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SEDIMENTATION RATE AND CHRONOLOGY OF As AND Zn IN SEDIMENT OF A RECENT FORMER TIN MINING LAKE ESTIMATED USING Pb-210 DATING TECHNIQUE

(Kadar Mendapan dan Kronologi As dan Zn dalam Sedimen Tasek Bekas Lombong Menggunakan Teknik Pentarikhan Pb-210)

Zaharidah Abu Bakar[1], Ahmad Saat[2*], Zaini Hamzah[1], Abdul Khalik Wood[3] and Zaharuddin Ahmad[3]

[1]Faculty of Applied Sciences,
[2]Institute of Science,
Universiti Teknologi MARA, Malaysia, 40450 Shah Alam, Malaysia
[3]Malaysian Nuclear Agency,
43000 Bangi, Malaysia.

**Corresponding author: ahmad183@salam.uitm.edu.my*

Abstract

Sedimentation in lake occurred through run-off from the land surface and settles on the bottom lake. Past mining activities might enhance sedimentation process in the former tin mining lakes either through natural or human activities. Former tin mining lakes were suspected to have high sedimentation rate due undisturbed environment for almost 50 years. To estimate sedimentation rate and metals contamination in this lake, Pb-210 dating technique was used. Two sediments cores were sampled using gravity corer from a former tin mining lake then analyzed using alpha-spectrometry and Neutron Activation Analysis (NAA). From this study, the results showed the sedimentation rate for sediment cores S1 and S2 are 0.26 cm y^{-1} and 0.23 cmy^{-1} respectively. According to sediment chronological sequences, high concentrations of As and Zn in the upper layer indicated that human activities contributed to these metals contamination in the lake sediment. Sedimentation rate and metals contamination possibly due to recent anthropogenic activities around the lake such as human settlement, farming and agricultures activities since the ceased of mining activities a few decades ago.

Keywords: Sedimentation, Pb-210, Former tin mining lake, Metals element

Abstrak

Pengenapan didalam tasik berlaku melalui aliran daripada permukaan tanah dan tenggelam ke dalam dasar tasik. Aktiviti - aktiviti perlombongan yang lepas mugkin mempercepatkan proses pemgenapan di bekas tasik lombong bijih timah samada secara natural atau oleh aktiviti manusia. Bekas lombong bijih timah disyaki mempuyai kadar penenapan yang tinggi disebabkan persekitaran yang tidak terganggu untuk hampir selama 50 tahun. Untuk mengangar kadar pengenapan dan pencemaran logam di dalam tasik, kaedah pentarikhan Pb-210 telah digunakan. Kajian dilakukan di bekas tasik lombong bijih timah di daerah Kampung Gajah, Perak. Dua turus sediment diambil menggunakan turus persampelan graviti daripada tasik bekas lombong bijih timah dan dianalisa menggunakan spektrometri alpha dan Analisa Pengaktifan Neutron (NAA). Daripada kajian ini, keputusan menunjukkan kadar pengenapan untuk turus sediment S1 dan S2 masing – masing adalah 0.26 cm y^{-1} dan 0.23 cm y^{-1}. Berdasarkan kepada turutan kronologi, kepekatan As dan Zn di lapisan atas sedimen menunjukkan aktiviti – aktiviti manusia menyumbangkan kepada pencemaran oleh metal ini didalam sedimen tasik. Kadar pemendapan dan pencemaran logam mungkin disebabkan oleh aktiviti – aktiviti antropogenik di sekiling tasik seperti penempatan manusia, penternakan dan pertanian sejak ditinggalkan beberapa dekad yang lepas.

Kata kunci: Mendapan, Pb-210, tasik lombong timah, unsur logam

Introduction

Over the past three decades, there has been an impressive increase in the volume and range of research on sedimentation. Accumulation of sediment in lakes facilitates us to obtain significant information to study about environmental changes [1, 2]. In Malaysia, a large number of former tin mining lakes have been abandoned for almost past 50 years. Lack of vegetation in abandoned mining lands render surface areas were directly exposed to rainfall impact and surface runoff. The effect of uncontrolled mining activities caused environmental problem such as deforestation and soil erosion that continue increasing the sedimentation rate [3]. Sedimentation profile also provides past evidence and rates of heavy metals contaminant deposition [4].

Pb-210 radiometric dating techniques introduced by Goldberg (1963) and expended to lake by Krishnaswami *et al*. (1971) has been used worldwide to reconstruct history of such recent sedimentation [5]. Pb-210 dating method has shown to be an ideal tracer for dating lake sediments deposited during the last 100 – 150 years which coincidence with period of environmental changes due to industrialization [6]. Conrad *et al*. (2007) recommended that Pb-210 dating method is a useful technique in investigation of changes in metals concentration on a decadal time scale. This is due to the fact that association of particles with Pb-210 making it a useful tracer for the fate of particle reactive contaminants such as trace metals.

Despite this useful potential, lack of Pb-210 dating method study has been applied in sediment of recent lakes in Malaysia. Thus this paper attempt to determined recent sedimentation rate, to reconstruct the sediment ages and metals input history using Pb-210 dating method.

Methodology

Study area

This study covers a former tin mining lake in Kampung Gajah area in the states of Perak, Malaysia that has been abandoned for more than 50 years. The lake is bordered within latitude 04°14.816″ N - 04°15.107″ N and longitude 101°2.889″ E - 101°3.125″ E. The total area of this lake is approximately 2.5 x 10^5 m^2 and the volume of water is about 1.0 x 10^7 m^3. The deepest part of lake is about 10 m, receives seasonal water flow from an opening canal at the north-east that is connected to another lake used as a duck farm and flow out to the west of lake. Thus, there is a possibly of waste from other lakes enter the study area lake and flow out through other lakes to a nearest river. The study lake located 100 m from to the main road and around 1 km from residential areas. Although mining activities ceased long time ago, local peoples still used this area for growing cows and buffaloes, duck farming and fishing. Some fishes such as Temoleh (*Probarbus jullieni*), Lampan (*Puntius schwanenfeldii*), Tilapia Merah (*Oreochromis niloticus*), Catfish and Patin (*Pangasius sp*) are the main sources that essential to the diets and generating economies especially to the local peoples.

Samples collection

There are two points where sediment samples were collected, one at the middle of lake (04°14.928″ N, 101°03.043″E) and the edge of lake (04°14.854″ N, 101°03.134″ E). Sediment samples were collected using gravity corer sampling device (5 cm diameter, 40 cm long). Sediment cores were air dried for one week then sliced into 2 cm interval and oven dried at 60 °C until constant weight. The sediment cores showed light/dark-grey colour appearance. Sliced samples were ground and sieved in 450 μm stainless steel sieves to the ensure homogeneity.

Pb-210 analysis

Total Pb-210 ($Pb\text{-}210_{tot}$) was measured indirectly by determining the activity of its grandaughter, isotope Po-210. The Po-210 activity determined by alpha spectrometry is taken as a measurement of Pb-210 activity [2]. Pb-210 was assumed to be in radioactive equilibrium with Po-210 in the sediment samples. Chemical separation in analysis is controlled by Po-209 as internal standard [7].

About 2 g of sieved samples, standard and known amount of Po-209 tracer yield tracer were weighed and leached with HNO_3 and organic matter content digested with mixture of HNO_3 and H_2O_2 to get white residue. Remaining organic matter digested again using HCI and solution converted into 0.5M HCI medium. Residual solids were filtered and solution was treated with ascorbic acid to reduce Fe (II) to Fe (III) and prevent Fe deposition [8] and plated for 24 hours. Po-209 and Po-210 in solution then spontaneously deposited onto silver disc then counted using

alpha spectrometry. Po-210 and Po-209 emission from the disc releases alpha particles that have energy of 4843 keV and 5264 keV respectively were measured using alpha spectrometry with a 450 mm^2 area. Alpha radiations were then identified by EG & G ORTEC spectrometer using silicon surface barrier detectors with efficiency ≥ 25%, energy ranged 0-10 meV and a multi-channel analyzer system. Energy resolution is ≤ 20 keV (FWHM) with a detector – to – source spacing equal to the detector diameter. The outputs from the silicon detectors are amplified and transmitted through a multiplexer system into a computer based multi-channel analyzer. The system was calibrated using Mix Standard radioactive source. Samples activity and total uncertainties expressed in Becquerel per kilogram of dry weight (Bq kg^{-1} dry weight).

Pb-210 activity in a layer deposited at time, t in the past can be determined using the following equation:

$$\text{Po-210 Activity (dpm)} = \text{Po-209 Added (dpm))} \times \text{Po-209 Peak Area x } PDC \text{ /(Po-210 Peak Area)} \quad (1)$$

where $PDC = e^{-\lambda \times \Delta t}$ is the plating date correction, with λ for Po-210 = 1.833 yr^{-1}, and Δt = date counted – date plated.

The Pb-210 activity concentration was calculated by dividing the activity of Po-210 to the mass of sample (Pb-210 Activity concentration (dpm g^{-1}) = Po-210 Activity (dpm) / Sample weight (g)). Supported Pb-210 activity at each site was determined as the average of the bottom segments that had exhibited a relatively constant activity indicative of the supported Pb-210 [9].

Metal analysis

For metal analysis, about 0.2 g of sieved samples and standard were weighed and sealed in small polyethylene vials for neutron activation analysis. The weighted samples and standards were irradiated in 750 kW TRIGA PUSPATI II nuclear reactor at Malaysian Nuclear Agency (MNA) with thermal neutron flux of 2 $x10^{12}$ $n.cm^{-2}s^{-1}$. The irradiation time, decay time and measuring time used for metals analysis are given in Table 1.

Table 1: Irradiation time, decay time and measuring time for metals analysis

Element	Irradiation time (hours)	Cooling Time (days)	Counting Time (min)	Distance from detector (cm)
As	6	4	60	7
Zn	6	21	60	2

Gamma emission were counted using Gamma spectrometer GEM series with HPGe Coaxial detector system (GEM-20180 model) equipped with multi-channel analyzer. The Canberra GEM series HPGe detector has the following specification: energy resolution of detector system is 1.88 keV and 25.4% relative efficiency at 1.33 MeV Co-60. Spectra were analyzed using GammaVision (version 6.01) and photopeak was selected based on interested element to be analyzed. Arsenic (As) and Zinc (Zn) were selected for metal analysis. For As and Zn the major photopeak at energy of 559 keV and 1116 keV were used for metals analysis. To ensure the accuracy of results, application of Certified Reference Materials (CRM) IAEA SOIL-7 and IAEA Sediment Lake-1 (SL-1), IAEA-368 (Pacific Ocean Lake), duplicate of samples and blank samples were also analyzed. Z - Score calculation was used as a measurement of acceptable variation of measured value of elements in the CRM from the certified value.

The Z-score is computed based on following equation:

$$Z = (X - C)/\sqrt{(U^2_x + U^2_c)} \quad (2)$$

where X is analytical result, C is certificate value, U_x is uncertainty of analytical result and U_c is the uncertainty of certificated value. For acceptance of the result, $-2 < Z < 2$ is anticipated while $Z < -3$ or $Z > 3$ is considered 'out of control' and need corrective action [10].

Results and Discussion

Data Verification

The quality of analyzed data for metal elements and radionuclides determined using IAEA SL-1 (Sediment Lake) and IAEA-368 (Pacific Ocean Sediment) and evaluated by Z-score is shown in Table 2. Replicate analyses of IAEA SL-1 and IAEA-368 confirm good agreement of Pb-210 activities determined with certified value .The result showed concentration of As, Zn and Pb-210 were agreed well with the certified values and the measured values in which the Z-score values lies within $-2 < z < 2$. Relative Percentage Difference (RPD) in CRM and analyzed samples were expressed as percentage (%) below than 10%. Relative Standard Deviation (RSD) was within 10% for both metals analyses and radionuclides. Blank samples did not indicate any significant contamination for the studied elements.

Table 2: Analytical value for sediment certified reference materials in comparison to certified reference values using NAA and Alpha spectrometry technique

Element	*As (µg g^{-1})	*Zn (µg g^{-1})	**Pb-210
Measured value	30.5±1.5	215.6±15.9	22.40±3.0
Certified value	27.5±2.9	223±10	23.2±3.7
Z-score	0.63	0.38	0.36
% RPD	7.17	3.31	3.42
% RSD	9.42	7.79	9.35

*SL-1, **IAEA – 368

Core Profiles

Vertical profile of $Pb\text{-}210_{tot}$ activities in the sediment cores is shown in the Figure 1. $Pb\text{-}210_{tot}$ activities versus depth for S1 showed exponentially decline, with activity concentration 175.48 – 305.86 Bq kg^{-1}. The $Pb\text{-}210_{exc}$ varies from 6.07 – 106.24 Bq kg^{-1}. $Pb\text{-}210_{tot}$ activity concentration for S2 varies from 295.58 – 367.51 Bq kg^{-1} with depth. $Pb\text{-}210_{tot}$ activities showed non linear decreased with the depth to a constant value. The $Pb\text{-}210_{exc}$ varies between 4.75 – 67.40 Bq kg^{1}.

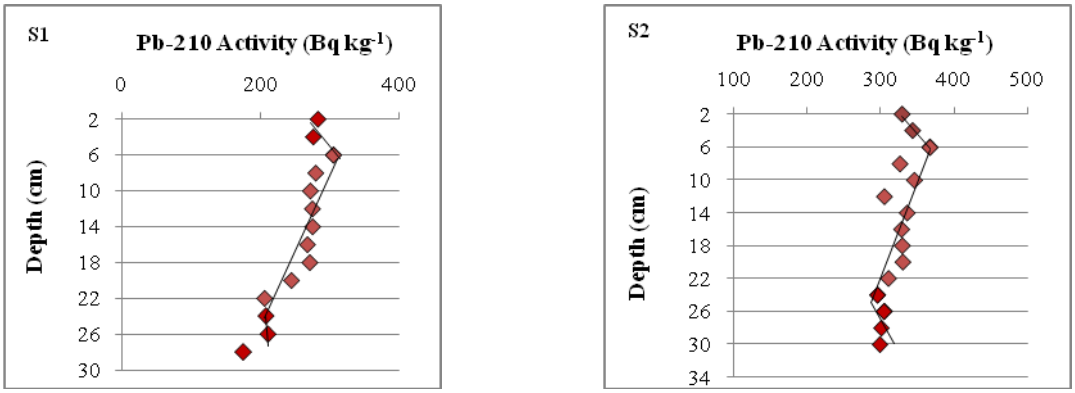

Figure 1: $Pb\text{-}210_{tot}$ depth profiles in sediment cores

$Pb\text{-}210_{exc}$ inventory was calculated as a sum of $Pb\text{-}210_{exc}$ at all depths [14]. Inventory of $Pb\text{-}210_{exc}$ in the core of S1 and S2 were estimated to be 16468 ± 128 Bq m^{-2} and 7547 ± 87 Bq m^{-2} is shown in Table 3. High inventory of $Pb\text{-}210_{exc}$ at location S1 might be attributed to high deposition of materials and acted as pathway movement of sedimentary material [1]. $Pb\text{-}210_{exc}$ inventory from atmospheric input alone is ca. 3000 – 4000 Bq m^{-2} [11]. There is

no evidence of global atmospheric fallout due to atmospheric weapon testing reached Southeast Asia region [12]. Therefore, besides atmospheric input, the Pb-210 inventory may be due to surface run–off.

Table 3: $Pb\text{-}210_{exc}$ inventory, sedimentation rate and flux in sediment cores

Location	$Pb\text{-}210_{exc}$ Inventory ($Bq\ m^{-2}$)	Sedimentation rate ($cm\ y^{-1}$)	Flux ($dpm\ cm^{-2}\ y^{-1}$)
S1	16468 ± 128	0.21 – 0.57	0.25
S2	7547 ± 87	0.24 – 0.74	0.14

$Pb\text{-}210_{exc}$ flux of the present study of in S1are similar to input of $Pb\text{-}210_{exc}$ in sediments both of the Penang Island (5° N latitude) and Johor Strait (1° – 2° N latitude) (small difference in latitude) which are about 0.30 dpm cm^{-2} y^{-1} [13]. This finding consistence with Khalik *et al*. (2004) [13] and clearly confirmed that atmospheric origin seems the main source of $Pb\text{-}210_{exc}$ supply. This is in contrast to location S2, where $Pb\text{-}210_{exc}$ inventory is two times lower than the location S1. There is a possibility that Pb-210 has been diluted or due to mobilization process.

Sedimentation rate and dating model

Sedimentation within lake occurred through run-off from the land surface and settles on the bottom lake. Once Pb-210 deposited in the sediment, they are rapidly adsorb to fine grain materials and settled down to the bottom layer. Absorption of Pb-210 to the sediment lake has been assumed a uniform process and without of sediment redistribution. In present study, CRS model used to determine the sediment age and sedimentation rate within lake sediment by assuming sediment reworking (physical and biological) and diagenesis factors were neglected.

CRS model expressed as equation below:

$$t = (1/\lambda) \ln (A_{(0)}/A_{(1)}) \quad (3)$$

where $A_{(0)}$ is total inventory of excess Pb-210 in the sediment column, $A_{(1)}$ is inventory of excess Pb-210 below depth *x*, λ is the decay constant of Pb-210 (0.693/22.3yr = 0.03114/yr) [6]. Mean sedimentation rate was determined from the slope of the least square fit for estimated age plotted versus depth.

Sedimentation rates in lake were found to vary 0.21 – 0.57 cm y^{-1} and 0.24 – 0.74 cm y^{-1} with mean sedimentation rate of sediment cores for location S1 and S2 are 0.26 and 0.23 cm y^{-1} respectively (Figure 2). Sedimentation rate determined only for the interval 2 – 16 and 2 -18 cm depth core for S1 and S2 sediment cores. Below this depth, sediment has been dominated by soil or original base of lake. Area bordering former mining areas is left without vegetation and with different contour. Depending on the gradient of bank contour this will facilitated surface runoff of rainwater into the lake that carries along overburden that causes sedimentation at different rate. This may be could be reasonable explanation for dissimilarity of depth in the sediment. Location S1 located almost at the center of lake while S2 located away from the edge of lake but closest to the central of lake. Comparable sedimentation rate for both location could be explained possibly receiving constant water flow thus created thickness of sediment at the both location almost same and have better movement of water velocity with a little obstacle.

In comparison to study by Theng *et al*. (2003) [14] at the coastal water of Sabah, Malaysia showed low sedimentation rate at Sipitang which is 0.027 cm y^{-1} and high sedimentation rate at Teluk Kimanis which is 5.53 cm y^{1} as compared to the present study. Present study showed comparable sedimentation rate with Al - Oteibeh Lake, Syria which is 0.100 – 0.79 cm y^{-1} and Lago Verde crater lake, Mexico which is 0.09 – 0.23 cm y^{-1} [2, 15].

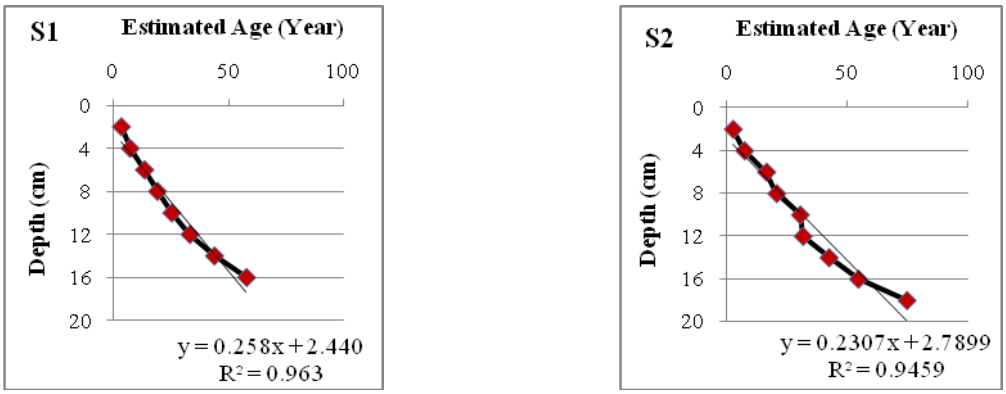

Figure 2: Profile of sedimentation rate in sediment cores

Sediment Chronology

Chronology of sediment was constructed using the CRS model. This is due to the fact that some sediment cores showed non–linear characteristics. Using CRS model, those non–linear features be interpreted by assuming a constant net rate of Pb-210_{exc} supply from the lake waters to the sediment [6]. Using the model, the supply of Pb-210_{exc} to the sediment surface is assumed constant and varying sedimentation rate between each depth. The model assumes no alteration after deposition of Pb-210, although terms of mixing and other diagenetic process can be incorporated into the calculation of dates [16]. The dating model for CRS calculation is expressed as in the equation 3. Depth profiles of Pb-210 activity and metals concentration were determined from same cores. Sediment depth converted to time based and used to date the sediment age then compare to metals contaminant within the period of time. The Pb-210 geochronology allowed the dating of sediments up to depth of 18 cm, corresponding to a period of ~ 40 years as showed in Table 4. Age of sediment core calculated using Pb-210 dating method yields reasonably precise date from ~1940 to present. S1 and S2 showed age of sediment nearly to each other. Age of sediment for core S1 and S2 showed sediment ages ranged 1940 to 2004 and 1939 – 2003 respectively.

Table 4: Chronology of sediment age with function of depth

Depth (cm) / Point	S1	S2
2	2004	2003
4	2000	1998
6	1994	1991
8	1990	1989
10	1984	1980
12	1978	1977
14	1970	1968
16	1959	1957
18	1940	1939

Depth profiles of anthropogenic metal elements concentration with age are showed in Figure 3 and Figure 4. Range of Arsenic (As) and Zinc (Zn) concentration with mean value for S1 and S2 showed in Table 5. Dating method showed small changes of sediment age within similar period and profile concentration of metals particularly were not same at each location. Constant concentration of As could be observed in the bottom sediment at both locations from year of ~1939 – 1980 possibly due to past mining activities. However, concentration of As increased beginning from year of ~ 1984 to a recent years possibly coincident with intensive development in the region of the

study area also increasing in industrialization and agriculture activities in late ~1990. Increasing of As concentration could be influence by atmospheric deposition as well as combustion, road construction and agricultural activities. Low concentration of As observed in the surface layer of sediment core S1 possibly caused by physical mixing during sampling or bioturbation by benthic microorganisms [17].

Concentration of As in the sediment lake is sensitive to redox changes as well as easily to distinguished either due to remobilization or loading changes [1]. Being a multiple oxidation state element, As is sensitive to redox changes condition in similar manner as Fe and Mn. Since as sensitive elements, As in the deeper sediment could be released into pore water via changes from As (V) to As (III) that is more mobile, soluble and toxic [18]. Some studies suggested high concentration of As at the upper layer could be affected by redox condition and adsorption of As onto Fe or Mn oxides particulate [2]. Thus, by considering of this factor, high concentration of As in the upper sediment core S2 could be possibly affected by redox condition.

Table 5: As and Zn concentration in sediment cores

	Location	**S1**	**S2**
As (ppm)	Range	39.88 – 49.55	33.41 – 104.77
	Mean	43.90	54.51
Zn (ppm)	Range	229.42 – 336.03	136.74 – 150.72
	Mean	266.64	143.68

Depth profile of Zn showed almost double concentration of Zn in the sediment core S1 as compared to S2. This is suggests that each location received different rate of metals input, since S1 being centrally location possibly acted as depocenter sedimentary material as mentioned earlier [19]. The other factor possibly due to different water flow rate causes precipitation of pollutants or variable pattern of sediment redistribution in the lake [15, 20]. Contrasting pattern was observed for S1 and S2 which lower at the recent years and vice versa for S2 as showed in Figure 4. Generally high concentration of As and Zn during the last few years possibly due to increasing use of fertilizer and pesticides in agriculture such as in some synthetic phosphorus fertilizer [20] and soil erosion due to human activities. Road construction and land development for housing areas as well as human settlement cause recent erosion in the study area and this coincide with the increase amount of metal introduced into the lake.

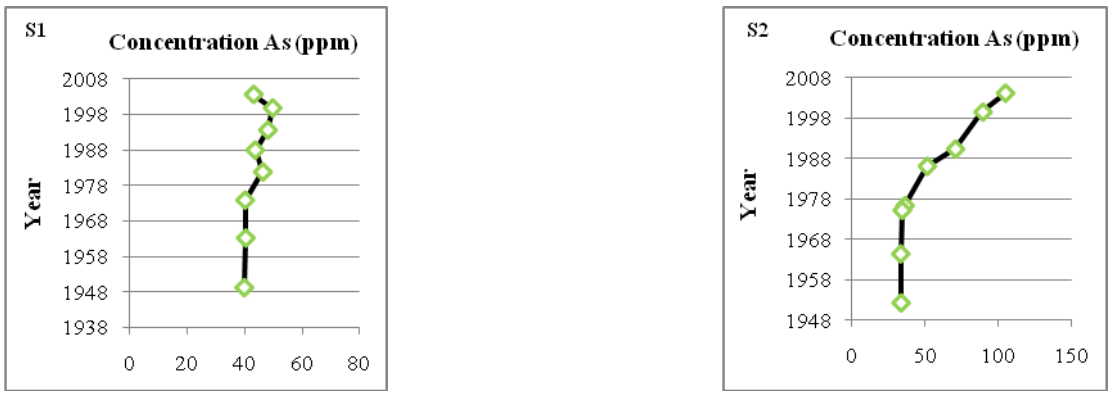

Figure 3: Profile of As concentration with respect to sediment age

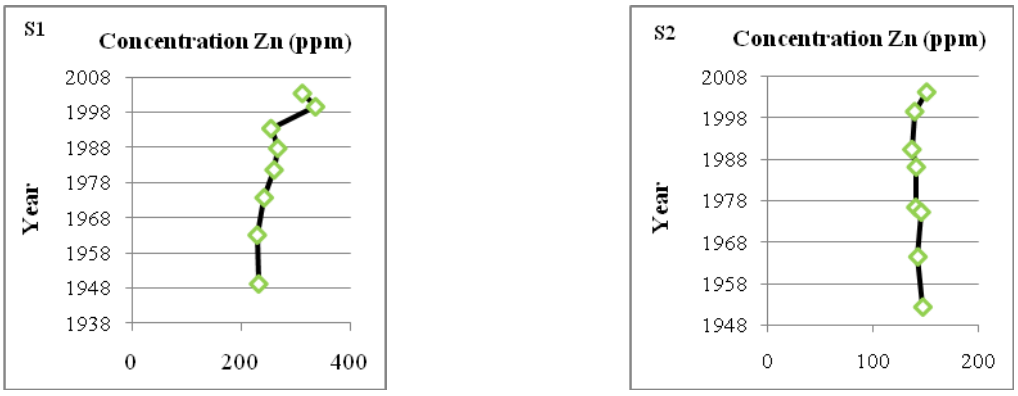

Figure 4: Profile of Zn concentration with respect to sediment age

Result of analysis showed As and Zn elements in sediment lake are good agreement with presence of these metal as earlier study by Ingham & Bradford (1960) [8] in the Geology of Kinta Valley. They were pointed out that As was found in contaminated tin-ore and removed by roasting and washing. The byproduct of As (arsenious oxide) sometimes collected for sale as an insecticide and weed killer. While, Zinc Sphalerite (ZnS) also found from several location in Kinta but there is no significant for economic value. The mineral was identified in a small quantity in limestone pipes.

Conclusion

Pb-210 dating method is applicable technique to establish recent sedimentation rate and sediment chronology in a former tin mining lake. Similar sedimentation rate showed constant flow rate of water yields same sedimentation rate at both locations. An attempt has been made to relate the metals pollution input with temporal changes in a former tin mining lake and chronology of metals input has been successfully determined. Increasing concentration of As and Zn in recent years possibly due to human activities around the lake area.

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