Sedimentation Tests Of Small Scale Gold Mining Wastewater

Jessie O. Samaniego, Maria Antonia N. Tanchuling

Abstract: Wastewater from small scale gold mining (SSGM) milling facility contains high amount of suspended solids and elevated concentration of Hg and other heavy metals. Most of these milling facilities have inadequately designed sedimentation tanks as its only treatment method, thus releasing wastewater to the receiving water bodies with heavy metals and suspended solids. In this study, sedimentation tests were conducted to investigate the removal of suspended solids and heavy metals (As, Hg, Pb) from actual small scale gold mining (SSGM) wastewater gathered from SSGM area in Paracale, Camarines Norte using settling column. A glass column with a diameter of 7.5 cm and a height of 120 cm was used to determine the removal rate of total suspended solids at settling time up to 720 mins. After 600 min of sedimentation, 100% removal of TSS was achieved. Heavy metal removal in the wastewater were tested at 100 cm from the bottom and results showed that after 480 min, removal efficiencies of 98.69%, 93.60% and 93.70% for As, Hg and Pb, respectively were achieved. The sediments collected from the settling column were analyzed for heavy metal concentrations and particle size distribution by atomic absorption spectrophotometer (AAS) and hydrometer, respectively. The X-ray diffraction (XRD) analysis result of the sediments indicates that the major component is silica.

Index Terms: SSGM, heavy metals, TSS, sedimentation, column settling test.

1 INTRODUCTION

Small scale gold mining (SSGM) in developing countries such as the Philippines are artisanal in nature and use amalgamation method to extract gold from the mined ores [1], [2], [3]. Amalgamation process uses mercury (Hg) which is cheap, easy and effective at capturing gold under the condition found in SSGM sites [1], [4], [5]. However, most SSGM ball milling facilities dispose of contaminated wastewater untreated to the tailings pond and overflows to the rivers and creeks in the area. This wastewater contains broad range of suspended solids and with high concentration of heavy metals which are detrimental to the environment and people living nearby [3], [6]. In the Philippines, most SSGM ball milling facilities have inadequately designed sedimentation tanks as its only treatment method before disposing of overflow wastewater [3]. This effluent coming out from these tanks is not completely treated and therefore it is releasing high concentrations of suspended solids and heavy metals to receiving bodies of water that do not meet the effluent standards [7]. Suspended solids carry pollutants such as organic contaminants, nutrients, bacteria and trace metals adsorbed in fine solid particles [8], [9]. Heavy metal removal in the sedimentation process is primarily influenced by metal solubility and the settleability of insoluble forms [10] as well as its adsorption to fine suspended solids, especially to the fraction < 63 μm [9].

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Environmental parameters such as pH, ORP and OM are the most important factors affecting heavy metal distributions wherein pH and OM can directly change metals distributions in sediment [11]. Previous sedimentation studies in raw sewage removed 40–70% of Cd, Cr, Cu, Pb and Zn [10]; in real industrial wastewater removed 83% and 93% for V and Pb, respectively [12], in tannery wastewater removed 71.2-83.2% of Cr [13]. Therefore, proper sedimentation process is important to reduce the suspended solids and heavy metal concentrations as it contributes to the overall removal efficiency of the treatment works before discharging it to the receiving bodies of water. Among the methods used for determining the parameters necessary for design of wastewater sedimentation tanks, settling column tests are used most commonly because of their simplicity and low costs. These tests partly mimic the actual settling processes and allow the evaluation of TSS removal by settling [14]. Other sedimentation/settling tests were carried out by using Imhoff cone [12] and settling column made of cylinder tubing [13]. Settling process of suspended solids of SSGM wastewater can be described as Class II sedimentation where heavier particles will settle fastest and some of the particles may join lighter particles to form big heavy particles that will settle in mass form [8], [15], [16]. Design of sedimentation tank can be optimized by analyzing the characteristics of the wastewater to be treated. Settling column tests can be done to determine the suspended solids removal efficiency of the settling tank and to determine the required depth and detention time of water in the tank. This study is aimed to investigate the removal efficiencies of suspended solids and heavy metals (As, Hg, Pb) from actual SSGM wastewater gathered from Paracale, Camarines Norte using column settling tests. Results from this study can be used as basis on the design of appropriate sedimentation tanks in ball mill facilities in small scale mining areas.

2 MATERIALS AND METHODS

2.1 Characteristics of SSGM Wastewater

An actual SSGM wastewater from the tailing collection tank (Figure 1) of an active ball mill facility in Gumaus, Paracale, Camarines Norte (14°17’47.04”N, 122°43’47.46”E) was used in the experiment. The ball mill facility accepts ores from the small scale miners of Gumaus and villages of neighboring town Jose Panganiban and it operates daily and release wastewater on a continuous basis. This facility release high turbidity wastewater with high concentration of Hg and other heavy metals and TSS that exceeds the Philippine effluent limits [17].

Fig. 1. *Tailings collection tank of ball mill facility where wastewater samples gathered.*

The physico-chemical characteristics - temperature, pH, ORP, EC, turbidity, DO and TDS - were measured on site using Horiba Multi Water Quality Checker U-5000G (Japan). Wastewater samples were taken from the crushed ores collecting tank inside the ball mill facility and placed in polyethylene containers and brought to the laboratory for TSS analysis using U.S. EPA Method 160.2 [18]. Separate water samples were placed in 1,000-mL bottles and placed in a container with ice for sample preservation during transport from the site to laboratory for heavy metal analyses. Heavy metal analyses carried out by using Atomic Absorption Spectrophotometer (AAS) and followed the appropriate methods suggested by APHA-AWWA Standard Method for the Examination of Water and Wastewater [19]. Hydride generation AAS was used for As, flame AAS for Pb, and cold vapor AAS for Hg. The limit of detection for the method used in analyzing the metals are 0.001 mg/L for As, 0.01 mg/L for Pb and 0.0001 mg/L for Hg.

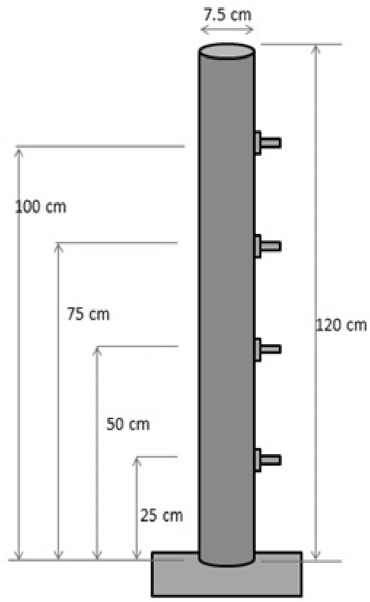

Fig. 2. *Settling column used in the experiment.*

2.2 Plain Settling

Sedimentation Class II, which is characterized as the sedimentation of suspensions that flocculate while settling [20], was adopted in this study. The settling column was made of a glass cylinder tubing with a 7.5 cm inside diameter and 120 cm high. The column diameter was estimated to be >300 times of the largest particle recorded in the sieve and hydrometer analysis of the suspended particles to minimize wall effect [14]. Sampling ports are uniformly spaced along the length of the column at a height of 25, 50, 75 and 100 cm from the bottom of the column and has a freeboard of 20 cm (Figure 2). The schematic diagram of the settling experiment followed in this study is presented in Figure 3. The bulk homogenous actual SSGM wastewater samples were analyzed for TSS using method suggested in U.S. EPA Method 160.2 [18] to determine its initial concentration. Water samples from the bulk were poured at the top of the column until the entire column was full. After 20 min, water samples (200 mL) were collected at every port of the column and analyzed for TSS concentrations. Results from this analysis denote the TSS concentrations at every port for the first settling time, 20 min. After the 20-min settling experiment, water retained in the column were removed and returned to the bulk wastewater. The same procedure of collecting water sample in every port was followed in the settling experiment for longer times of 40, 60, 80, 100, 120, 140, 180, 240, 300, 360, 420, 480, 600 and 720 min after pouring of wastewater at the top of the settling column. The collected samples throughout the experiment were analyzed to get the changes of TSS concentration against respective time and height of the column. The change in TSS concentration with respect to time data were used to produce iso-removal curves.

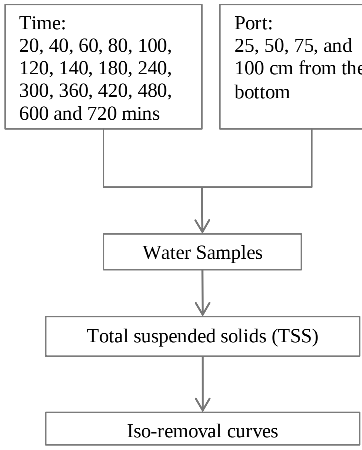

Fig. 3. *Schematic diagram of settling experiment.*

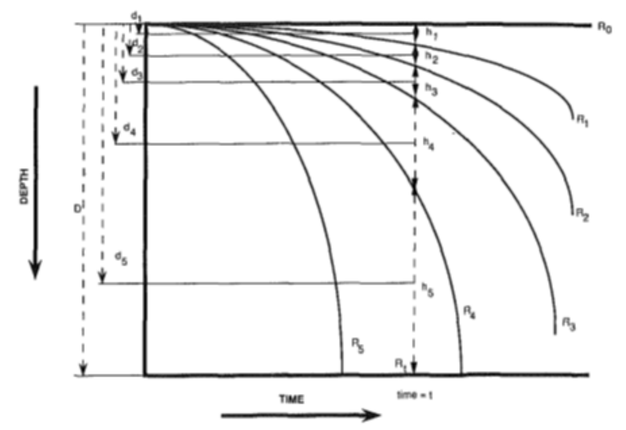

Fig. 4. *Results of Typical Settling-Column Test for Flocculent Suspension [20].*

The 200-mL water samples collected from each sampling port in every time indicated in the procedure were analyzed for TSS using method suggested in U.S. EPA Method 160.2. Well-mixed collected water samples are filtered through a standard Whatman type GF/F glass fiber filter and the residue retained on the filter was dried to constant weight at 103-105°C. Total suspended solids were calculated as follows:

$$TSS, mg/L = (A-B)\times\frac{1000}{C} \quad (1)$$

where A is the weight of filter and dish+residue in mg, B is the weight of filter and dish in mg and C is the volume of sample filtered.

2.3 Iso-removal Curves

The TSS concentrations calculated were used to compute the percentage suspended solids removed at each depth and for each time, using the following expression:

$$R_{ij} = \left(\frac{C_o}{C_{ij}} - 1\right)\times 100 \quad (2)$$

where R_{ij} is the percent TSS removed at the i_{th} depth and at the jth time interval, C_o is the initial TSS concentration and C_{ij} is the TSS concentration at the i_{th} depth at the j_{th} time interval. The percentage removals obtained from the test data were plotted at the appropriate depths and times, a plot of equal concentration are prepared to illustrate the settling path for different removal percentage [16]. In this study, calculating the overall removal efficiency followed the method suggested by Srivastava [20]. From a hypothetical test results and formed a set of curves for constant percentage removal, shown in Figure 4, the overall removal R at time t was:

$$\%\,Removal_t = \frac{h_1}{D}\left(\frac{R_0+R_1}{2}\right)+\frac{h_2}{D}\left(\frac{R_1+R_2}{2}\right) + \frac{h_3}{D}\left(\frac{R_2+R_3}{2}\right)+\frac{h_4}{D}\left(\frac{R_3+R_4}{2}\right)+\frac{h_5}{D}\left(\frac{R_4+R_t}{2}\right) \quad (3)$$

where D is the distance between the lowest sampling port and the water surface in the column settling test; R_i are the curves for constant percentage removal; R_t is the constant percentage removal curve passing through point (t,D); and h_i are the distances as shown in Figure 4.

2.4 Heavy Metal Removal Due to Sedimentation

During sedimentation experiment, water samples were collected from a single port (100 cm from the bottom) to analyze the heavy metal concentrations and determine its reduction over time. Each time (0, 30, 60, 120, 180, 360, 480 min), 500 mL of water were collected from the port and brought to the laboratory for heavy metal analyses with the same procedure used in the characterization. At the same port and time interval, separate water samples (200 mL) were collected and analyzed for TSS. To determine the effect of the settling particles to the reduction of heavy metals in the wastewater during sedimentation, correlation studies were performed between reduction of TSS and reduction of heavy metals. Initial heavy metal concentrations of the wastewater used specifically for the sedimentation tests are 0.23 mg/L for As, 0.0906 mg/L for Hg and 1.27 mg/L for Pb.

2.5 Characterization of Sediment

Settled sediments collected after the sedimentation tests were analyzed its physico-chemical properties and heavy metal concentrations following the procedures suggested by Qiu and Sego [21]. Other parameters were also performed to test the specific gravity, particle size distribution, pH, and soil classification according to the Unified Soil Classification System (USCS). Sediment particle size distribution analysis followed the ASTM D422-63 using the combination of sieve and hydrometer analyses [22]. Sediments were air-dried, oven dried and pulverized as required in the procedure. Sediment particles passed on the sieve No. 200 analyzed using ASTM 152H type hydrometer with sodium hexametaphosphate ((NaPO3)6) as the deflocculating agent. Particles retained on the No. 200 sieve undergone sieve analysis employing the procedure in ASTM D422-63. Same sediment samples were subjected to X-ray diffraction analysis to determine its mineral composition while heavy metal concentrations were analyzed using acid digestion (As and Pb) and following US EPA Method 245.5 for Hg then measured using hydride generation AAS for As, flame AAS for Pb, and cold vapor AAS for Hg.

3 RESULTS AND DISCUSSIONS

3.1 Physico-chemical Characteristics of Wastewater

The physico-chemical characteristics and heavy metal concentrations of raw SSGM wastewater are presented in Table 1. Out of the six physico-chemical parameters analyzed in this study, only two are regulated by DENR [23] for Class C waters. The average pH (6.22) is within the range while measured average TSS is around 9 times higher than the regulated 0.10 g/L limit for Class C waters. The raw wastewater samples used in the experiment was highly turbid. Though not regulated, turbidity suggests that the water contains a lot of suspended particles that affected its clarity. Oxidation-reduction potential (ORP) of the wastewater used in the experiment is 296 mV, this shows that the entire wastewater has high oxidizing agent such as oxygen. Electrical conductivity (EC) is the capability of water to carry electric current and is related to the dissolved minerals present in the wastewater, has measured value of 0.41 mS/cm. This value is lower compared to the EC values ranging from 0.6 to 10 mS/cm measured from different types of mining wastewater [24], [25]. Total dissolved solids (TDS) is related to EC since these are the sum of all ionized solutes and its direct relationship in wastewater samples has been established in previous correlation studies and rough estimation. The measured TDS value is 0.27 g/L which is also lower than the typical gold mine effluent TDS concentration of 2.9 g/L [25]. Heavy metals, As, Hg and Pb concentrations were compared to regulated effluent limits for Class C waters [23]. The concentration of As (0.23 mg/L) is higher than the effluent limit of 0.04 mg/L. Pb concentration of the water samples used has a value of 1.27 mg/L, which is higher than the effluent limit of 0.10 mg/L. Arsenic can be traced from its association (Au-As) with gold deposit, where a quasi-steady As-bearing pyrite extracts solid solution Au from hydrothermal fluids through absorption [26]. High Pb concentration of the samples can be attributed to the high Pb concentration of soil and rocks mined in Paracale mining district [27]. The measured concentration of Hg (0.0906 mg/L) in the wastewater samples was above the effluent limit of 0.004 mg/L. High concentration of Hg was directly associated from the use of Hg in amalgamation process that are widespread practice in the study area [3], [17].

3.2 Iso-removal Curves

The results of the TSS measured at each port and time intervals are presented in Table 2. From the initial TSS concentration of 0.87 g/L, the water collected from different ports were reduced to around 50% in the first 30 min, around 80% in 180 min at all ports and it was completely removed after 600 min. From the TSS concentrations data, the percentage removals were obtained and plotted in a contour map with its respective depths and times. Iso-removal curves were produced by interpolating the plotted values in the contour map (Figure 5). The curves thus produced to represent the maximum settling path for the indicated removal efficiency percentage.

From the graphs of removal curves shown in Figure 5, the TSS percentage removal vs detention time graph (Figure 6) was created by calculating the TSS removal efficiency at times 60, 120, 180, 240, 300, 420 and 600 min. This curve is useful in the design and estimate of sedimentation tank by setting the TSS removal efficiency to determine the detention time of wastewater in the tank. The TSS removal percentage vs overflow rate graph (Figure 7) was also produced from the curves of Figure 3 by calculating the corresponding overflow rate at 50, 60, 70, 80, 90 and 100 % TSS removal using (4);

$$V_o = \frac{H}{t_i} \quad (4)$$

where V_o is the overflow rate, H is the height of the column, t_i is the time defined by intersection iso-removal curve and x-axis. This curve is important in estimating the dimension and surface area of the sedimentation tank to be designed in the ball mill facilities in SSGM area.

TABLE 1: *PHYSICO-CHEMICAL CHARACTERISTICS OF SSGM WASTEWATER USED IN THE EXPERIMENT*

Parameter	Unit	Value	DAO 2016-08
pH	pH	6.22	6.0-9.5
Oxidation-Reduction Potential (ORP)	mV	296	--
Electrical Conductivity (EC)	mS/cm	0.41	--
Turbidity	NTU	725	--
Total Dissolved Solids (TDS)	g/L	0.27	--
Total Suspended Solids (TSS)	g/L	0.87	0.10
Arsenic (As)	mg/L	0.23	0.04
Mercury (Hg)	mg/L	0.0906	0.004
Lead (Pb)	mg/L	1.27	0.10

TABLE 2: *TOTAL SUSPENDED SOLIDS (TSS) CONCENTRATIONS (G/L) AFTER SEDIMENTATION*

Depth(cm)	Time (min)														
	20	40	60	80	100	120	140	180	240	300	360	420	480	600	720
100	0.483	0.337	0.235	0.235	0.143	0.138	0.125	0.095	0.072	0.060	0.043	0.010	0.000	0.000	0.000
75	0.448	0.341	0.242	0.242	0.164	0.131	0.130	0.099	0.072	0.064	0.054	0.015	0.000	0.000	0.000
50	0.428	0.344	0.278	0.278	0.177	0.147	0.135	0.100	0.098	0.070	0.065	0.011	0.009	0.000	0.000
25	0.394	0.383	0.333	0.333	0.182	0.144	0.142	0.104	0.100	0.072	0.070	0.025	0.017	0.000	0.000

In applying iso-removal curves to sedimentation tank design, scale-up factors of 1.75 for detention time and 0.65 for overflow rate are used to compensate for the side wall effects of the batch settling column [28]. Based on the given volumetric flow, Q, and overflow rate, V_o, the area, A of the sedimentation tank can be computed by;

$$A = \frac{Q}{V_0} \qquad (5)$$

The maximum volume, V_{max}, of the sedimentation tank is calculated using (6).

$$V_{max} = Qt \qquad (6)$$

where Q is the volumetric flow, t is the detention time.

3.3 Reduction of Heavy Metal Concentrations

The retention percentages of heavy metal and TSS concentrations during sedimentation are shown in Figure 8. The values were come from the water samples collected from the port 100 cm from the bottom of the settling column. After 60 min of sedimentation in the column, there was a reduction of 97.83%, 95.14% and 93.70% of As, Hg and Pb, respectively. By increasing the period, no further reduction was observed on all heavy metals analyzed. Total suspended solids was reduced by 72.99% after 60 min, 84.14% in 120 min and completely removed after 480 min. This shows that the heavy metal ions settle to the bottom of the column together with the suspended solids. Although other factors such as competition among metals, presence of organic matter content and other components of settling particles were not considered, the results from this study is found to be useful in the design of sedimentation tank for the ball mill facilities. In the correlation study, the reductions of heavy metals and TSS concentrations are highly correlated (Figure 9) with R^2 values of 0.86 (TSS and As), 0.91 (TSS and Hg) and 0.94 (TSS and Pb). The results further show that the reductions on TSS and heavy metals happened in the first 180 min of sedimentation which suggest that settling of TSS and heavy metals happening in the introduction of influent if applied to actual sedimentation tank. The results further indicate that the reduction of heavy metals during sedimentation process is attributed to the attachment of metal ions to the dominant fine particle size (<74 µm) of the sediments which promotes high adsorption capacity due to its large surface area. The results are in agreement with the report of Sprenger et al. [9] wherein the suspended solids (<63 µm) identified as the carrier of 66 – 88% of heavy metal concentrations (Pb, Cd, Zn, Cu, Ni) from the wastewater samples.

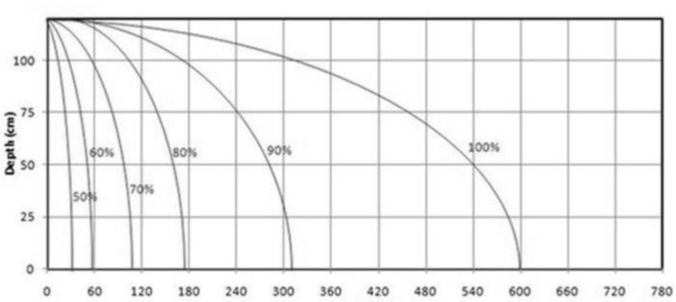

Fig. 5. *Iso-removal curves obtained from sedimentation analysis.*

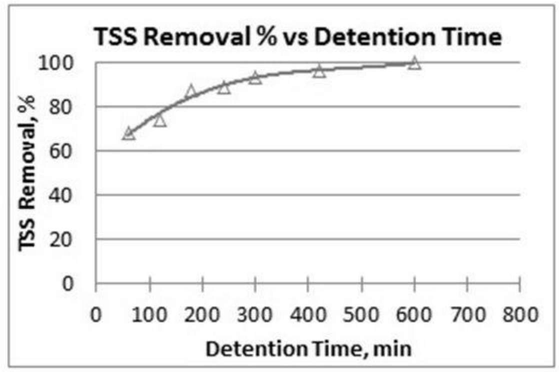

Fig. 6. *TSS Removal % vs detention time curve.*

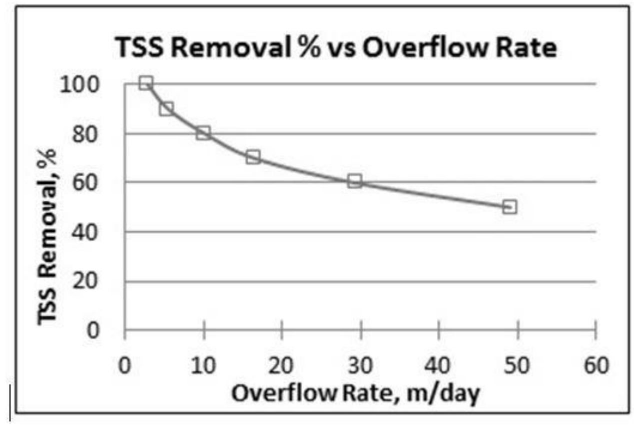

Fig. 7. *TSS Removal % vs overflow rate curve.*

Results from this study implies that the sediment with heavy metals (e.g. Hg) if disposed of untreated, Hg will undergo methylation and transform to methylmercury (MeHg), which when leached out to water bodies may be transferred and concentrated in fish and enter the food chain, where it then poses health risk to the people living in the area.

3.4 Sediment Characteristics

3.4.1 Particle Size Distribution of Sediments

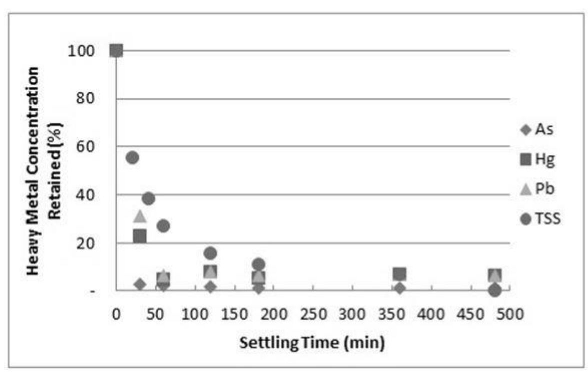

Fig. 8. *Heavy metal and TSS concentrations change measured at port 100 cm from the bottom during sedimentation.*

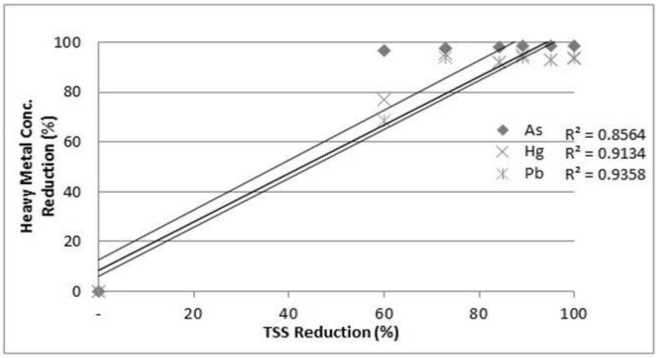

Fig. 9. *Correlation of heavy metal and TSS concentrations reduction during sedimentation.*

The particle size distribution of the combined results of sieve and hydrometer analyses of the settled suspended solids are shown in Figure 10. The sediment of the SSGM tailings is composed of around 22% clay size particles and sand content of around 49%. Fines content (<74μm) is dominant at around 54% of the entire soil particles tested. These fine sizes of suspended particles were the results of ball milling of ores during amalgamation process inside the facility. Based on the Unified Soil Classification System (USCS), the tailings from the SSGM ball mill facility is ML or silty soil which is the same characteristics with the gold mine tailings properties studied by Qiu and Sego [21] presented in Table 3.

TABLE 3: *PHYSICAL PROPERTIES OF DIFFERENT MINE AND SSGM TAILINGS*

Parameter	Tailings (Study of Qiu and Sego [21])				SSGM (This study)
	Copper	Gold	Coal	Consolidated	
Specific Gravity	2.75	3.17	1.94	2.60	2.61
pH value in process water	[illegible]	[illegible]	7.2	7.7	6.88
Liquid Limit (%)	--	--	40	--	--
Plasticity Index (%)	--	--	16	--	--
Shrinkage Limits	24.4	21.6	21.1	25.2	--
Clay size particles (< 2μm; %)	1.3	5.3	22.5	8.9	22
Sand content (> 0.06 mm; %)	74.5	33.3	40	77	49
Fines content (< 74μm; %)	31.3	81.3	66.4	21.2	54
D_{10} (μm)	16.28	5.0	1.31	2.7	0.9
D_{30} (μm)	72.25	19.0	4.13	11.2	4.0
D_{50} (μm)	120.6	44.8	29.2	182	20.0
D_{60} (μm)	153.5	54.0	60.0	204	150.0
USCS classification	SM	ML	CL	SM	ML

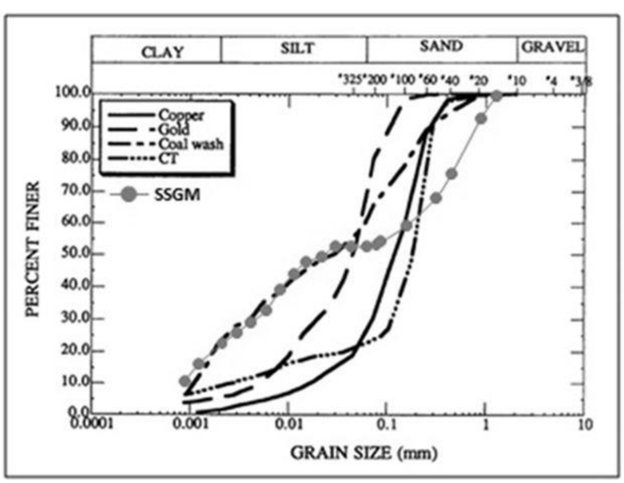

Fig. 10. *Particle size distribution of sediment in this study compared to the other tailings studied by Qiu and Sego [21].*

3.4.2 Physico-chemical Properties

The SSGM ball mill facility use tap water in the milling of ores. Tap water has almost neutral pH value (6.88) compared to the basic pH of other tailings in the list in Table 3. The specific gravity (2.61 g/cm^3) of the sediment is within the range of the various values in the study and it is close to the quartz density (2.65 g/cm^3) [29] which is the major mineralogical constituent of the sediment.

3.4.3 Heavy Metals and Mineral Contents

Concentrations of heavy metals present in the sediment collected after the settling column experiments are 0.005 mg/kg, 0.64 mg/kg, and 0.0126 mg/kg for As, Pb, and Hg, respectively. Hg concentration in sediment was directly related to the use of Hg in amalgamation in SSGM area of Paracale, Camarines Norte, while other heavy metals (As and Pb) were traced from the composition mined ores in the area. The diffractogram scan for the sediment of the SSGM is shown in Figure 11. This diffractogram clearly shows that the tailings are predominantly composed by quartz (SiO_2) with small amount of illite ($(K,H_3O)(Al,Mg,Fe)_2(Si,Al)_4O_{10}[(OH)_2,(H_2O)]$, sphalerite ($(Zn,Fe)S$), and halloysite ($Al_2Si_2O_5(OH)_4$) which is in agreement with the site geology studied by Giese, et.al [30].

4 CONCLUSIONS

Small scale gold mining wastewater from Paracale, Camarines Norte is characterized with high concentrations of heavy metals (As, Hg, Pb) that exceed the Philippines effluent limit. Mercury in the wastewater confirms that the miners use Hg in the amalgamation process in their gold extraction. Sedimentation tests using settling column found to be appropriate method in investigating the removal of suspended solids and heavy metals from SSGM wastewater. Sedimentation experiment showed that after 600 min, 100% removal of TSS was achieved from the 120 cm column height. Heavy metal removal efficiency on the wastewater were tested at 100 cm from the bottom and results showed that after 480

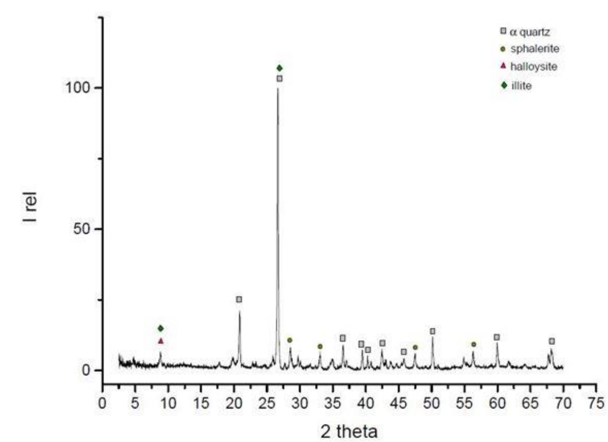

Fig. 11. *X-ray diffractogram for the sediment.*

min, removal efficiencies of 98.69%, 93.60% and 93.70% for As, Hg and Pb, respectively were achieved and by increasing the period, there were no more increase in the efficiency. In the correlation study shows that the reductions of heavy metals and TSS concentrations are highly correlated which implies that the heavy metals from the wastewater were adsorbed to the solid particles which settled in the bottom of the column. Concentrations of heavy metals present in the sediment collected after the settling column experiments were 0.005 mg/kg, 0.64 mg/kg, and 0.0126 mg/kg for As, Pb, and Hg, respectively. The dominant particle size of the sediment was 54% fine contents (<74 µm) and was characterized as silica according to USCS which has a potential for its reuse if heavy metal recovery will be done. The result of this study can be a basis for the design and estimate of sedimentation tank in SSGM ball mill facilities.

5 ACKNOWLEDGMENTS

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