Global J. Environ. Sci. Manage. 5(4): 461-470, Autumn 2019

Global Journal of Environmental Science and Management
(GJESM)

Homepage: https://www.gjesm.net/

ORIGINAL RESEARCH PAPER

Treatment of small scale gold mining wastewater using pilot-scale sedimentation and Cocopeat filter bed system

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ARTICLE INFO

Article History:
Received 09 May 2019
Revised 29 July 2019
Accepted 30 August 2019

Keywords:
Adsorption
Cocopeat
Heavy metals
Mining wastewater
Sedimentation

ABSTRACT

The use of amalgamation process to recover gold from mined ores by the small-scale gold miners in the Philippines and other developing countries produces and dispose of untreated wastewater to the receiving water bodies. In this study, a field-scale filter bed system was constructed to treat heavy metal metal-laden wastewater collected from small-scale gold mining site in Paracale, Camarines Norte, Philippines. The filter bed system was consists of sedimentation tank and filter bed with Cocopeat, a by-product of coconut husk, as adsorbent. Physico-chemical parameters (temperature, pH, oxidation-reduction potential, electrical conductivity, turbidity, dissolved oxygen, total dissolved solids, salinity, total suspended solids, color) and heavy metal (As, Ba, Cd, Hg, Pb) concentrations were monitored during the 50 days experiment at a flow rate of 40 Liter per hour for 3 hours daily wastewater application. Significant reduction was achieved on heavy metals; As (97.11%), Ba (39.75%), Cd (74.24%), Hg (97.02%), Pb (98.82%) from small-scale gold mining (SSGM) wastewater in sedimentation phase and further reductions on As (1.39%), Ba (28.00%), Cd (4.95%), Hg (2.91%), Pb (0.97%) were achieved by adsorption in the Cocopeat filter bed. Measured effluent physico-chemical parameters and heavy metal concentrations were within the respective regulatory limits. Other effluent parameters with strong correlation with total suspended solids such as turbidity and color, though not regulated, were reduced significantly. All adsorbed heavy metals accumulated in the upper 25 cm of the Cocopeat column in the filter bed. Measured heavy metal concentrations in Cocopeat suggest that the adsorbent was not saturated and further application of small-scale gold mining wastewater is recommended to determine its useful life.

DOI: 10.22034/gjesm.2019.04.06

NUMBER OF REFERENCES	NUMBER OF FIGURES	NUMBER OF TABLES
26	3	4

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Note: Discussion period for this manuscript open until January 1, 2020 on GJESM website at the "Show Article.

INTRODUCTION

Artisanal and small scale gold miners in the Philippines and other developing countries use amalgamation process to recover gold from mined ores with the use of mercury (Hg) (Opiso *et al.*, 2018; Esdaile and Chalker, 2018; Israel and Asirot, 2002). This method is commonly used in gold-rush areas by small scale miners since it is simple to apply and requires relatively low investment. However, the use of amalgamation process produces wastewater with elevated amount of Hg and other heavy metals and with high concentration of suspended solids (SS) that are being released to the water bodies untreated (Soriano and Soriano, 2017; Samaniego and Tanchuling, 2018b). This practice becomes the basis of opposition to amalgamation method because it affects the environment and human health. Mercury, as one of the dominant heavy metals in small scale gold mine tailings, is one of the most toxic heavy metals released in the environment. This metal if present in the water bodies may be eaten by fish and in turn may be consumed by the people. Its major effects to human include neurological and renal disturbances and impairment of pulmonary function (Jaishankar *et al.*, 2014). It is necessary, therefore, to prevent heavy metals from reaching the natural environment. The commonly used procedures to remove heavy metals from mine tailings and other wastewater are reverse osmosis, electrodialysis, ultrafiltration, ion-exchange, chemical precipitation and phytoremediation. But these procedures were found to have disadvantages like incomplete metal removal, high reagent and energy requirements, generation of toxic sludge and other waste sludge that require careful disposal which makes it not cost-effective (Kulbir *et al.*, 2018). Among the known and recommended treatment technologies in removing heavy metals from the wastewater, adsorption is one of the most promising techniques because it is economical and it promotes recycling and reuse of the sorbed metals (Montes-Atenas and Valenzuela, 2017). Adsorption is also the most frequently studied and widely applied for the treatment of the heavy metal-contaminated wastewater (Zhao *et al.*, 2016). Several raw materials, agricultural refuses, and inorganic materials such as soils have been studied as adsorbent in the past decades. Such raw materials can be further processed to produce carbon-based adsorbent which found to have large surface area compared to the raw materials and it is the most recognized effective absorbent available in the market (Renu *et al.*, 2016). Special type of soils, such as volcanic ash and clays has been thoroughly investigated by several researchers to determine its potential in adsorbing heavy metals from wastewater. Cocopeat (coir pith or coir dust), soft biomass separated from coconut husks during extraction of coir fiber, is found to be effective adsorbent to remove heavy metals from mining wastewater in batch and column experiments as well as in field scale filter bed system (Tanchuling *et al.*, 2012). Coco coir dust consists mainly of lignin, cellulose, hemicellulose, and some pectin and extractives (mainly fat, fatty acids, fatty alcohols, phenols, terpenes, steroids, resin acids, rosin and waxes). Metal ions sorb mainly to carboxylic (primarily present in pectin and hemicellulose but also extractives and lignin), phenolic (lignin and extractives) and to some extent hydroxylic (cellulose, hemicellulose, lignin, extractives, and pectin) and carbonyl groups (lignin) (Panamgama and Peramune, 2018; Conrad and Bruun Hansen, 2007; Guo *et al.*, 2008). Previous studies reported that sorbents, including coir, with high lignin content adsorb high metal ions (Lee and Rowell, 2004). Several studies demonstrated the efficacy of coir pith in sorption of heavy metals such as zinc (Zn), lead (Pb), cadmium (Cd), cobalt (Co), nickel (Ni) and Hg in laboratory batch and column experiments using aqueous solutions (Conrad and Bruun Hansen, 2007; Gupta *et al.*, 2015). In the Philippines, Cocopeat is abundant since the country is considered as one of the biggest producers of coco fiber in the world as its utilization are mainly for soil conditioner, organic fertilizer and materials for soil erosion control. In other countries, Cocopeat is used as an adsorbent to treat industrial wastewater containing dyes (Rahman, 2016) and a peat substitute in growing containerized ornamental plants (Carlile *et al.*, 2015). Despite these known uses of coco peat, it is still considered as underutilized agricultural wastes. To add its value, researchers become interested to use it as adsorbent for heavy metal removal from aqueous solutions. In this study, a pilot-scale sedimentation and Cocopeat filter bed system, constructed in 2015 at University of the Philippines – Diliman, Quezon City, was used to treat small-scale gold mining (SSGM) wastewater. The present study aimed to evaluate the potential of using the sedimentation and Cocopeat filter bed system in removing heavy metals suspended solids from the wastewater of small-scale gold mining processing plant. This study has been carried out in the Department of Environmental Engineering compound of the University of the Philippines in Diliman, Quezon City throughout the period of August 2015 to January 2016.

MATERIALS AND METHODS

Sedimentation and filter bed system

The pilot-scale sedimentation and filter bed system was constructed in 2015 at the Environmental Engineering compound at the University of the Philippines-Diliman in Quezon City. The filter bed system is a combination of sedimentation tank and filter bed reactor with gravel, sand and Cocopeat substrates (Fig. 1). Sedimentation tank acted as the primary treatment of the system where SS can be reduced by settling and heavy metals adsorbed into the Cocopeat in the filter bed that contributed in the reduction of heavy metals from the wastewater. Gravel, with size range of 16 – 32 mm, was washed with distilled water to removed impurities and contaminants. It was placed above the Cocopeat and in the zone of distribution pipes to act as the energy dissipator and make the flow evenly distributed in the filter bed area with the aid of perforated pipes. Sand was screened to select the size range of 0.6 – 2 mm and likewise washed with distilled water to removed impurities and contaminants. It was placed under the Cocopeat layer to prevent its particles from moving downward. Another layer of gravel placed underneath the sand and in the zone of drainage pipes to give enough pore spaces for the water entering the perforated pipes that are connected to the drainage pipe. The distribution and drainage pipes as well as the control valves and connectors are made of polyvinyl chloride (PVC). Table 1 presents the selected physical characteristics of sedimentation tank and filter bed system design used in the experiment.

Water sampling and analysis

A total of 6,000 L of wastewater sample was gathered from the outlet of tailings tank of an

Table 1: Selected physical characteristics of sedimentation tank and filter bed system design

Characteristics	Values
I. Sedimentation Tank	
Flow rate, L/h	40
Detention time, h	6.17 (17.50 maximum)
Overflow rate, m/h	0.16 (0.078 minimum)
II. Filter Bed	
Flow rate, L/h	15 (1,200 maximum)
Surface loading	1.0 m^3/day
III. Substrates (height)	
Gravel (16 – 32 mm)	0.3 m
Cocopeat (0.075 – 2.00 mm)	0.5 m
Sand (0.2 – 2 mm)	0.2 m

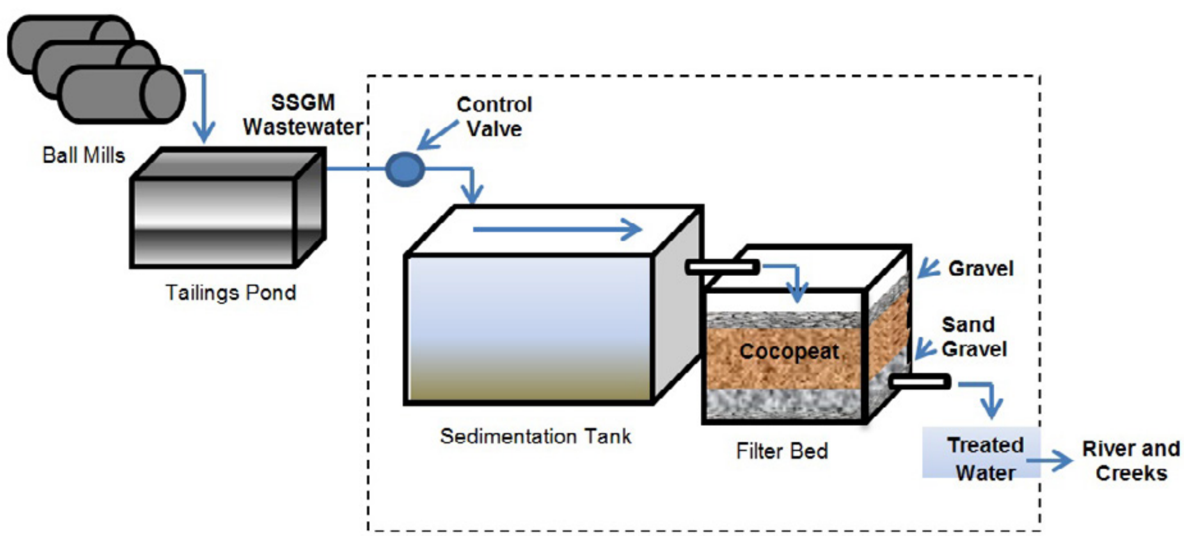

Fig. 1: Schematic of filter bed system if installed in small-scale gold mining processing plant

active ball mill facility in SSGM area in Paracale, Camarines Norte, Philippines, 320 km south of Quezon City. These samples were placed in 20-L polypropylene containers and were transported to Quezon City where the filter bed system was installed. One hundred twenty (120) liters of SSGM wastewater were poured to reservoir tank daily for 50 days of experiment. The reservoir tank has control valve to check the desired flow of water onto the sedimentation tank. Sampling and testing of the wastewater were also done in the reservoir for the testing of physico-chemical parameters and heavy metal concentrations of raw wastewater. Raw wastewater, marked as "RAW" flows into the sedimentation tank at a flow rate of 40 L/hr. Water that overflows from sedimentation tank become the influent "INF" for Cocopeat filter bed, while the effluent "EFF", was the treated water from the filter bed. Water samples at each collection stages were collected daily within the 50 days operation. Physico-chemical parameters; temperature, pH, oxidation-reduction potential (ORP), electrical conductivity (EC), turbidity, dissolved oxygen (DO) and total dissolved solids (TDS) were measured on site using Horiba Multi Water Quality Checker U5000G (Japan). Total suspended solids (TSS) were measured using gravimetric method and dried at 105°C. Apparent color of water was measured employing colorimetric platinum cobalt method using Hanna Color of Water meter (USA). Separate water samples were placed in 500-mL bottles and placed in a container with ice for sample preservation for heavy metal analyses using Atomic Absorption Spectrophotometer (AAS) following appropriate method suggested by APHA (1998). Hydride generation AAS was used for As, flame AAS for Ba, Cd and 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, 0.2 mg/L for Ba, 0.003 mg/L for Cd, 0.01 mg/L for Pb, and 0.0001 mg/L for Hg. Results of the measured physico-chemical parameters and heavy metal concentrations were compared to the government effluent limits for Class C waters (DENR, 2016).

Cocopeat Sampling and Analysis

Cocopeat was sourced from the stockpiles of coconut husk decorticating plant in Sariaya, Quezon Province. The same Cocopeat samples were used as adsorbent in the previous studies on batch and column tests that removed heavy metals from aqueous solution conducted in University of the Philippines-Diliman (Tanchuling *et al.*, 2012). Cocopeat were sorted using Test Sieve No. 10 (W.S. Tyler, USA) to get particle size of ≤ 2 mm that were used in the experiment. The sieved Cocopeat maintained its natural condition before placing to the filter bed without washing or any chemical pre-treatment. Initial concentrations of all heavy metals, except form Hg (0.208 mg/kg), were not detected using AAS. The presence of Hg in the Cocopeat samples used in the experiment can be traced from the environmental condition of stockpiles situated in an open area that is prone to sorbed heavy metals including organic and inorganic Hg from different environmental media such as water, soil, vegetation and others. A total of 50.50 kg of Cocopeat was stacked in the filter bed with the dimension of 100 x 100 x 50 cm (length x width x height). After 50 days of experimental runs, Cocopeat samples from two (2) layers, top and bottom (25 cm each) along the depth of Cocopeat were collected and analyzed for heavy metal concentrations to determine the position of adsorbed heavy metals in the Cocopeat column. These samples were air-dried, sieved and ground. Heavy metal concentrations were determined by AAS for As, Ba, Cd and Pb following acid digestion with 65% nitric acid, while U.S. EPA Method 245.5 for Hg. To identify the magnitude of differences in metal accumulation between the top and bottom layers of the Cocopeat adsorbent, a top/bottom ratio was determined using Eq. 1.

$$top \,/\, bottom = \frac{C_{top}}{C_{bottom}} \qquad (1)$$

Where, C_{top} was the metal concentration in Cocopeat sampled from the top 25 cm and C_{bottom} was the metal concentration in Cocopeat sampled from the bottom 25 cm (mg/kg).

Statistical analysis

The mean values of physico-chemical parameters and heavy metal concentrations of raw SSGM wastewater and effluent were obtained from 50 samples. Two-sample z-test was used to test for the significant differences in the mean values of raw SSGM wastewater and effluent after the sedimentation and adsorption in filter bed experiments. The results were investigated by using confidence level of 99.9%.

RESULTS AND DISCUSSION

The following results apply to the performance of the sedimentation and Cocopeat filter bed system during the treatment of raw SSGM wastewater. A total of 6,000 L of SSGM wastewater (120 L daily) for 50 days with a flow rate of 40 L/h were considered in the experiment.

Changes in physico-chemical properties

The physico-chemical characteristics and heavy metal concentrations of the SSGM wastewater collected from the ball mill facility and its effluent concentrations are presented in Table 2. Comparing these values to the effluent regulatory limits set by DENR (2016) for Class C waters, pH values are within the limit, while TSS was above the limit. Turbidity, which is one of the parameters that are not being regulated, has a very high value of >800 PCU. Based on these values of the physico-chemical parameters of wastewater samples gathered from the ball mill facility, the effluent are not acceptable for discharge to the rivers or creeks in the area. Temperature of water during sedimentation slightly increased due to the exposure to ambient temperature during the daytime measurement and it became cooler after it percolated through the Cocopeat filter bed. The level of pH remains near neutral during the sedimentation, but in adsorption phase, the pH became alkaline as demonstrated in the pH development over time in adsorption batch tests using Cocopeat and SSGM wastewater (Samaniego and Tanchuling, 2019). Electrical conductivity, TDS and salinity have the same trend in the concentration changes during sedimentation and Cocopeat adsorption. The increase in concentration of these parameters was attributed to desorption and release of native content of Cocopeat such as sodium and potassium (Conrad and Bruun Hansen, 2007). Dissolved oxygen of

Table 2: Mean of physico-chemical parameters and heavy metal concentrations of the SSGM wastewater used in the experiment (n=50)

Parameters	Unit	Raw SSGM Mean (SD)	Effluent Mean (SD)	DAO 2016-08 Limits
Temperature	°C	26.73 (1.59)	25.50 (1.39)*	25-31
pH	pH	6.88 (0.42)	7.24 (0.60)*	6.0-9.5
ORP	mV	340.24 (41.52)	311.30 (37.83)*	-
EC	mS/cm	0.13 (0.04)	0.97 (0.98)*	-
Turbidity	NTU	>800[a]	82.9 (82.64)	-
DO	mg/L	5.69 (0.75)	4.18 (1.26)*	-
TDS	g/L	0.09 (0.02)	0.62 (0.62)*	-
Salinity	ppt	0.06 (0.05)	0.49 (0.52)*	-
TSS	g/L	3.82 (3.14)	0.025 (0.028)*	0.10
Color (Apparent)	PCU	9,683.20 (1,860.08)	834.40 (672.86)*	-
As	mg/L	0.1550 (0.1512)	0.0023 (0.0016)*	0.04
Ba	mg/L	0.1544 (0.0820)	0.0498 (0.0873)*	6.00
Cd	mg/L	0.0079 (0.0045)	0.0016 (0.0003)*	0.01
Hg	mg/L	0.1434 (0.1535)	0.0001 (0.000005)*	0.004
Pb	mg/L	7.9385 (7.4029)	0.0163 (0.0141)*	0.10

[a] not tested due to non-availability of exact values from the instrument used
*highly significant at $p < 0.001$ from two-tailed z-test

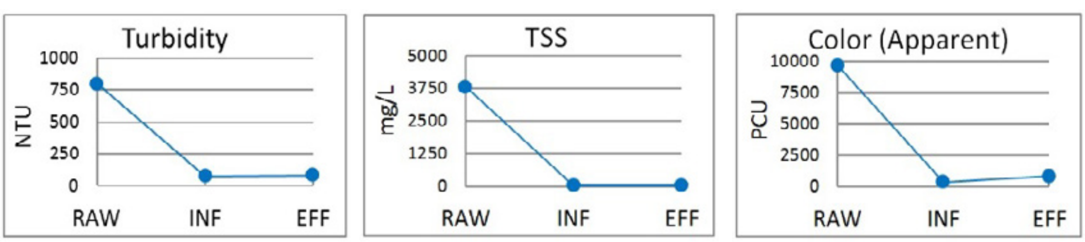

Fig. 2: Changes in mean concentration on turbidity, TSS and apparent color in different stages of treatment

wastewater during sedimentation were reduced due to settling action in a still water without mixing, but as the influent flows through the filter bed, ambient air and air in the pore spaces of filter bed mix and in effect the effluent tend to increase its concentration. Shown in Fig. 2 are the changes in levels of turbidity, TSS and apparent color of wastewater during the experimental runs. In the sedimentation phase, the concentrations of turbidity and TSS were reduced significantly as discussed in the sedimentation tests of the present study (Samaniego and Tanchuling, 2018a). Further slight decrease in the concentrations of turbidity and TSS observed during filter bed adsorption. The average color of raw wastewater was 9,683.20 PCU which was mainly caused by elevated concentration of TSS. After the sedimentation stage, the apparent color dropped to a mean of 370.20 PCU due to the settling of SS. During adsorption, the average color of water measured in the effluent was higher (834.40 PCU) than the INF due to the washing of Cocopeat in the early stage of adsorption. In effect, the colloidal particles found in the Cocopeat materials were washed away and caused discoloration to the effluent. Increase in color can further explained by the solubility of lignin and its derivatives by oxidative delignification which generates highly colored filtrate rich in phenolic compounds (Rojith and Bright Singh, 2012). High color concentrations were measured in the first 23 days and after which the color concentration went down and the last measurement was 280 PCU in the 50th day. Overall, the values of different physico-chemical parameters of the effluent had high significant difference ($p < 0.001$) from the raw SSGM wastewater after undergoing sedimentation and adsorption onto Cocopeat in the filter bed.

The correlation matrix on the physico-chemical properties monitored in the experiment is presented in Table 3. Temperature has a strong positive correlation with ORP (0.97) and has negative correlations with the rest of parameters. Positive correlations were found between pH and EC, DO, TDS, Salinity, while it has significant negative correlation with ORP (-0.99). Oxidation-reduction potential was found to have negative correlation with the rest of the parameters. Electrical conductivity has significant correlation with TDS (0.99) and salinity (0.99) since high dissolved salt content contains negatively charged ions that can conduct electricity. Electrical conductivity and TDS are the water quality parameters that are being used to describe salinity level (Rusydi, 2018). Turbidity has a very significant correlation with DO (0.91), TSS (0.99), and color (0.99) while DO has a strong relationship with TSS (0.90).

Heavy metal removal efficiencies

The mean raw SSGM wastewater and effluent concentrations of heavy metals measured in the experiment was presented in Table 1. After 50 days of operation, all heavy metal concentrations of the effluent are below the DAO 2016-08 limits for Class C waters. The mean removal efficiencies at the sedimentation and Cocopeat adsorption are shown in Fig. 3. These are the mean of 50 samples analyzed daily for heavy metal concentrations from different sampling points (RAW, INF, EFF). After 50 days of operation in sedimentation phase, significant reduction was achieved on heavy metals; As (97.11%), Ba (39.75%), Cd (74.24%), Hg (97.02%), Pb (98.82%) from the SSGM wastewater. Further reductions on As (1.39%), Ba (28.00%), Cd (4.95%), Hg (2.91%), Pb (0.97%) were attained when the wastewater underwent adsorption along Cocopeat column in the filter bed. The overall removal efficiencies for all heavy metals tested after undergoing sedimentation

Table 3: Correlation matrix on physico-chemical properties monitored in the experiment.

Parameter	Temp	pH	ORP	EC	Turb	DO	TDS	Sal	TSS	Color
Temp	1.00									
pH	-0.93	1.00								
ORP	0.97	-0.99	1.00							
EC	-0.44	0.73	-0.63	1.00						
Turb	-0.30	-0.05	-0.08	-0.72	1.00					
DO	-0.68	0.37	-0.49	-0.36	0.91	1.00				
TDS	-0.45	0.74	-0.64	0.99	-0.71	-0.35	1.00			
Sal	-0.48	0.76	-0.66	0.99	-0.69	-0.32	0.99	1.00		
TSS	-0.30	-0.06	-0.07	-0.73	0.99	0.90	-0.72	-0.70	1.00	
Color	-0.34	-0.02	-0.11	-0.70	0.99	0.92	-0.69	-0.66	0.99	1.00

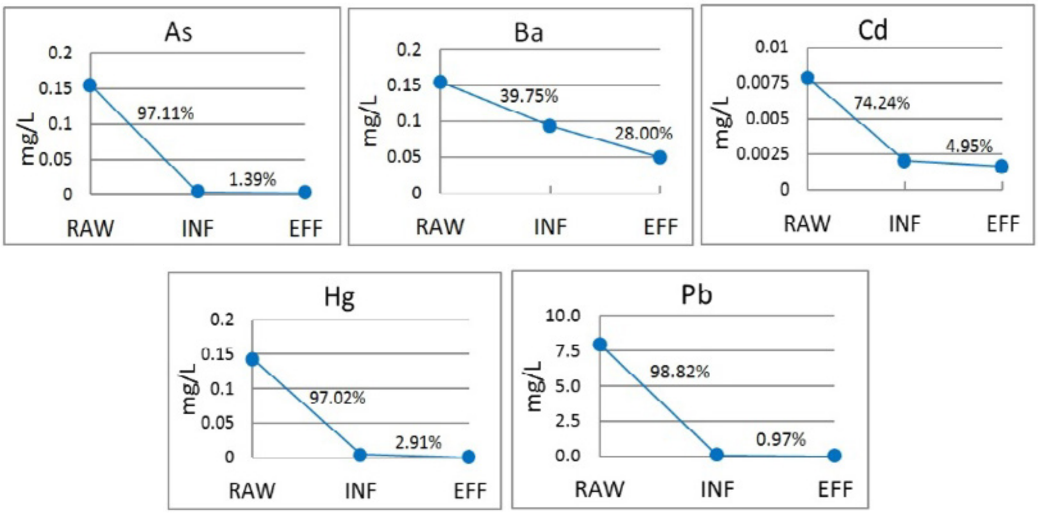

Fig. 3: Heavy metal removal efficiencies by sedimentation and filter bed

Table 4: Ratio of top/bottom heavy metal concentrations in the Cocopeat column after 50 days

Heavy Metals	Top/bottom[a] Mean Concentration (mg/kg)	Ratio
As	0.53 / 0.01	53.00
Ba	52.50 / 19.51	2.69
Cd	0.0274 / 0.0001	274.00
Hg	0.5609 / 0.0785	7.14
Pb	27.94 / 7.37	3.79

[a] Values above 1 indicate that Cocopeat from the top 25 cm of the column have a higher metal concentration than the bottom 25 cm

and Cocopeat adsorption in the filter bed were highly significant ($p < 0.001$). The sequence from high to lower removal efficiency is given as Hg>Pb>As>Cd>Ba. All heavy metal concentrations of the effluent after 50 days are within the government effluent limits.

Location of heavy metals within the column

All heavy metals were mainly adsorbed onto Cocopeat at the top 25 cm of the column (Table 4). The main mechanism for metal enrichment of all heavy metals onto Cocopeat is best represented by chemisorption with its applicability to pseudo-second-order kinetics model (Samaniego and Tanchuling, 2019). These processes are enhanced with the presence of high lignin content (Lee and Rowell, 2004), pectin and hemicellulose in the Cocopeat that adsorb high metals ions (Conrad and Bruun Hansen, 2007). The adsorption in the upper part of the column was further enhanced with the presence of organic matter in Cocopeat that contains polar functional groups that dissociate and take part in metal uptake through surface complexation and exchange of metal cations (Hasany and Ahmad, 2006). After 50 days of experiment, As and Cd exhibited high ratio of top/bottom heavy metal concentrations in the Cocopeat column. However, Ba, Hg and Pb has lower top/bottom ratio due to the significant heavy metal concentrations accumulated in the bottom from the possible leaching occurred during the experiment. High organic matter in Cocopeat caused the possible leaching Hg from the top to bottom of the Cocopeat column. Substantial concentration of Pb in the lower part of the Cocopeat column can be attributed to the high amount of Pb entered the filter bed and the high affinity of Pb to lignin caused the Pb ions go deeper (Guo *et al.,* 2008). Based on this experiment, measured heavy metal concentrations in Cocopeat suggest that the adsorbent was not saturated and further application of wastewater is recommended to determine the useful life of Cocopeat.

CONCLUSION

The combined sedimentation and Cocopeat filter bed system is found to be suitable treatment option in removing heavy metals from the effluents of ball mill facilities in small-scale gold mining area. Concentration of heavy metals and physico-chemical parameters such as TSS, apparent color and turbidity in the effluent during the 50 days operation were significantly reduced by the effect of the combined processes of sedimentation and adsorption in the filter bed. Measured effluent physico-chemical parameters and heavy metal concentrations were within its respective government regulatory limits for Philippine Class C waters. The properly designed sedimentation tank that served as primary treatment showed high removal of heavy metals and TSS while Cocopeat in the filter bed further reduced the heavy metals by adsorption. The measured heavy metal concentrations in Cocopeat column after 50 days has high top/bottom ratio which shows that adsorbed heavy metal ions are accumulated in the upper 25 cm of the Cocopeat column in the filter bed and it shows that the amount of heavy metal ions adsorbed in Cocopeat suggest that the adsorbent is not saturated after 50 days. Further tests need to be conducted with this treatment system by applying more volume of raw wastewater with a constant flow rate to determine its optimal conditions for TSS and heavy metals removal before it can be applied at full scale.

ACKNOWLEDGMENTS

The authors acknowledge the Department of Science and Technology - Philippine Council for Industry, Energy and Emerging Technology Research and Development (DOST-PCIEERD) for the research grant under the Mineral Extraction with Responsibility and Sustainability Program; Project C: Optimizing the Effectively of Coco-peat filter bed in field applications [Project Code No. 02423]. Special credit is given to the Department of Science and Technology- Engineering Research and Development for Technology (DOST-ERDT) for the scholarship grants and additional research fund for the study.

CONFLICT OF INTEREST

The author declares that there is no conflict of interests regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/or falsification, double publication and/or submission, and redundancy has been completely observed by the authors.

ABBREVIATIONS

°C	Degrees Celcius
%	Percentage
AAS	Atomic Absorption Spectrophotometer
APHA	American Public Health Association
As	Arsenic
AWWA	American Water Works Association
Ba	Barium
Cd	Cadmium
Cm	Centimeter
Co	Cobalt
Conc.	Concentration
CV-AAS	Cold Vapor - Atomic Absorption Spectrophotometer
DAO	DENR Administrative Order
DENR	Department of Environment and Natural Resources
DO	Dissolved oxygen
EC	Electrical conductivity
EFF	Effluent
g/L	Gram per liter
h	Hour
Hg	Mercury
INF	Influent
L	Liter
L/h	Liter per hour
m^3*/day*	Cubic meter per day
mg/kg	Milligram per kilogram
mg/L	Milligram per liter
mS/cm	Microsiemens per centimeter
mV	Millivolts
Ni	Nickel
NTU	Nephelometric turbidity unit
ORP	Oxidation-reduction potential
Pb	Lead
PCU	Platinum-cobalt unit
pH	Potential of hydrogen
ppt	Parts per trillion

PVC	Polyvinyl chloride
RAW	Raw SSGM wastewater
Sal	Salinity
SS	Suspended solids
SSGM	Small-scale gold mining
TDS	Total dissolved solids
Temp	Temperature
TSS	Total suspended solids
Turb	Turbidity
U.S. EPA	United States Environment Protection Agency
Zn	Zinc

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HOW TO CITE THIS ARTICLE

Samaniego, J.O.; Tanchuling, M.A.N., (2019). Treatment of small scale gold mining wastewater using pilot-scale sedimentation and Cocopeat filter bed system. Global J. Environ. Sci. Manage., 5(4): 461-470.

DOI: 10.22034/gjesm.2019.04.06

url: https://www.gjesm.net/article_36261.html