concentration in drinking water 0.05, 0.015 and 0.01 mg/L respectively [20-23]. The significance of morphological and elemental compositional characterization of chemical compounds has recently been receiving great importance, because these studies provide useful information about chemical properties, sources and history of the chemical compounds [29, 30, 31]. So the elemental analysis of a particle gives information about the source, formation and effect on environment health [30, 34]. Therefore Scanning electron microscopy with Energy-dispersed X-ray analysis (SEM/EDX) was commonly used for morphological and elemental compositional analysis of precipitated chemical compounds [32, 33]. Based on the above analysis, in this paper morphology and chemical compositional analysis was performed using SEM-EDX for identification of lead (Pb) and investigated concentration of Pb with the physicochemical parameters in the effluent from Lead acid battery industry.

2. MATERIALS AND METHODS

2.1. Chemicals

All the chemicals used in the protocols were purchased from Sigma, Himedia and Merck.

2.2. Sample collection

Effluent sample was collected and preserved according to the APHA from Nile Limited battery manufacturing, Nalgonda, Telangana, India [19].

2.3. Lead (Pb) detection

Effluent sample pH was increased by adding NaOH. Then samples were incubated 48 hours. Formed precipitation was removed and then completely dried at 50^0C in the hot air oven (Thermo scientific). After that that dried precipitate was converted into powder form and sent to computer controlled SEM (VEGA3 TESCAN LMU) and EDX (INCAx-act, 51-ADD0007) for morphological and elemental analysis [25]. Powder was poured and spread over on the plastic stubs for gold coating. Gold coating was done on the powder sample using vacuum coating unit called Gold Sputter Coater (SPI-MODULE). This was made the sample electrically conductive. Then the sample was placed in the SEM-EDX chamber. Then the operating condition was set to 15 kV voltages. Images were taken at different ranges. Then the EDX analysis was also performed at specific points in terms of both qualitatively and quantitatively. The weight and atomic percentage of the elements present in the spectrum 1 & 2 were measured.

2.4. Lead Determination

Lead concentration in the effluent was determined by inductively coupled plasma method using ICP-AES (Model: ARCOS from M/s. Spectro, Germany). General Specification of ICP-AES as given in the source website IIT Bombay [24].

Table 1: General Specification of ICP-AES.

R.F. Generator	Maximum of 1.6 KW, 27.12 MHz
Plasma	Radial plasma, having capability to analyze aqueous solutions with high dissolved solid content even up to 30 wt %. Aqueous solutions can be acidic, basic or neutral
Spectrometer	Wavelength Range: 130 nm to 770 nm, Resolution: approx. 9 Pico meter, having capability to scan full spectrum to have qualitative information about the content of the sample
Detector	Charge Coupled Devices (CCD)
Torch	Vertical Torch assembly having fully demountable quartz torch with individual tubes as well as a Ceramic Fully demountable torch for HF based solutions
Nebulizers	Concentric, cross flow, organic nebulizer (hydrocarbons, solvents) as well as cross flow nebulizer for HF containing solutions
Spray chambers	HF Resistant Cyclonic Chamber and hydrocarbon solution spray chamber. Spray chambers suitable for cross flow Nebulizers are available

For the measurement of Pb concentration, the EPA 200.7 certificated sample preparation method was used (Method 200.7, 1994; APHA, AWWA, WPCF, 1992).

2.5. Physical and Chemical Characterization

The pH and temperature were measured using pH meter with their manual (Oakton pH 700) [28]. COD and BOD were also determined [19]. Turbidity was measured using nephlometer (Elico CL 52D).

3. RESULTS and DISCUSSION

3.1 Lead (Pb) Detection

Many reactions are usually pH dependant. So pH plays an important role in those reactions. Due to the pH adjustment compounds present in the solution are being precipitated. Based on this, the effect of pH on the effluent solution was investigated in this research. Precipitation was formed in the effluent sample, when the pH was increased to pH 14.0 using NaOH from initial pH, since initial pH of the effluent was 2.34. After incubation for 48 hours precipitation formation was started at pH 3.23. More amount of precipitate was formed at pH 7.57 [25 & 26]. This precipitation was completely dried after removal and converted into powder. Then precipitate powder was captured in different resolutions using SEM. The precipitate powder looks as small rod like particles as shown in all figures. Figures 1, 2, 3 and 4 show images of precipitate powder particles in 2, 5, 10 and 20 µm resolutions respectively. Single rod image of precipitate particle was shown at 2 µm range in figure 1. The elemental composition analysis of precipitate particle was done using EDX at spectrum 1 and 2 as shown in figures 5 and 6. Analysis on the precipitate particles revealed that both spectrums contain O (Oxygen), S (Sulphur) and Pb (Lead). The elemental composition percentages are tabulated in Table 2 & 3 for spectrum 1 and 2 respectively. From the Table 2, it shows that the precipitate particles at Spectrum 1 contain O, S and Pb with the weight percentages of 27.47, 9.71 and 62.81 respectively.

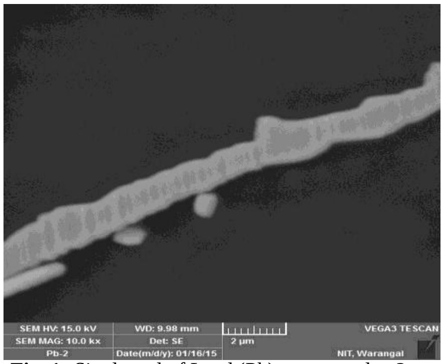


Fig. 1: Single rod of Lead (Pb) compound at 2µm.

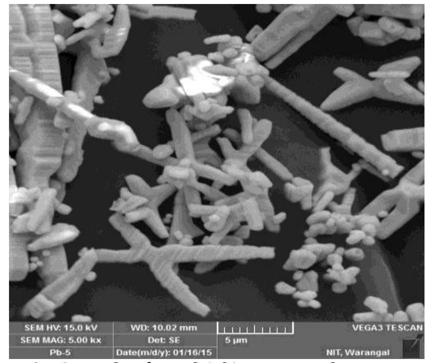


Fig. 2: Rods of Lead (Pb) compound at 5µm.

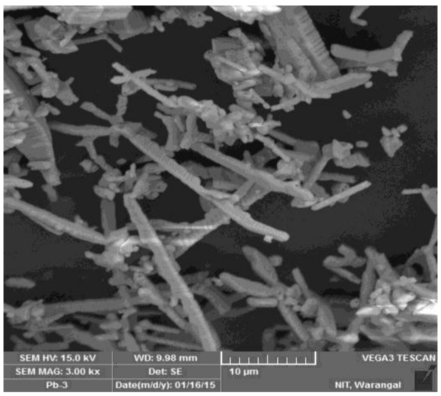


Fig. 3: Rods of Lead (Pb) compound at **10µm**.

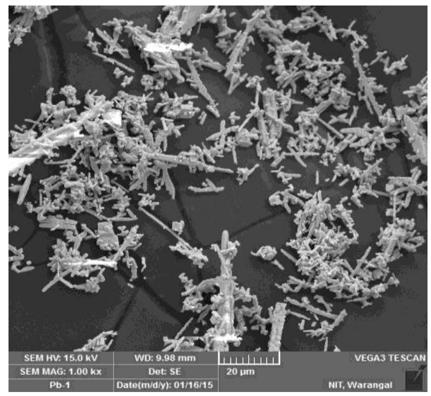
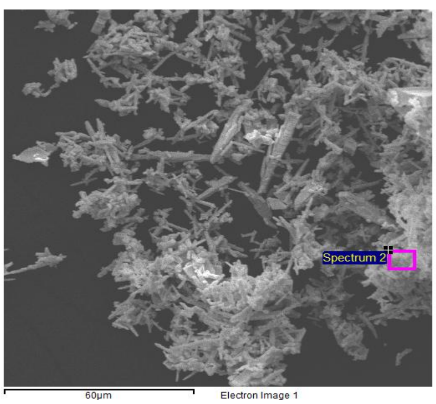


Fig. 3: Rods of Lead (Pb) compound at 20µm.



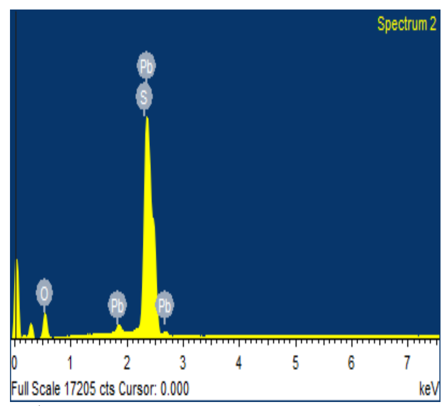
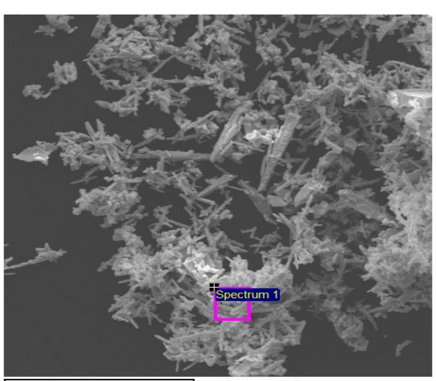


Fig. 5: EDX analysis for spectrum 1.



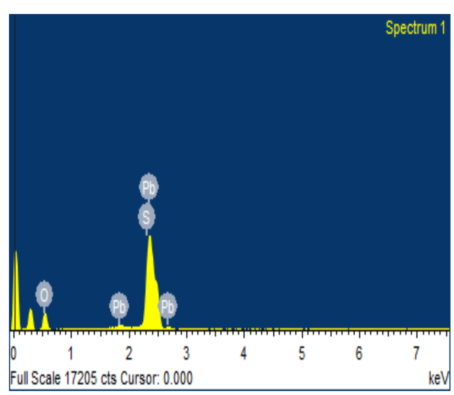


Fig. 6: EDX analysis for spectrum 2.

percentages of 19.78, 10.90 and 69.32 respectively. However, the analysis of both spectrums revealed that the element with the highest weight percentage is Lead (Pb). A higher percentage of Pb reflected a higher amount of Pb element throughout the spectrum.

Table 2: Elemental analysis of Spectrum 1

Element	Weight%	Atomic%
O	27.47	73.91
S	9.71	13.04
Pb	62.81	13.05

Table 3: Elemental analysis of Spectrum 2

Element	Weight%	Atomic%
O	19.78	64.69
S	10.90	17.80
Pb	69.32	17.51

3.2. Lead Determination

Lead (Pb) concentration in the effluent was analyzed by ICP-AES. The concentration of Pb was determined as 1.21 mg/L. This value shows that the concentration is more than the permissible range according to the EPA, WHO and Indian standards. So the effluent from this industry must be treated to reduce the concentration of Pb before releasing into the environment.

3.3. Physicochemical characteristics

Temperature, pH, COD, BOD and turbidity values are 14 ^{0}C, 2.34, 448 mg/L, 173 mg/L and 163.4 NTU (Nephelometric turbidity units) respectively as shown in the Table 4. Temperature is below than the room temperature. This pH is less than the Indian standards.

This indicates that the effluent was in acidic nature. COD, BOD and turbidity values are also greater than the Indian standards, which are in dangerous level to the aqua living organisms. If this effluent releases into the water bodies with the above parameter values, then the water bodies will be polluted. Consequently, this will affect the health of the organisms living in that environment and can reach to human beings through food cycle.

Table 4: Physicochemical Characteristics.

Parameter	Value	Indian Standards for effluent discharges
Temperature	14^{0}C	
pH	2.34	5.5 to 9.0 [21]
COD	448 mg/L	250 mg/L [21]
BOD	173 mg/L	30 mg/L [21]
Turbidity	163.4 NTU	20 NTU

4. CONCLUSION

In this study lead compounds were precipitated based on the pH dependant reaction. Pb, S and O elemental composition and morphology was analyzed in that precipitation using SEM-EDX system. And also Pb concentration was determined by ICP-AES with the physicochemical parameters. This high concentration of Pb confirms that it is far greater than the permissible range and this effluent can increase the Pb concentration in the environment, which becomes hazardous to the living organisms. Physicochemical characteristics such as pH, COD, BOD and turbidity of the effluent tell the dangerous situation of the environment that leads to the severe damage to the aqua organisms and finally damages the tissues of human by accumulation, if this effluent gets released into the environment. To keep the pollution under control, the effluent must be treated well to reduce Pb concentration and other physicochemical properties before releasing into the environment.

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