Examiner’s commentary

This is an interesting essay that, while clearly focused on chemistry, has an environmentally caring backbone. The research question is correct, it is “chemistry focused” and gives a hint of what comes next, though the words “to what extent” could have been avoided. The background information is relevant and helps framing the whole work within the context of the subject. It also explains the rationale behind the choice of variables which is commendable. It is evident that chemical principles and ideas behind the research question and techniques applied are understood, as they are fluently incorporated into the main body of the essay. Sources are well chosen, and technical vocabulary is consistently used in a sound fashion. Analysis includes mathematical treatment, as well as graphs, something highly appreciated in this context, together with a critical evaluation of the investigation, not only practical aspects. The only criterion with room for improvement here is communication: there is a lot of information regarding safety and materials which in the case of an Extended Essay belong better in the appendix.

IBDP – Chemistry Extended Essay

Leaching potential of heavy metals in coal ash

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To what extent does change in pH (3.0-6.0) of sample of coal affect the leaching potential of toxic heavy metals (chromium, copper, and manganese) in groundwater and soil measured using spectrophotometry to analyze leachate from a lab scale prototype of soil over a period of 4 weeks?

Word count: 3997

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Leaching potential of heavy metals in coal ash

Introduction

Since industrial revolution, petroleum and coal have been major energy sources. However, continuous use has resulted in **perennial pollution**. In India, about 75% energy demand is met by non-renewable sources, including 44% from coal.[1] Hence, global paradigm shift towards renewable or green energy made many researchers contemplate reasons which lead to this course.

Coal-fired power plants produce combustion residue called **coal ash** (commonly known as fly-ash) that is a fine powder containing heavy metals like arsenic, chromium, manganese, mercury, copper, zinc, and vanadium.[2] According to **Central Electricity Authority of India**[3], 63% of this coal ash is used to manufacture concrete or wallboards and remaining 37% is disposed in ash ponds or landfills.[4] Coal ash is composed of many trace level heavy metals and when mixed in water, to dispose in landfills, these heavy metals reach underground water via leaching in soil, making its way to food and water that humans consume. **The Times of India** article "**Across India, high levels of toxins in groundwater"** by **Vishwa Mohan**[5] reveals the seriousness of heavy metal contamination of underground water in many districts across India. Due to careless disposal of coal ash, residents living near industries face critical health issues.

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[1] *Birol, Fatih, and Tim Gould. "Key Trends in India: Demand." India Energy Outlook. Paris: OECD/IEA, 2015. 22-23. Web. 28 July 2018.* https://www.iea.org/publications/freepublications/publication/IndiaEnergyOutlook_WEO2015.pdf

[2] *Abel,K.H.,andL.A.Rancitelli."Major,Minor,andTraceElementCompositionofCoalandFlyAsh,as Determined by Instrumental Neutron Activation Analysis." Advances in Chemistry. ACS Publications, 01 Sept. 1975. Web. 28 July 2018.* https://pubs.acs.org/doi/10.1021/ba-1975-0141.ch010.

[3] *Verma, Ravindra Kumar. "Fly Ash Generation at Coal/Lignite Based Thermal Power Stations and Its Utilization in the Country for the Year 2016-17." Central Electricity Authority. Central Electricity Authority, 2017. Web. 28 July 2018.*http://www.cea.nic.in/reports/others/thermal/tcd/flyash_201617.pdf

[4] *"Coal Ash Basics." EPA. Environmental Protection Agency, 26 Apr. 2017. Web. 28 July 2018*https://www.epa.gov/coalash/coal-ash-basics

[5] *Mohan, Vishwa. "Across India, High Levels of Toxins in Groundwater." The Times of India. The Times of India, 31 July 2018. Web. 06 Aug. 2018.*https://timesofindia.indiatimes.com/india/govt-body-finds-high-levels-of-groundwater-contamination-acro ss-india/articleshow/65204273.cms

In addition, air pollution due to carbon dioxide (CO_2) and oxides of sulphur and nitrogen results in acid rain, decreasing in pH and increasing hydrogen ion (H^+) concentration which displaces heavy metal cations (M) from their complexes in coal ash, thus, increasing desorption and concentration of heavy metals leaching into underground water.

$$\mathrm{M} + 2\mathrm{H}^+ \rightarrow \mathrm{M}^{2+} + \mathrm{H}_2$$

Hence, pH plays a role in leaching of heavy metals in soil from coal ash. This study examined the extent to which acid rain (caused by greenhouse gases) augments heavy metal pollution and how they toxicate drinking water by leaching into soil and underground water. Additionally, food crops grown in soil containing heavy metals get poisoned as they imbibe water containing these heavy metals and can enter the food chain, thus making this investigation prudent.

The context described above, led to a pre-lab wherein concentrations of chromium, copper, and manganese in coal ash were found to be above detection limit (as their samples showed colour changes). The maximum leaching potential was achieved in about 4 weeks as concentrations of chromium, copper, manganese did not increase significantly after that. Hence, the pre-lab led to the research question:

To what extent does pH (3.0-6.0) of sample of coal affect the leaching potential of toxic heavy metals (chromium, copper, and manganese) in groundwater and soil measured using spectrophotometry to analyze leachate from a lab scale prototype of earth over a period of 4 weeks?

This investigation involved making solutions containing coal ash of varying pH (3.0, 4.0, 5.0 and 6.0) which were left for 4 weeks. After each week, heavy metal analysis for chromium, copper and manganese was done of each sample using UV-Vis Spectrophotometer to determine

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[redacted]

maximum leaching potential at a specific pH over time. Variation in pH helped observe the effect of acid rain on heavy metal leaching. After 4 weeks, samples of pH 3.0 and 6.0, were passed through a lab prototype of soil as these pH levels represent an extreme case of acid rain and optimal case respectively. The research question was answered by comparing heavy metal concentration that leached into water from coal ash and into filtrate collected from soil prototype (representing underground water), showing the extent of heavy metal pollution. The difference in heavy metal concentrations suggested the amount of heavy metal retained by soil. Thus, both groundwater and soil pollution were investigated.

Background Information

Heavy metal composition of coal ash

300 million years ago, organic material like vegetation (ferns) and dead animals got buried and converted into peat due to compression, which under high pressure and temperature turned into coal via **carbonization**.[7] Thus, even though coal mostly consists of carbon, it also consists of few toxic heavy metals like chromium, manganese, copper etc. that damage environment to a great extent.[8] Incorrect disposal of coal ash leads to leaching of heavy metals into underground water which is utilized by humans directly for drinking and indirectly from food crops, consumption of which leads to health issues like neurotoxicological disorders, and damage of liver, kidneys and reproductive system.[9] According to **Environmental Protection Agency**[10], presence of arsenic in drinking water can lead to cancer.

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[7] *NationalGeographicSociety."Coal."NationalGeographicSociety.NationalGeographicSociety,18Dec.2012. Web. 23 Dec. 2018. https://www.nationalgeographic.org/encyclopedia/coal/*

[8] *Abel,K.H.,andL.A.Rancitelli."Major,Minor,andTraceElementCompositionofCoalandFlyAsh,as Determined by Instrumental Neutron Activation Analysis." Advances in Chemistry. ACS Publications, 01 Sept. 1975. Web. 28 July 2018. https://pubs.acs.org/doi/10.1021/ba-1975-0141.ch010.*

[9] *Lenntech. "Water Treatment Solutions." Lenntech Water Treatment & Purification, Lenntech, 1998.Web.www.lenntech.com/processes/heavy/heavy-metals/heavy-metals.htm.*

[10] *RTI. "Human and Ecological Risk Assessment of Coal Combustion Wastes." Southeastcoalash.org. RTI International, 6 Aug. 2007. Web. 30 Aug. 2018. http://www.southeastcoalash.org/wp-content/uploads/2012/05/epa-coal-combustion-waste-risk-assessment.pdf*

To make heavy metal removal from drinking water efficient and inexpensive, a **metal organic framework (MOF)** has been developed. It has interlinked metal and organic struts that pull-out water in vapor form leaving trace level heavy metals behind.[11] Water purifiers in households, use **Reverse Osmosis (RO) Technology** that is inexpensive and removes heavy metals at a low level. Also, **selective ion exchange resin** is used for low level removal of heavy metals and **ferric hydroxide** is used as it adsorbs toxic heavy metals.[12]

Heavy metal selection

According to **"Study on heavy metal contamination in groundwater at outskirts of Kota, India"**[13] iron, copper, chromium, zinc, manganese, and lead were found in groundwater around Kota. Out of these, chromium, manganese, lead exceeded the safe limit determined by **Bureau of Indian Standards**. During pre-lab, concentrations of zinc and iron were below detection limit of the spectrophotometer and showed no colour change upon addition of indicators. Concentrations of chromium, copper, and manganese were low but present in an amount that showed colour change during analysis. Also, analysis of lead required use of potassium cyanide (KCN) which is poisonous and the school safety protocol does not allow its use. Finally, **chromium, copper, and manganese** were selected for heavy metal analysis.

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[11] *Papageorgiou, Nik. "Removing Heavy Metals from Water." News RSS. EcolePolytechniqueFederale De Lausanne, 15 Mar. 2018. Web. 27 Aug. 2018.*
https://actu.epfl.ch/news/removing-heavy-metals-from-water/

[12] *Lenntech. "Water Treatment Solutions." Lenntech Water Treatment & Purification. Lenntech, 1998. Web. 28 Sept. 2018.*
https://www.lenntech.com/processes/heavy/heavy_metals_removal.html.

[13] *Gupta, Nitin, S.M. Nafees, M.K. Jain, and Kalpana S. "Study on heavy metal contamination in ground water at outer skirts of kota city, rajasthan, india." study on heavy metal contamination in ground water at outer skirts of kota city, rajasthan, india(2011). Web. 28 Sept. 2018.*
https://www.tsijournals.com/articles/study-on-heavy-metal-contamination-in-ground-water-at-outer-skirt s-of-kota-city-rajasthan-india.pdf.

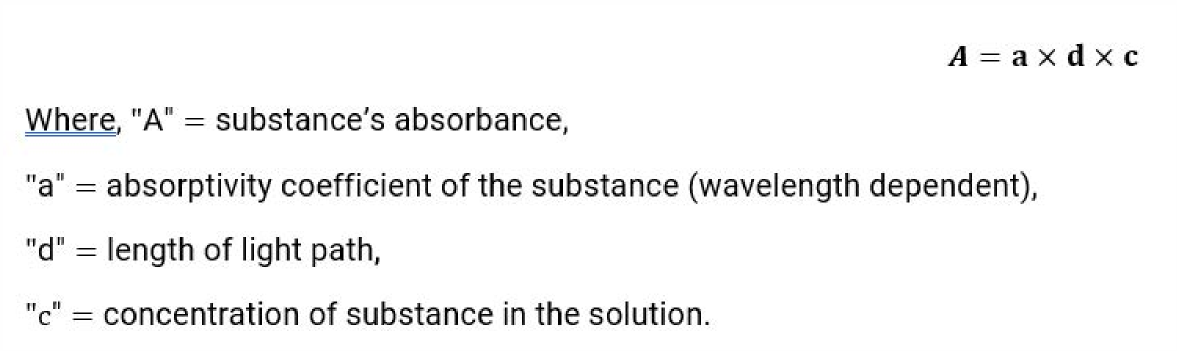
Selection of pH levels

Samples of pH 3.0, 4.0, 5.0, and 6.0 were prepared because pH 3.0 represented severe case of acid rain[14] and pH 6.0 was close to optimal pH of rainwater, i.e. 5.6 due to presence of weak carbonic acid formed by dissolution of atmospheric CO_2.[15]

In part II of the investigation, samples with pH 3.0 and 6.0, after 4 weeks, were chosen to flow through lab prototype of soil because this pH represented extreme and optimal cases. Moreover, duration of 4 weeks was chosen so that heavy metals could reach the maximum leaching potential.

Spectrophotometric method

When light is passed through a solution containing an analyte, some is absorbed by particles present in the solution. According to **Beer-Lambert Law**, concentration of a substance in a solution is proportional to amount of light (specific wavelength) absorbed by that substance, over a constant length of path. Quantitatively,:



$$A = \mathbf{a} \times \mathbf{d} \times \mathbf{c}$$

Where, "A" = substance's absorbance,

"a" = absorptivity coefficient of the substance (wavelength dependent),

"d" = length of light path,

"c" = concentration of substance in the solution.

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[14] Gandhi, Aadit, Parth Patel, and Girish Bagale. "A Study on Acid Rain: Effects and Control Measures." *Ijirset.com*. International Journal of Innovative Research in Science, Engineering and Technology, 04 Apr. 2017. Web. 09 Aug. 2018. http://www.ijirset.com/upload/2017/april/61_A%20Study.pdf

[15] *Dowdey, Sarah. "How Acid Rain Works." HowStuffWorks Science. HowStuffWorks, 28 June 2018. Web. 09 Aug. 2018.* https://science.howstuffworks.com/nature/climate-weather/atmospheric/acid-rain1.htm

[redacted]

Many researchers use sensitive spectroscopy methods for accurate determination of heavy metal concentration in waste water like **Atomic Absorption Spectroscopy (AAS)** and **Flame Emission Spectroscopy (FES)**. However, **UV-Visible Spectrophotometry** was chosen for this research as AAS and FES required highly sophisticated instruments that were not available in school laboratory. Moreover, UV-Visible Spectrophotometry uses simpler approach as it is capable of analyzing chromium, copper, and manganese by calibrating it to respective wavelengths, however, AAS requires different lamps for analysis different heavy metals.

To analyze **chromium**, diphenylcarbazide reacts with hexavalent chromium ions (all chromium ions converted to hexavalent by reaction with potassium permanganate) in acidic medium (shown in figure 1[17]) giving a magenta solution measured at 540nm to give chromium concentration in a range of 0.03mg/L to 20mg/L.[18]

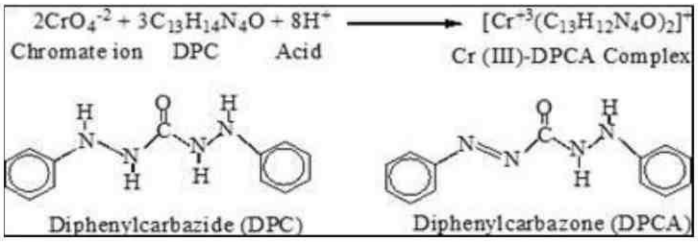

Figure 1: Reaction that aids in the analysis of chromium

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[17] *Sharma, Devesh K., and Rekha Sharma. "Use of Variamine Blue Dye in Spectrophotometric Determination of Water Soluble Cr(VI) in Portland Cement." Oriental Journal of Chemistry. Oriental Journal of Chemistry, 16 Dec. 2015. Web. 11 Aug. 2018.*
http://www.orientjchem.org/vol31no4/application-of-variamine-blue-dye-in-spectrophotometric-determination-of-water-soluble-crvi-in-portland-cement/

[18] *Bureau of Indian Standards. "METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER [Part 52 Chromium]."Part 52 Chromium (2003): 1-1. Web. 11 Aug. 2018.*
https://ia800404.us.archive.org/28/items/gov.in.is.3025.52.2003/is.3025.52.2003.pdf

In determination of **copper**, neocuproine reacts with copper (I) ions (converted from copper (II) ions) at pH 5.0, (shown in figure 2[redacted]), to give yellow solution measured spectrophotometrically at 457nm to give copper concentration in a range of 0.05mg/L to 5mg/L.[20]

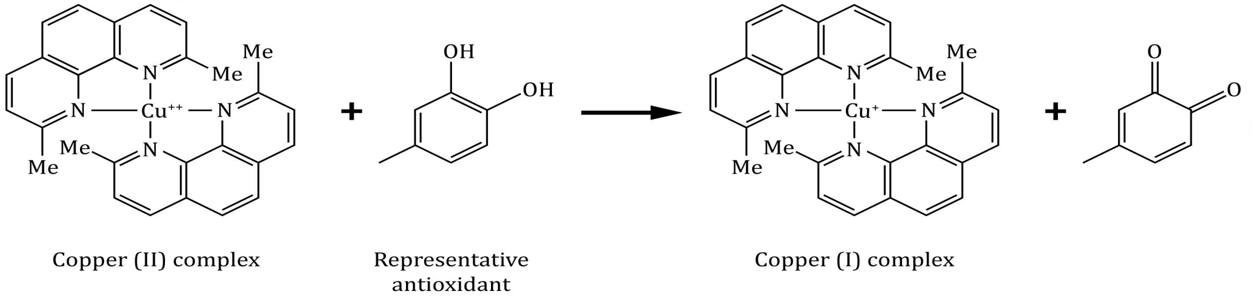

Figure 2: Reaction that aids in the analysis of copper

In determination of **manganese**, potassium periodate oxidizes manganese ions to permanganate ions to give a pink solution measured at 530nm to give manganese concentration in a range of up to 0.2mg.[21]

$$\mathrm{Mn^{2+} + 4H_2O - MnO_4^- + 8H^+ + 5e^-}$$

[redacted]

[20] *Bureau of Indian Standards. "METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER [Part 42 Copper]."Part 42 Copper (1992): 1-1. Web. https://archive.org/details/gov.law.is.3025.42.1992/page/n5 27 Aug. 2018.*

[21] *Bureau of Indian Standards. "METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER [Part 59 Manganese]."Part 59 Manganese (2006): 1-1. Web. https://archive.org/details/gov.law.is.3025.59.2006 27 Aug. 2018.*

[redacted]

Research variables and hypotheses

It was hypothesized that **decrease in pH (6.0, 5.0, 4.0, and 3.0) would lead to increment in solubility of heavy metals thus, leaching potential of chromium, copper and manganese would increase**, as compared to control with pH 6.0. Heavy metal leaching is pH dependent because of mechanism (described in Introduction). On the basis of hypothesis, following **dependent, independent and controlled variables** were chosen:

Table 1: List variables that were dependent or independent

Variable	Description
Type of heavy metal	**"Heavy metals leaching in Indian coal fly ash"** by **Prasad and Mondal**[23], stated that variety of heavy metals leach into groundwater, which was very broad for study, but, *copper, chromium, and manganese* were chosen as their extractions were feasible in the school laboratory.
pH of coal ash and water solution	Three solutions of *pH 3.0 4.0, 5.0, and a fourth solution of pH 6.0 as a control* were prepared which acted as rainwater of specific pH. Normally, rainwater has pH 5.6 and goes down to 3.0[24], hence, to maintain uniformity in the pH whose effect was being observed, a pH of 6.0 was taken as control instead of 5.6.
Time of contact of coal ash and water	Hazardous heavy metals leaching into earth from landfills full of industrial wastes is a continuous process, however, a period of *4 weeks* was chosen to make this research practical and get significant changes in heavy metal concentration over this time. Heavy metal analysis was done for each sample after *week 1, 2, 3, and 4*.
Solubility of heavy metals	Solubility is pH dependent as described in Introduction.
Absorbance of Cu, Cr and Mn.	Absorbance of Cu (at wavelength: 457nm), Cr (at wavelength: 540nm) and Mn (at wavelength: 530nm) depended on amount of these heavy metals in coal ash that dissolved in water.
Concentration of Cu, Cr, and Mn in leachate	It depended on amount of heavy metals that the soil retains.

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[23] *Prasad, B., and K KMondal. "Heavy Metals Leaching in Indian Fly Ash." Pubmed.gov. U.S. National Library of Medicine, Apr. 2008. Web. 06 Aug. 2018. https://www.ncbi.nlm.nih.gov/pubmed/19295096.*

[24] *Zheng, Shun-an, XiangqunZheng, and Chun Chen. "Leaching Behavior of Heavy Metals and Transformation of Their Speciation in Polluted Soil Receiving Simulated Acid Rain." PLOS ONE. Public Library of Science, 21 Nov. 2012. Web. 06 Aug. 2018. https://journals.plos.org/plosone/article?id=10.1371%2Fjournal.pone.0049664*

Table 2: Variables which were controlled, reasons for them to be controlled and method used to control them

Variable	Description	Why was it controlled?
Standard chromium solution	Five standard solutions of concentrations 1.5mg/L,3.0mg/L, 4.5mg/L, 6.0mg/L, 9.0mg/L were prepared.	These concentrations were prepared using $3cm^3$, $6cm^3$, $9cm^3$, $12cm^3$ and $18cm^3$of the stock solution of coal ash. These concentrations were within range of 0.03mg/L to 20mg/L as only concentrations within this range can be spectrophotometrically analyzed[25].
Standard copper (II) solution	Five standard solutions of concentrations 0.05mg/L,0.1mg/L, 0.5mg/L, 1.0mg/L, 5.0mg/L were prepared.	Following instructions as given in standard procedure for heavy metal analysis for copper, five standard solution were prepared whose concentrations were within range of 0.05mg/L to 5mg/L as only concentrations within this range can be spectrophotometrically analyzed[26].
Standard manganese solution	Five standard solutions of concentrations 1.95mg/L,3.90mg/L,5.86mg/L, 7.82mg/L and 9.77mg/L were prepared.	These concentrations were prepared using $0.5cm^3$, $1.0cm^3$, $1.5cm^3$, $2.0cm^3$, $2.5cm^3$. According to the research paper, **"Spectrophotometric determination of manganese in groundwater in Shillong city using bisulphate oxidation method",** the minimum limit for detection of manganese using visible spectrophotometry is 0.1mg/Land the calibration curve obeys Beer-Lambert's law till a concentration of 10mg/L[27].Thus, all concentrations were within this range for spectrophotometric analysis.
Amount of sample	A fixed amount of 10g of coal ash was mixed with a fixed volume of water ($200cm^3$) of varying pH.	If the amount of coal ash varies the amount of heavy metals present in it will also vary, hence, the amount was fixed.
Filter paper	Filter papers of the same manufacturer and same pore size were used.	All particles with a size more than the size of the pores were blocked as these might interfere in the heavy metal analysis of the samples. Even though some interfering particles might have passed through the filter paper, they were same in all samples as the pore size is constant.

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[25] *Bureau of Indian Standards. "METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER [Part 52 Chromium]."Part 52 Chromium (2003): 1-1. Web. 20Dec. 2018 https://ia800404.us.archive.org/28/items/gov.in.is.3025.52.2003/is.3025.52.2003.pdf*

[26] *Bureau of Indian Standards. "METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER [Part 42 Copper]."Part 42 Copper (1992): 1-1. Web. 20Dec. 2018 https://archive.org/details/gov.law.is.3025.42.1992/page/n5*

[27] *Marbaniang, D.G. "Spectrophotometric Determination of Manganese in Ground Water in Shillong City Using Bismuthate Oxidation Method." International Journal of Environmental Protection. World Academic Publishing, 2012. Web. 09 Aug. 2018. http://www.academicpub.org/ijep/paperInfo.aspx?paperid=1140*

Alternatively, it was hypothesized that decrease in pH (6.0, 5.0, 4.0, and 3.0) would lead to decrement in solubility of heavy metals thus, leaching potential would decrease, as compared to control with pH 6.0.

It was also assumed that there might be a case where decrease in pH (6.0, 5.0, 4.0, and 3.0) would have no effect on leaching potential as compared to the control.

Materials required

Table 3: Materials required

S. No.	Apparatus	Quantity
1.	Electronic weighing scale (± 0.001g)	1
2.	Vernier Magnetic stirrer	1
3.	Vernier SpectroVis plus Spectrophotometer	1
4.	Vernier pH meter (± 0.005 pH units)	1
5.	Vernier Drop counter	1
6.	Erlenmeyer flasks ($250cm^3$ and $100cm^3$)	8
7.	Beakers ($250cm^3$ and $100cm^3$)	6
8.	Measuring cylinders ($5cm^3 \pm 0.25cm^3$, $100cm^3 \pm 0.5cm^3$, $250cm^3 \pm 5cm^3$, $1000cm^3 \pm 12.5cm^3$)	1 each
9.	Separating funnel	1
10.	Spatula	As required
11.	Watch glass	As required
12.	China dish	As required
13.	Whattman filter paper	20
14.	Dropper	1
15.	Glass rod	1
16.	Pipette with suction pump ($10cm^3$ and $20cm^3$) ($\pm 0.1cm^3$)	2
17.	Bunsen burner	1
18.	Laptop with Logger Pro software	1
19.	Clean cloth	1
20.	Cotton	As required

Table 4: Chemicals required

S. No.	Chemicals	Molecular Formula	Manufacturer	Quantity
1.	Coal ash	-	-	537.2g
2.	0.1N and 0.5N Sulphuric acid	H_2SO_4	Fisher Scientific	200.0cm^3
3.	Potassium Dichromate	$K_2Cr_2O_7$	Merck	0.2828g
4.	6M Nitric acid	HNO_3	Rankem	20cm^3
5.	Phosphoric acid	H_3PO_4	Ases	10cm^3
6.	Methyl orange indicator	$C_{14}H_{14}N_3NaO_3S$	Rankem	20cm^3
7.	Ammonium hydroxide	NH_4OH	Merck	100cm^3
8.	Potassium permanganate	$KMnO_4$	Merck	0.7890g
9.	Sodium azide	NaN_3	CDH	10cm^3
10.	Diphenylcarbazide	$C_{13}H_{14}N_4O$	CDH	0.5000g
11.	Acetone	C_3H_6O	Rankem	100.0cm^3
12.	Chloroform	$CHCl_3$	Fisher Scientific	100.0cm^3
13.	Hydroxylamine Hydrochloride	$HONH_2 \cdot HCl$	Rankem	28.00g
14.	2-Propanol	C_3H_8O	Emplura	100.0cm^3
15.	Neocuproine	$C_{14}H_{12}N_2$	CDH	0.2000g
16.	Trisodium citrate	$Na_3C_6H_5O_7$	Merck	35.00g
17.	Hydrated copper sulphate	$CuSO_4.5H_2O$	Merck	0.2000g
18.	Potassium periodate	KIO_4	CDH	10g
19.	Methanol	CH_3OH	Rankem	
19.	Distilled water	H_2O	Prepared in lab using Distillation Plant	5.000dm^3

Image 1: A close up of the materials and chemicals used

Safety, environmental and ethical issues

As sulphuric acid, potassium dichromate, nitric acid, phosphoric acid, ammonium hydroxide, acetone, copper sulphate pentahydrate,[28] methyl orange indicator[29], sodium azide[30], diphenylcarbazide[31], chloroform[32], hydroxylamine hydrochloride[33], neocuproine[34]and potassium periodate[35]could cause severe damage to skin and eyes and were corrosive, so lab coat, gloves and safety glasses were worn with hair tied back. They were stored in tightly sealed container and suction pump was used to pipette them. An eyewash station was available if any chemical splashed into the eyes. Highly combustible chemicals like 2-propanol[36] were kept away from flame. Moreover, some chemicals (potassium dichromate, ammonium hydroxide, potassium permanganate and sodium azide) are harmful for the environment as they toxicate aquatic life and were labeled **hazardous** before carefully disposing.

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[28] Cleapss. *Student Safety Sheets*. Uxbridge: Cleapss, 2016. Print.

[29] *Lobachemie.com. "METHYL ORANGE INDICATOR SOLUTION MSDS."Lobachemie.com., 2017. Web. 20 Dec. 2018* https://www.lobachemie.com/lab-chemical-msds/MSDS-METHYL-ORANGE-INDICA TOR-SOLUTION-CASNO-04671-EN.aspx

[30] *Cdhfinechemical.com. "Sodium Azide MATERIAL SAFETY DATA SHEET SDS/MSDS."Cdhfinechemical.com.,2008. Web. 20 Dec. 2018* https://www.cdhfinechemical.com/images/product/msds/101_2069514636_SodiumAzide-CASNO-26628-22-8-MSDS.pdf

[31] Cdhfinechemical.com. "*1,5-Diphenyl Carbazide MATERIAL SAFETY DATA SHEET.*" Cdhfinechemical.com., 2008. Web. 20 Dec. 2018 https://www.cdhfinechemical.com/images/product/msds/13_1000140028_1,5-DiphenylC arbazide-CASNO-140-22-7-MSDS.pdf

[32] Beta-static.fishersci.com. "*Safety Data Sheet Chloroform, Reagent Grade*."Beta-static.fishersci.com., 2015.Web. 20 Dec. 2018 static.fishersci.com/content/dam/fishersci/en_US/documents/programs/educat ion/regulatory-documents/sds/chemicals/chemicals-c/S25248.pdf

[33] Sciencelab.com. "*Material Safety Data Sheet Hydroxylamine hydrochloride MSDS*." Sciencelab.com., 2005. Web. 20 Dec. 2018 http://www.sciencelab.com/msds.php?msdsId=9927192

[34] Cdhfinechemical.com. "*Neocuproine MATERIAL SAFETY DATA SHEET SDS/MSDS*."Cdhfinechemical.com., 2008. Web. 20 Dec. 2018 https://www.cdhfinechemical.com/images/product/msds/37_141396444_Neocuproine-C ASNO-484-11-7-MSDS.pdf

[35] Sciencelab.com. "*Material Safety Data Sheet Potassium periodate MSDS*." Sciencelab.com., 2005. Web. 20 Dec. 2018 http://www.sciencelab.com/msds.php?msdsId=9924688

[36] Labchem.com. "*Isopropyl Alcohol (2-Propanol) Safety Data Sheet*." Labchem.com., 2013. Web. 20 Dec. 2018 http://www.labchem.com/tools/msds/msds/LC15750.pdf

As thermal power plants, underground water and soil are crucial for economy and environment, authenticity and honesty was ensured while collecting and assessing data as the results would be beneficial in determining steps to preserve underground water and land while preventing thermal power plants from complete endangerment.

Procedure

Part 1: Determination of heavy metal leaching potential in sample of coal ash with varying pH of 3.0, 4.0, 5.0, and 6.0

Pre-lab for preparation of solutions of different pH (3.0, 4.0, 5.0 and 6.0)

$$H_2SO_4 \rightarrow 2H^+ + SO_4$$

For PH 3.0 solution

Concentration of H^+ ions $= 10^{-3}M$

Concentration of $H_2SO_4, C_1 = 0.5 \times 10^{-3}M$

Volume of resultant solution, $V_1 = 200cm^3$

Molarity of $0.1N$ $H_2SO_4, C_2 = \frac{\text{Normality}}{\text{Bascity}} = \frac{0.1}{2} = 0.05M$

$C_1 \times V_1 = C_2 \times V_2$

$$V_2 = \frac{0.5 \times 10^{-3}moldm^{-3} \times 0.2dm^3}{0.05moldm^{-3}} = 0.002dm^3 = 2cm^3$$

Similarly, volume of H_2SO_4 required for preparation of pH 4.0, 5.0, and 6.0 were found as $0.2cm^3$, $0.02cm^3$, and $0.002cm^3$. As $0.02cm^3$ and 0.002 cm^3 were very small to be measured accurately, $1000cm^3$ solutions were prepared and $200cm^3$ was used.

[redacted]

Sample preparation

Coal ash was obtained from a thermal power station in Kota, Rajasthan. 10g coal ash sample each was put in four different 250cm^3 Erlenmeyer flasks. 200cm^3 solutions with pH 3.0, 4.0, 5.0 and 6.0 were prepared using 0.1N H_2SO_4 (volume of H_2SO_4 calculated in pre-lab) and added to each flask respectively. After each week (till Week 4), each sample was analyzed for Cu, Cr, and Mn.

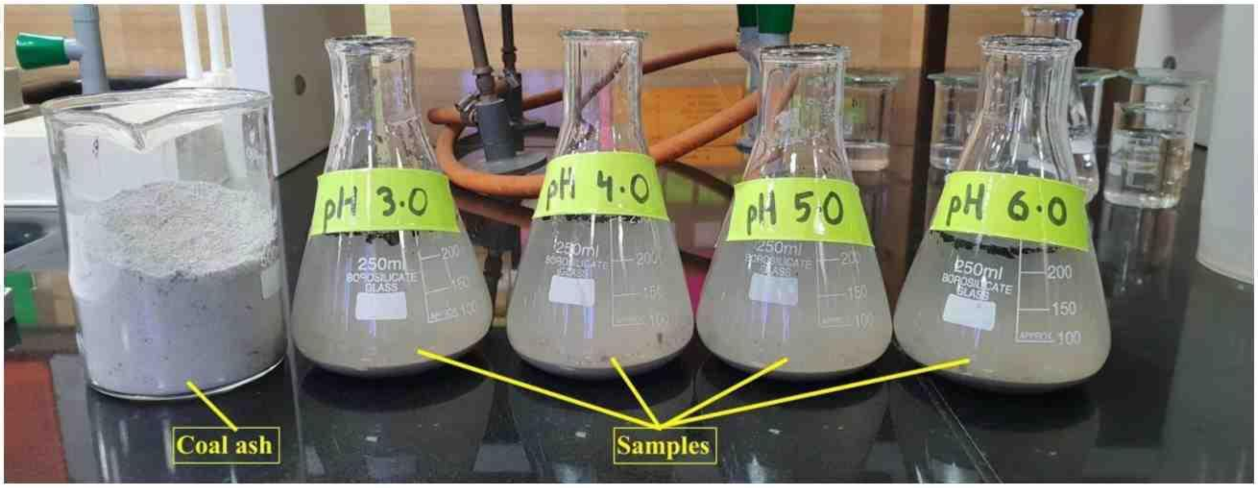

Image 2: Coal ash used in the investigation and the samples of varying pH prepared

Heavy metal analysis

The procedures of chromium[39], and copper[40] analysis were adapted from standard procedures given by the **Bureau of Indian Standards.** And for manganese[41] analysis it was adapted from **University Of Canterbury.**

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[39] *Bureau of Indian Standards."METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER [Part 52 Chromium]."Part 52 Chromium (2003): 1-1. Web. 20Dec. 2018. https://ia800404.us.archive.org/28/items/gov.in.is.3025.52.2003/is.3025.52.2003.pdf*

[40] *Bureau of Indian Standards. "METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER [Part 42 Copper]."Part 42 Copper (1992): 1-1. Web. 20Dec. 2018https://archive.org/details/gov.law.is.3025.42.1992/page/n5*

[41] College of Science, University of Canterbury. "Determination of Manganese Concentration in Fertiliser." *Www.canterbury.ac.nz*. University of Canterbury. Web. 20 Dec. 2018. https://www.canterbury.ac.nz/media/documents/science-outreach/manganese_fertiliser.pdf

Diphenylcarbazide method for chromium for calibration of spectrophotometer:

0.1414g potassium dichromate was mixed in 100cm^3 water to make 0.005moldm^{-3} stock chromium solution. Standard solutions (different concentrations) were prepared by using proportions given below:

Table 5: Composition of chromium standard solutions

S.No.	Volume of stock (cm^3) (±0.5cm^3)	Volume of distilled water used (cm^3) (±1.0cm^3)	Concentration (mg/L)
1.	3	47	1.50
2.	6	44	3.00
3.	9	41	4.50
4.	12	38	6.00
5.	18	32	9.00

0.5N H_2SO_4 was added dropwise, with pH meter in the beaker to reduce pH to 1.0 because diphenylcarbazide works in acidic medium. 2cm^3 diphenylcarbazide (0.25g diphenylcarbazide in 50cm^3 acetone) was added and diluted to 100cm^3. While full color developed, blank reagent was prepared in 10cm^3 water, using above method, and was used to calibrate spectrophotometer for plotting absorbance curves for standard solutions at 540nm.

Image 3: Chromium standard solutions and blank prepared

Absorbance of chromium in sample:

10cm^3 sample with pH 3.0 was taken and diluted to 50cm^3. Concentrated NH_4OH was added after adding methyl orange, until solution just turned pink. Next, it was boiled, while adding 0.25M $KMnO_4$ until the pink colour became permanent. Boiling was continued for few more minutes while adding 1.0cm^3 NaN_3 till pink colour faded. After letting the solution cool, 5 drops of 14.8M $H_3PO_4$was added and pH was brought to 1.0 using H_2SO_4. This solution was transferred to 100 cm^3 beaker, 2.0cm^3 diphenylcarbazide was added, and volume was made up to 100cm^3 using distilled water and absorbance was measured at 540nm. Same method was repeated for remaining samples.

Image 4: Solution after colour changed to pink

Neocuproine method for copper for calibration of spectrophotometer:

0.0125moldm^{-3} stock copper sulphate was prepared by dissolving 0.2g hydrated copper sulphate in 100cm^3 water then making up to 500cm^3. Standard solutions (different concentrations) were prepared using proportions given below:

Table 6: Composition of copper standard solutions

S.No.	Volume of stock (cm^3) (±0.05cm^3)	Using distilled water make up to (cm^3) (±1.0cm^3)	Concentration (mg/L)
1.	0.1	200	0.05
2.	0.1	100	0.10
3.	0.5	100	0.50
4.	1.0	100	1.00
5.	5.0	100	5.00

Each standard was shaken with 5cm^3 hydroxylamine-hydrochloride, 10cm^3 trisodium citrate, and 10cm^3 neocuproine. First standard solution was poured in a separating funnel and 20cm^3 chloroform was added and shaken for a minute. The chloroform layer was separated, collected, mixed and diluted to 50cm^3 using propan-2-ol, in an Erlenmeyer flask. Using this method, blank reagent was prepared in 50cm^3 distilled water and calibration graph was plotted at 457nm.

Image 5: Copper standard solutions and blank prepared

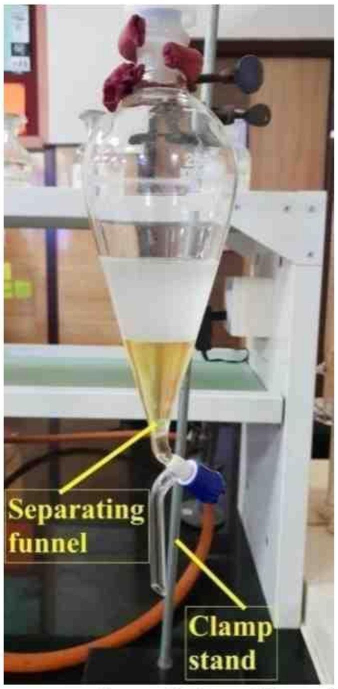


Image 6: Solution after separation of the chloroform layer at the bottom

Absorbance of copper in sample:

The method described above was used to treat 50cm^3 of all samples and their absorbance was measured at 457nm.

Periodate spectrophotometric method for manganese for calibration of spectrophotometer:

0.789g $KMnO_4$ was taken in 1000cm^3 measuring cylinder and diluted to mark to prepare stock manganese solution. Standard solutions (different concentrations) were prepared using proportions given below:

Table 7: Composition of manganese standard solutions

S. No.	Volume of stock (cm^3) (±0.5cm^3)	Using distilled water make up to (cm^3) (±1.0cm^3)	Concentration (mg/L)
1.	0.5	200	1.95
2.	1.0	200	3.90
3.	1.5	200	5.86
4.	2.0	200	7.82
5.	2.5	200	9.77

50cm^3 blank reagent was used as reference to plot a calibration graph at 530 nm.

Absorbance of manganese in sample:

20cm^3 sample with pH 3.0 was pipetted into 250cm^3 Erlenmeyer flask and 5cm^3 6M HNO_3 and 2.0g potassium periodate was added. The solution was diluted to 50cm^{3}and boiled for ten minutes. A pink colour developed and solution was diluted to 100cm^3 after cooling. Absorbance was measured at 530nm and all samples were analyzed in the same way.

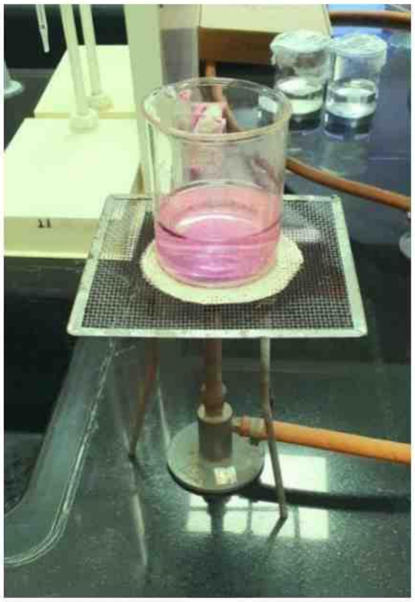


Image 7: Solution after colour change

Part 2: Determination of heavy metal concentration after leaching through soil prototype

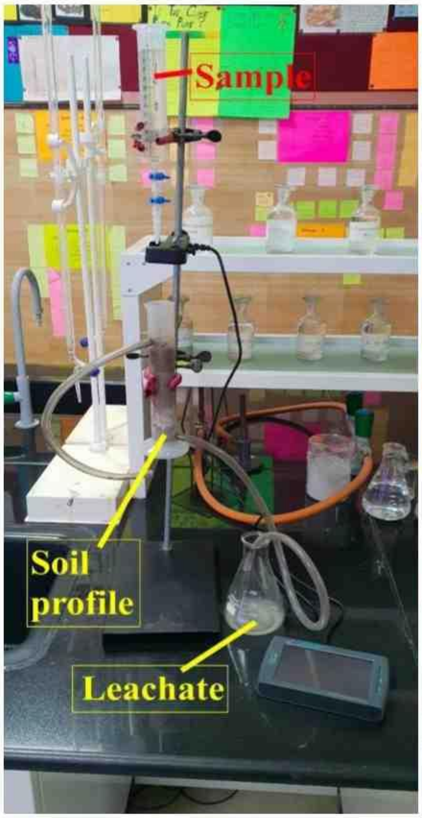


Image 8: Layers in the earth prototype

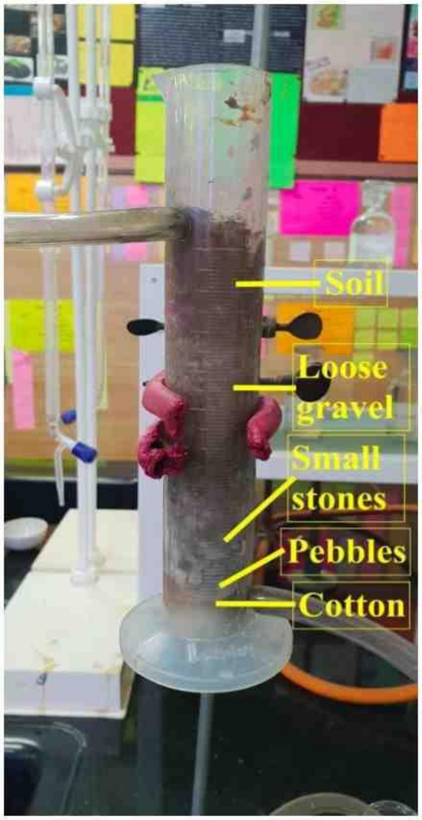


Image 9: Setup for observing leaching

At the bottom of the measuring cylinder (with openings at the top and bottom) a layer of cotton was put so that soil did not slip through the opening. Then a soil prototype was built starting with a layer of pebbles, second layer of small stones, third layer of loose gravel, and top layer of soil from the field in school. Measuring tube attached on top of measuring cylinder contained sample of pH 3.0 whose flow into the measuring cylinder was controlled by a drop counter. Leachate was collected in the flask placed below the measuring cylinder which was analyzed for heavy metal concentrations using methods described in Part 1. This was repeated for samples of pH 6.0 **after allowing distilled water to pass through to remove traces of previous samples.**

Data collection

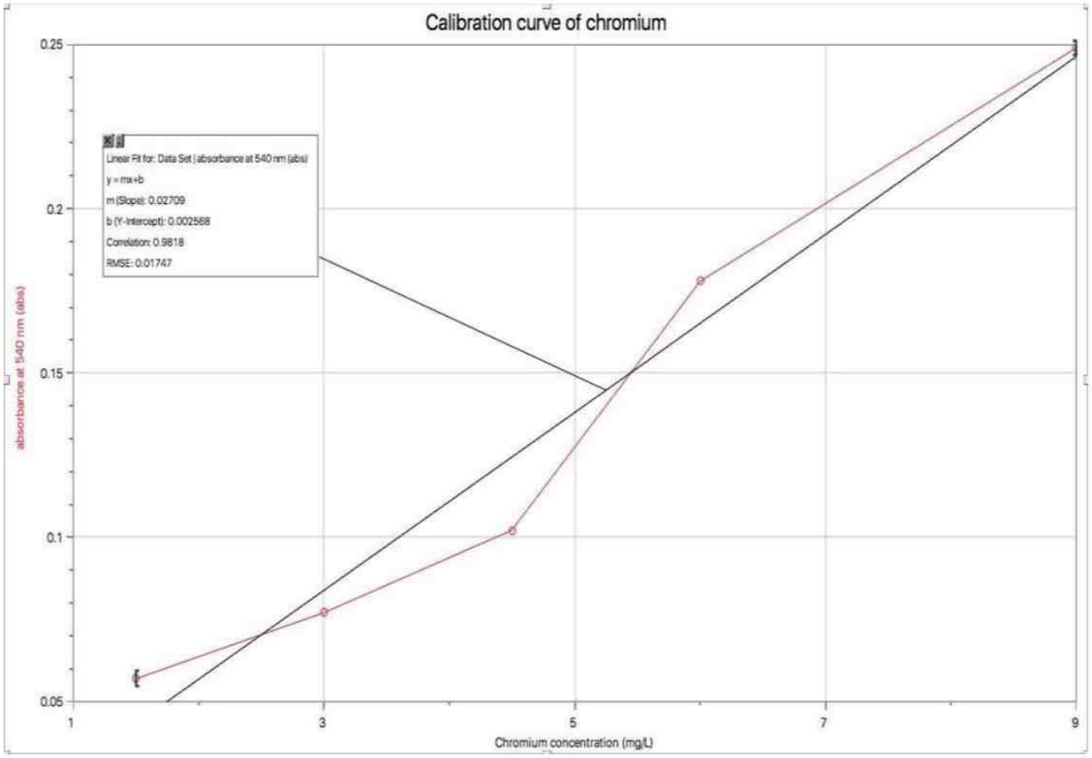
Qualitative data

After chromium analysis, solutions with more chromium had darker pink colour while solutions with low chromium had lighter pink colour. In copper analysis, the collected solutions from the separating funnel with high copper concentration showed dark yellow colour (mustard-like) while low copper concentration showed light yellow colour. In manganese analysis, solutions with high manganese showed dark violet-pink colour and solution with low manganese showed light pink colour.

Calibration curves

Table 8: Absorbance of chromium standard solutions

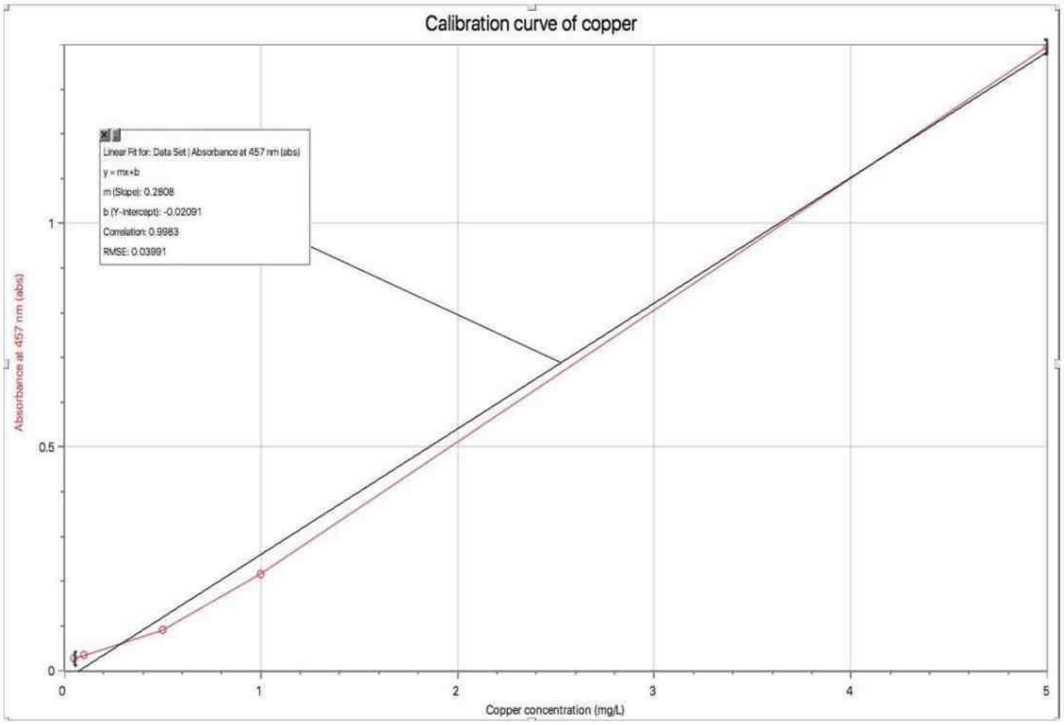
S. No.	Concentration of chromium standard solution (mg/L)	Absorbance at 540 nm (abs)
1.	1.50	0.067
2.	3.00	0.077
3.	4.50	0.102
4.	6.00	0.178
5.	9.00	0.249



Graph 1: Calibration curve of chromium

Table 9: Absorbance of copper standard solutions

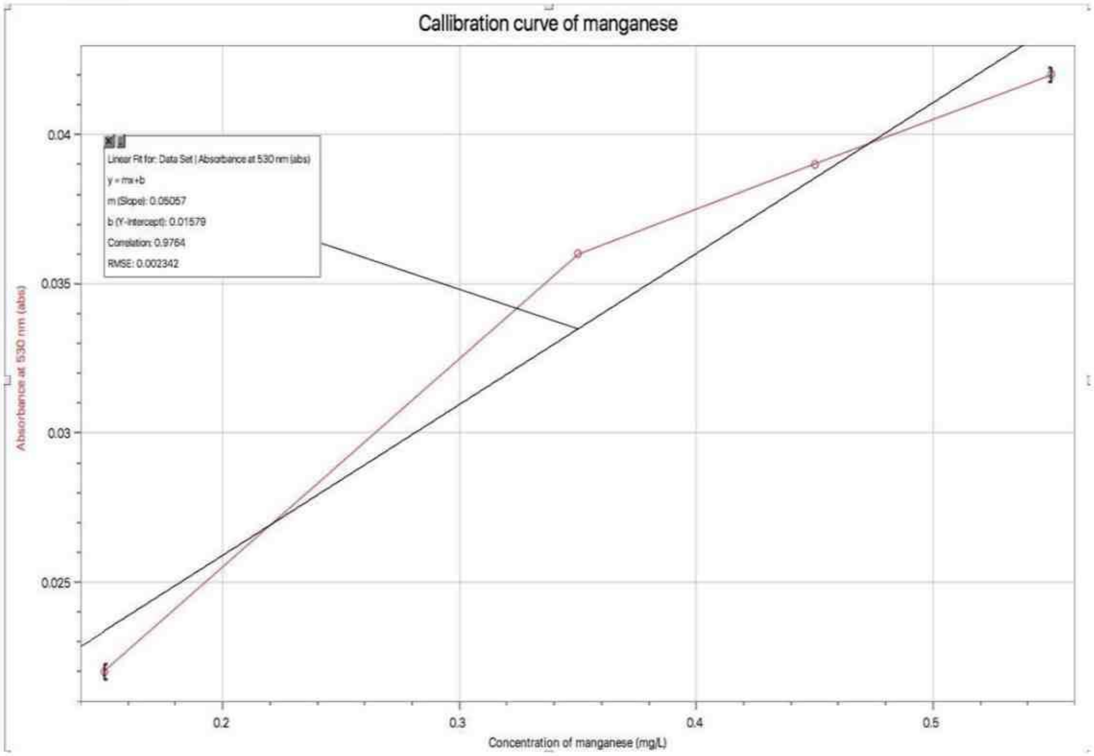
S. No.	Concentration of copper standard solution (mg/L)	Absorbance at 457 nm (abs)
1.	0.05	0.027
2.	0.1	0.035
3.	0.5	0.091
4.	1.0	0.216
5.	5.0	1.394



Graph 2: Calibration curve of copper

Table 10: Absorbance of manganese standard solutions

S. No.	Concentration of manganese standard solution (mg/L)	Absorbance at 530 nm (abs)
1.	0.15	0.022
2.	0.25	0.027
3.	0.35	0.036
4.	0.45	0.039
5.	0.55	0.042



Graph 3: Calibration curve of manganese

Part 1:

Table 11: Absorbance of chromium at 540nm (abs) after week 1

Week 1	Trial 1 Absorbance (abs)	Trial 2 Absorbance (abs)	Trial 3 Absorbance (abs)
3.0	0.043	0.038	0.034
4.0	0.036	0.038	0.036
5.0	0.035	0.039	0.034
6.0	0.034	0.034	0.032

Table 12: Absorbance of chromium at 540nm (abs) after week 2

Week 2	Trial 1 Absorbance (abs)	Trial 2 Absorbance (abs)	Trial 3 Absorbance (abs)
3.0	0.042	0.042	0.040
4.0	0.040	0.038	0.037
5.0	0.039	0.035	0.036
6.0	0.033	0.031	0.039

Table 13: Absorbance of chromium at 540nm (abs) after week 3

Week 3	Trial 1 Absorbance (abs)	Trial 2 Absorbance (abs)	Trial 3 Absorbance (abs)
3.0	0.041	0.045	0.039
4.0	0.039	0.039	0.038
5.0	0.039	0.036	0.040
6.0	0.031	0.040	0.033

Table 14: Absorbance of chromium at 540nm (abs) after week 4

Week 4	Trial 1 Absorbance (abs)	Trial 2 Absorbance (abs)	Trial 3 Absorbance (abs)
3.0	0.041	0.043	0.043
4.0	0.043	0.044	0.040
5.0	0.039	0.039	0.038
6.0	0.038	0.041	0.040

Table 15: Absorbance of copper at 457nm (abs) after week 1

Week 1	Trial 1 Absorbance (abs)	Trial 2 Absorbance (abs)	Trial 3 Absorbance (abs)
3.0	0.250	0.254	0.253
4.0	0.209	0.214	0.211
5.0	0.212	0.212	0.215
6.0	0.152	0.160	0.157

Table 16: Absorbance of copper at 457nm (abs) after week 2

Week 2	Trial 1 Absorbance (abs)	Trial 2 Absorbance (abs)	Trial 3 Absorbance (abs)
3.0	0.254	0.258	0.250
4.0	0.202	0.209	0.201
5.0	0.215	0.213	0.215
6.0	0.201	0.204	0.202

Table 17: Absorbance of copper at 457nm (abs) after week 3

Week 3	Trial 1 Absorbance (abs)	Trial 2 Absorbance (abs)	Trial 3 Absorbance (abs)
3.0	0.255	0.259	0.252
4.0	0.223	0.218	0.221
5.0	0.215	0.218	0.20
6.0	0.203	0.201	0.207

Table 18: Absorbance of copper at 457nm (abs) after week 4

Week 4	Trial 1 Absorbance (abs)	Trial 2 Absorbance (abs)	Trial 3 Absorbance (abs)
3.0	0.252	0.255	0.256
4.0	0.221	0.222	0.224
5.0	0.223	0.224	0.224
6.0	0.207	0.202	0.205

Table 19: Absorbance of manganese at 530nm (abs) after week 1

Week 1	Trial 1 Absorbance (abs)	Trial 2 Absorbance (abs)	Trial 3 Absorbance (abs)
3.0	0.026	0.028	0.025
4.0	0.027	0.024	0.026
5.0	0.022	0.025	0.020
6.0	0.183	0.187	0.182

Table 20: Absorbance of manganese at 530 nm (abs) after week 2

Week 2	**Trial 1 Absorbance (abs)**	**Trial 2 Absorbance (abs)**	**Trial 3 Absorbance (abs)**
3.0	0.028	0.025	0.027
4.0	0.027	0.028	0.024
5.0	0.021	0.025	0.026
6.0	0.022	0.027	0.024

Table 21: Absorbance of manganese at 530nm (abs) after week 3

Week 3	**Trial 1 Absorbance (abs)**	**Trial 2 Absorbance (abs)**	**Trial 3 Absorbance (abs)**
3.0	0.026	0.029	0.028
4.0	0.029	0.026	0.024
5.0	0.023	0.026	0.025
6.0	0.029	0.029	0.027

Table 22: Absorbance of manganese at 530nm (abs) after week 4

Week 4	**Trial 1 Absorbance (abs)**	**Trial 2 Absorbance (abs)**	**Trial 3 Absorbance (abs)**
3.0	0.028	0.029	0.029
4.0	0.027	0.023	0.030
5.0	0.022	0.027	0.028
6.0	0.029	0.023	0.024

Part 2:

Table 23: Absorbance of chromium, copper and manganese (abs) after leaching at pH 3.0 and 6.0

pH	**Absorbance of leachate (abs)**								
	Chromium			**Copper**			**Manganese**		
(± 0.005)	**Trial 1**	**Trial 2**	**Trial 3**	**Trial 1**	**Trial 2**	**Trial 3**	**Trial 1**	**Trial 2**	**Trial 3**
3.0	0.061	0.062	0.059	0.196	0.191	0.193	0.025	0.029	0.026
6.0	0.046	0.048	0.049	0.154	0.153	0.153	0.024	0.023	0.028

Processed data tables:

Part 1:

- **Sample calculation**

$$\text{Average absorbance} = \frac{Trial\ 1 + Trial\ 2 + Trial\ 3}{3}$$

Average absorbance of chromium at pH 3.0 in week 1 = $\frac{0.043+0.038+0.034}{3}$ = 0.038abs

To calculate concentration,

$$Absorbance = m\ (concentration) + c,$$

Where m is slope and c is y intercept.

For chromium - pH 3.0 - week 1:

0.038 = 0.02709 (concentration) + 0.002568

Concentration = 1.31mg/L

- **Chromium**

Table 24: Average absorbance (abs) of chromium in all samples

Week	pH (± 0.005)			
	3.0	4.0	5.0	6.0
	Average absorbance (abs)			
1	0.038	0.037	0.036	0.033
2	0.041	0.038	0.037	0.035
3	0.042	0.039	0.038	0.035
4	0.043	0.042	0.039	0.040

- **Copper**

Table 25: Average absorbance (abs) of copper in all samples

Week	pH (± 0.005)			
	3.0	4.0	5.0	6.0
	Average absorbance (abs)			
1	0.252	0.211	0.213	0.157
2	0.254	0.204	0.214	0.202
3	0.255	0.221	0.219	0.203
4	0.254	0.222	0.224	0.205

- **Manganese**

Table 26: Average absorbance (abs) of manganese in all samples

Week	pH (± 0.005)			
	3.0	4.0	5.0	6.0
	Average absorbance (abs)			
1	0.026	0.025	0.023	0.184
2	0.028	0.026	0.024	0.024
3	0.028	0.026	0.025	0.028
4	0.029	0.027	0.0258	0.025

- **Chromium**

Table 27: Concentration of chromium (mg/L) in each sample after each week

Week	pH (± 0.005)			
	3.0	4.0	5.0	6.0
	Average concentration (mg/L)			
1	1.31	1.26	1.22	1.13
2	1.43	1.32	1.27	1.19
3	1.46	1.33	1.29	1.21
4	1.51	1.45	1.34	1.39

- **Copper**

Table 28: Concentration of copper (mg/L) in each sample after each week

Week	pH (± 0.005)			
	3.0	4.0	5.0	6.0
	Average concentration (mg/L)			
1	0.971	0.825	0.834	0.728
2	0.979	0.802	0.837	0.794
3	0.981	0.861	0.853	0.797
4	0.980	0.866	0.872	0.803

- **Manganese**

Table 29: Concentration of manganese (mg/L) in each sample after each week

Week	pH (± 0.005)			
	3.0	4.0	5.0	6.0
	Average concentration (mg/L)			
1	0.208	0.175	0.160	0.155
2	0.236	0.198	0.162	0.170
3	0.241	0.205	0.184	0.181
4	0.256	0.219	0.197	0.184

Part 2:

Table 30: Absorbance of chromium, copper and manganese (abs) in the leachate at pH 3.0 and 6.0

pH (± 0.005)	Absorbance of leachate (abs)		
	Chromium	**Copper**	**Manganese**
3.0	0.034	0.193	0.026
6.0	0.033	0.153	0.025

Table 31: Concentration of chromium, copper and manganese (mg/L) in the leachate collected through earth prototype at pH 3.0 and 6.0

	Concentration of chromium (mg/L)		Concentration of copper (mg/L)		Concentration of manganese (mg/L)	
pH (± 0.005)	**in sample**	**in leachate**	**in sample**	**in leachate**	**in sample**	**in leachate**
3.0	1.51	1.16	0.980	0.762	0.256	0.210
6.0	1.39	1.13	0.803	0.619	0.184	0.178

Standard deviation

$$\text{Standard deviation} = \sqrt{\frac{\sum(x-m)^2}{m}},$$

n = number of values of absorbance,

x = one of these values

m = mean

[redacted]

Table 32: Standard deviation in the absorbance of chromium

pH	Week 1	Week 2	Week 3	Week 4
3.0	0.192	0.199	0.200	0.201
4.0	0.188	0.192	0.193	0.201
5.0	0.186	0.188	0.192	0.193
6.0	0.180	0.182	0.183	0.195

Table 33: Standard deviation in the absorbance of copper

pH	Week 1	Week 2	Week 3	Week 4
3.0	0.434	0.435	0.436	0.435
4.0	0.408	0.403	0.415	0.416
5.0	0.409	0.410	0.408	0.417
6.0	0.363	0.402	0.403	0.403

Table 34: Standard deviation in the absorbance of manganese

pH	Week 1	Week 2	Week 3	Week 4
3.0	0.160	0.161	0.164	0.167
4.0	0.158	0.160	0.160	0.161
5.0	0.148	0.153	0.155	0.158
6.0	0.387	0.154	0.166	0.157

Uncertainty analysis

As concentration is in the form *absorbance = m (concentration) + c* where *m* and *c* are constants, applying rules of error propagation:

$$\text{Uncertainty in concentration} = \text{Uncertainty in absorbance} = \frac{\text{Maximum absorbance} - \text{Minimum absorbance}}{2}$$

[redacted]

Uncertainty chromium - pH 3.0 - week 1

$$= \frac{0.043 - 0.034}{2} = \pm 0.0045\text{mg/L}$$

Table 35: Uncertainty in the concentration of chromium

pH	Week 1	Week 2	Week 3	Week 4
3.0	0.0045	0.0010	0.0030	0.0010
4.0	0.0010	0.0015	0.0005	0.0020
5.0	0.0025	0.0020	0.0020	0.0005
6.0	0.0010	0.0040	0.0045	0.0015

Table 36: Uncertainty in the concentration of copper

pH	Week 1	Week 2	Week 3	Week 4
3.0	0.0020	0.0040	0.0035	0.0020
4.0	0.0025	0.0040	0.0025	0.0015
5.0	0.0015	0.0010	0.0090	0.0005
6.0	0.0040	0.0015	0.0030	0.0025

Table 37: Uncertainty in the concentration of manganese

pH	Week 1	Week 2	Week 3	Week 4
3.0	0.0015	0.0015	0.0015	0.0005
4.0	0.0015	0.0020	0.0025	0.0035
5.0	0.0025	0.0025	0.0015	0.0030
6.0	0.0025	0.0025	0.0010	0.0030

Data analysis

From the results (Table 8, 9 and 10), **as concentration of standard solutions increases, absorbance increased** because light passing through solution interacts with more light-absorbing molecules of solute.[44] Thus, all calibration curves were straight lines with positive slope (followed Beer-Lambert's law). The general trend (Table 27, 28 and 29) noticed was that as **pH increased from 3.0 to 6.0 concentration of each heavy metal in the solution decreased**. In coal ash, due to **chemisorption**, positively charged metal ions are chemically bonded to the surface of coal ash particles.[45] Hence, result obtained can be because when H^+ ion concentration increased, metal ions were released into the solution due to displacement from their complexes by H^+ ions. This aligns with the conclusion in "**Influence of pH on leaching behavior of inorganic components from municipal solid waste APC residues**"[46] that as pH increases leaching of inorganic ions like Cr^{2+}, Cu^{2+} and Mn^{2+} decreases. Moreover, **over 4 weeks, concentration of heavy metals increased**. This might be because conditions of the solution became better reducing agents over time which cause metal ions associated with complexes in coal ash to dissolve more.[47] Hence, lowest level of heavy metal concentration was in sample with pH 6.0 after week 1 (chromium: 1.13mg/L copper: 0.728mg/L manganese: 0.155mg/L) and highest was in sample with pH 3.0 after week 4

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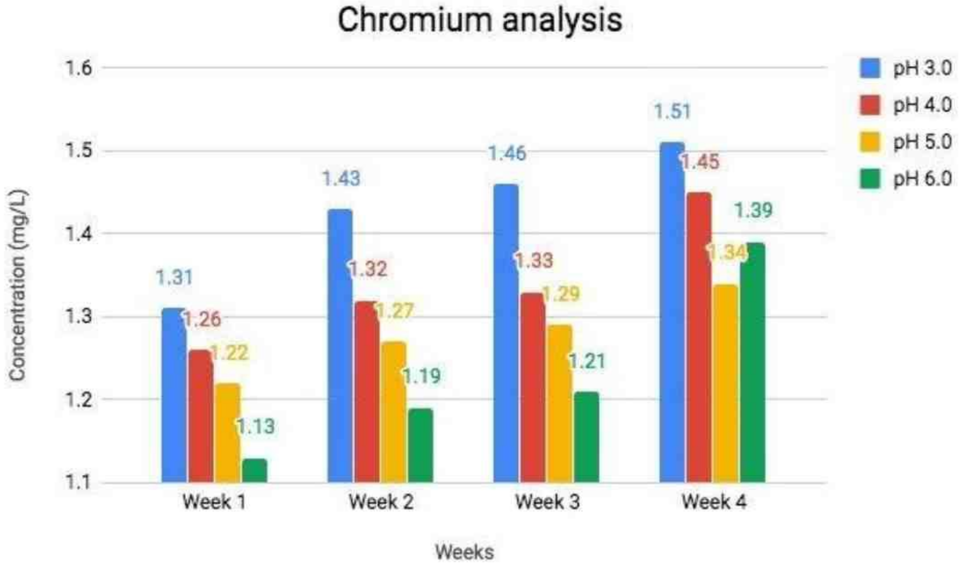
[44] *Kerner, Nancy. "Experiment II - Solution Color, Absorbance, and Beer's Law." Umich.edu. 25 Feb 2013. Web. 28 Dec. 2018. http://umich.edu/~chem125/softchalk/Exp2_Final_2/Exp2_Final_2_print.html.*

[45] Huang, Kai, Katsutoshi Inoue, Hiroyuki Harada, and Hidetaka Kawakita. "Leaching of Heavy Metals by Citric Acid from Fly Ash Generated in Municipal Waste Incineration Plants." *Researchgate.net*. Journal of Material Cycles and Waste Management, Aug. 2011. Web. 28 Dec. 2018. <https://www.researchgate.net/publication/227117364_Leaching_of_heavy_metals_by_citric_acid_from_fly_ash_generated_in_municipal_waste_incineration_plants>

[46] *Quina, Margarida J., João C.M. Bordado, and Rosa M. Quinta-Ferreira. "The Influence of PH on the Leaching Behaviour of Inorganic Components from Municipal Solid Waste APC Residues." Waste Management (2009). Web.28 Dec. 2018. https://www.ncbi.nlm.nih.gov/pubmed/19545989*

[47] Kedziorek, Monika A.M., Fabrice Compère, Alain Dupuy, and Alain C.M. Bourg. "CHAPTER 7 Heavy Metal Leaching from Contaminated Soils during the Percolation of EDTA: Observations and Modeling." *Applications of Chelating Agents for Land Decontamination Technologies*. American Society of Civil Engineers (ASCE), 2012. 171-97. *Researchgate.net*. The American Society of Civil Engineers (ASCE), 2012. Web. 28 Dec. 2018. <https://www.researchgate.net/publication/236002844_Heavy_Metal_Leaching_from_Contaminated_Soils_during_the_Percolation_of_EDTA_Observations_and_Modeling>

(chromium: 1.51 mg/L copper: 0.980mg/L manganese: 0.256mg/L). Moreover, **"Hazardous solid waste testing: First conference"**[48] mentioned that leaching potential of chromium, copper and manganese increases with increase in contact time which supported the result obtained.



Graph 4: Bar chart displaying trend in the concentration of chromium in the samples with increasing pH over 4 weeks

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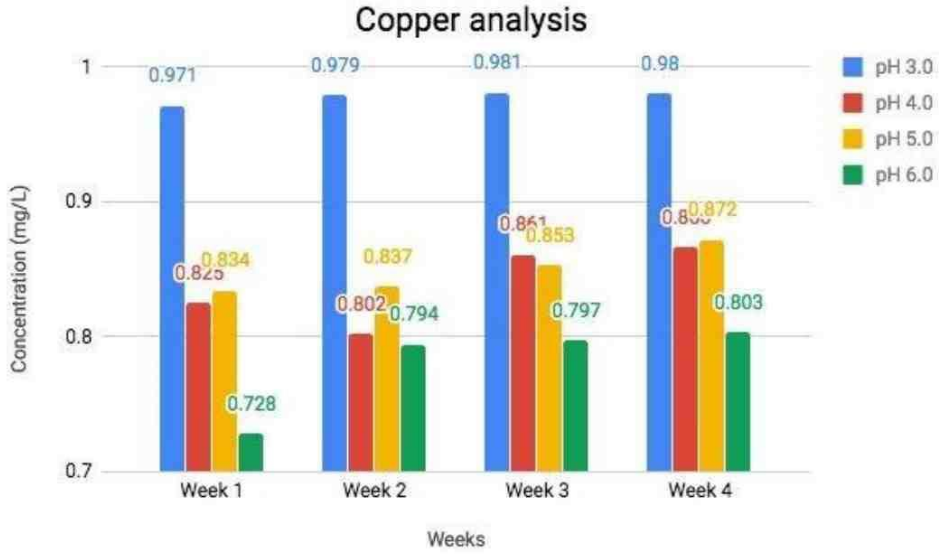
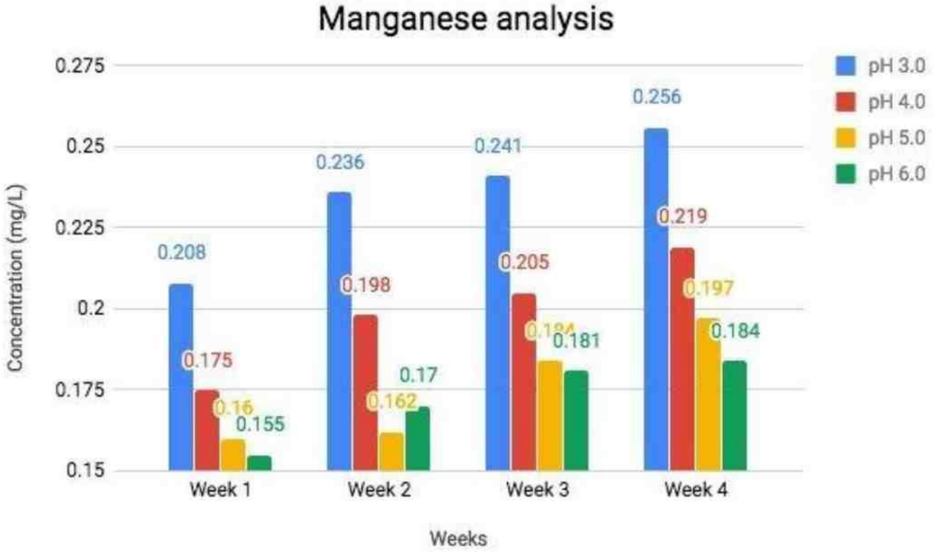
[48] *Conway. "Investigation of the Effect of Contact Time." Hazardous Solid Waste Testing: First Conference. By B.C. Malloy. Place of Publication Not Identified: American Society for Testing & Materials, 1981. 69-70. Web.31 Jan. 2019.*
https://books.google.co.in/books?id=JlEfQr9xo34C&pg=PA69&lpg=PA69&dq=increase+in+the+leaching+of+heavy+metals+with+increase+in+contact+time&source=bl&ots=wRktsh-1cl&sig=ACfU3U2ADwYixRf_I0WmcED4hv127QTJiw&hl=en&sa=X&ved=2ahUKEwiup_SyhJDgAhUdS48KHRKyBZQ4ChDoATADegQIBxAB#v=onepage&q=increase%20in%20the%20leaching%20of%20heavy%20metals%20with%20increase%20in%20contact%20time&f=false



Graph 5: Bar chart displaying trend in the concentration of copper in the samples with increasing pH over 4 weeks



Graph 6: Bar chart displaying trend in the concentration of manganese in the samples with increasing pH over 4 weeks

In part 2 (Table 34), it was noticed that **with increase in pH, concentration of copper and manganese in leachate decreased**. In the leachate, concentration of chromium at pH 3.0 to 6.0 decreased from 1.28mg/L to 1.13mg/L, of copper 0.762mg/L to 0.619mg/L and of manganese 0.210mg/L to 0.178mg/L. Moreover, **concentration of chromium, copper and**

manganese in each sample was more than their concentration in each leachate which suggests retention of some heavy metals by soil. This might be because of presence of organic and inorganic ligands that form stable complexes with metal ions, indicating extended soil pollution.[49] This aligned with the discussion in "**Impact of coal-powered electrical plants and coal ash impoundments on health of residential communities**"[50] that in addition to damage to atmosphere caused by smoke released by coal power plants, toxic waste in form of coal ash is of concern too.

In acid rain scenario (pH 3.0), percentage rise in leaching from week 1 to week 4, considering week 1 as base, for each heavy metal was:

$$\text{Percentage rise } = \frac{\text{final concentration} - \text{initial concentration}}{\text{initial concentration}} \times 100\%$$

Chromium: percentage rise = $\frac{1.51-1.31}{1.31} \times 100\% = 15.3\%$

Copper: percentage rise = $\frac{0.980-0.971}{0.971} \times 100\% = 9.27\%$

Manganese: percentage rise = $\frac{0.256-0.208}{0.208} \times 100\% = 23.1\%$

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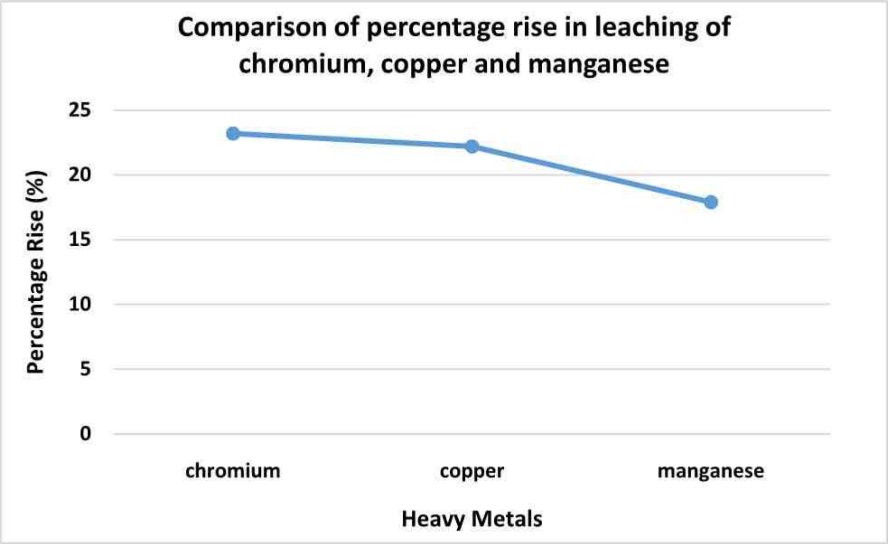
[49] Wuana, Raymond A., and Felix E. Okieimen. "Heavy Metals in Contaminated Soils: A Review of Sources, Chemistry, Risks and Best Available Strategies for Remediation." *Hindawi.com*. Hindawi, 23 Aug. 2011. Web. 30 Dec. 2018. <https://www.hindawi.com/journals/isrn/2011/402647/>.

[50] *Kravchenko, Julia, and H. Kim Lyerly. "The Impact of Coal-Powered Electrical Plants and Coal Ash Impoundments on the Health of Residential Communities." North Carolina Medical Journal. NCMJ, Sept.-Oct. 2018. Web. 28 Dec. 2018. http://www.ncmedicaljournal.com/content/79/5/289.full*

[redacted]



Graph 7: Percentage rise in leaching from week 1 to week 4 at pH 3.0

It was seen that **percentage rise in leaching was highest in manganese (23.1%) and lowest in copper (9.27%)**. Copper (a transition element) forms coordinate complexes with strong electrostatic attraction between electron pairs of ligands and central metal atom. High energy is required to separate central metal atom (copper) thus, taking more time to leach.[52] While, manganese leached maximum as low pH results in increase in solubility of Mn. According to, "**Mutual Cu, Fe and Mn solubility control under differentiated soil moisture status**" manganese becomes highly soluble due to its redox mechanism and produces *black water*. In acidic medium, insoluble forms of manganese (MnO_2, $MnOOH^{3+}$) can transform into soluble forms (Mn^{2+}) due to multiple oxidation states: [53]

$$MnO_2 + 4H^+ + 2e^- \rightarrow Mn^{2+} + 2H_2O$$

$$MnOOH^{3+} + 3H^+ + 4e^- \rightarrow Mn^{2+} + 2H_2O$$

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[52] *Brown, Catrin, and Mike Ford.Chemistry: Higher Level: Supporting Every Learner across the IB Continuum. Harlow, Essex: Pearson Education Limited, 2014. 139-209. Print.*

[53] *Diatta, Jean B. "MUTUAL Cu, Fe AND Mn SOLUBILITY CONTROL UNDER DIFFERENTIATED SOIL MOISTURE STATUS."Original Papers (2008): Pg 486. Web. http://www.uwm.edu.pl/jold/poj1342008/jurnal-01.pdf*

According to WHO, if manganese concentration in water is above 0.1mg/L[54], manganese (II) can oxidize and precipitate to form a black layer in water pipelines. This oxidation changes the taste of water and stains clothes if used in laundry.

It was observed that **soil retained certain amount of heavy metals as there was a difference in concentration of each heavy metal in the sample and the leachate** (Table 34). Hence, in acid rain scenario (pH 3.0) percentage concentration of each heavy metal in soil:

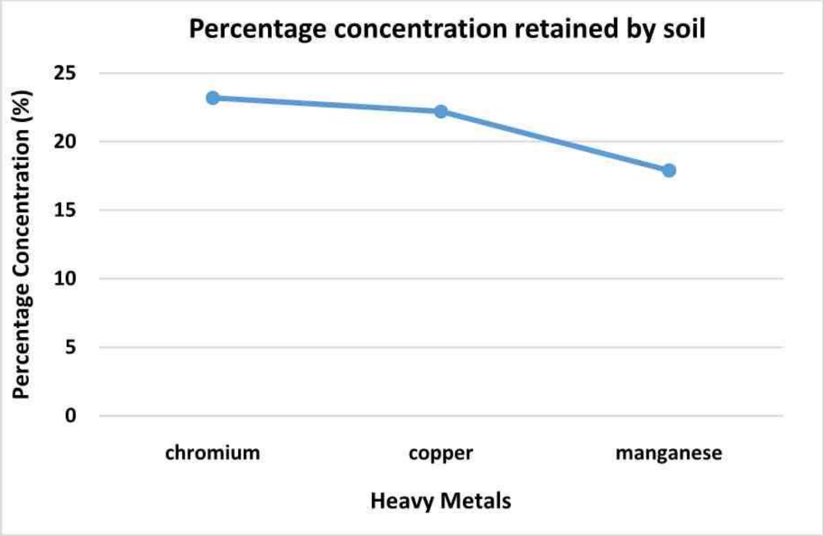
Percentage concentration retained by soil

$$= \frac{\text{concentration in sample} - \text{concentration in leachate}}{\text{concentration in sample}} \times 100\%$$

Chromium: Percentage concentration retained by soil $= \frac{1.51 - 1.16}{1.51} \times 100\% = 23.2\%$

Copper: Percentage concentration retained by soil $= \frac{0.980 - 0.762}{0.80} \times 100\% = 22.2\%$

Manganese: Percentage concentration retained by soil $= \frac{0.256 - 0.210}{0.256} \times 100\% = 17.9\%$



Graph 8: Percentage concentration retained by the soil at pH 3.0

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[54] *WHO."Manganese in Drinking-water."Background Document for Development of WHO Guidelines for Drinking-water Quality (1984): Pg2.Web.* https://www.who.int/water_sanitation_health/dwq/chemicals/manganese.pdf

Further, it was observed that **chromium was retained most by soil (23.2%) and manganese least (17.9%).** According to, "**Relationship between iron and trace level fractionation in soil**", chromium has tendency to form complexes with organic matter found in soil due to electrochemical properties like electronegativity and ionic potential. Also, chromium has affinity for iron oxides, thus, its presence in soil can be a reason for chromium retention[55] whereas the reason for lower retention of manganese can be because of its high solubility in water which allows it to flow away.

Evaluation

The study aimed **at finding the effect of pH and contact time on leaching potential of heavy metals** and the results obtained aligned with conclusions of various scholarly papers[56,57,58,59,60]. It was observed that as **pH decreased** (from 6.0 to 3.0), i.e. as H^+ ion concentration increased and as **contact time increased** (from week 1 to week 4), **leaching potential** of heavy metals (chromium, copper and manganese) **increased** along with **increase in heavy metal concentration in leachate** obtained from soil prototype. Moreover,

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[55] Sipos, Péter, Chung Choi, TiborNémeth, ZoltánSzalai, and TerézPóka."Relationship between Iron and Trace Level Fractionation in Soil."Chemical Speciation & Bioavailability (2014).Web. https://www.tandfonline.com/doi/abs/10.3184/095422914X13887685052506

[56] Quina, Margarida J., João C.M. Bordado, and Rosa M. Quinta-Ferreira."The Influence of PH on the Leaching Behaviour of Inorganic Components from Municipal Solid Waste APC Residues."Waste Management (2009).Web.28 Dec. 2018. https://www.ncbi.nlm.nih.gov/pubmed/19545989https://www.ncbi.nlm.nih.gov/pubmed/19545989

[57] Conway. "Investigation of the Effect of Contact Time." Hazardous Solid Waste Testing: First Conference. By B.C. Malloy. Place of Publication Not Identified: American Society for Testing & Materials, 1981. 69-70. Web.31 Jan. 2019. https://books.google.co.in/books?id=JlEfQr9xo34C&pg=PA69&lpg=PA69&dq=increase+in+the+leaching+of+heavy+metals+with+increase+in+contact+time&source=bl&ots=wRktsh-1cl&sig=ACfU3U2ADwYixRf_I0WmcED4hv127QTJiw&hl=en&sa=X&ved=2ahUKEwiup_SyhJDgAhUdS48KHRKyBZQ4ChDoATADegQIBxAB#v=onepage&q=increase%20in%20the%20leaching%20of%20heavy%20metals%20with%20increase%20in%20contact%20time&f=false

[58] Kravchenko, Julia, and H. Kim Lyerly."The Impact of Coal-Powered Electrical Plants and Coal Ash Impoundments on the Health of Residential Communities."North Carolina Medical Journal. NCMJ, Sept.-Oct. 2018. Web. 28 Dec. 2018. http://www.ncmedicaljournal.com/content/79/5/289.full

[59] Diatta, Jean B. "MUTUAL Cu, Fe AND Mn SOLUBILITY CONTROL UNDER DIFFERENTIATED SOIL MOISTURE STATUS."Original Papers (2008): Pg 486. Web. http://www.uwm.edu.pl/jold/poj1342008/jurnal-01.pdf

[60] Sipos, Péter, Chung Choi, TiborNémeth, ZoltánSzalai, and TerézPóka."Relationship between Iron and Trace Level Fractionation in Soil."Chemical Speciation & Bioavailability (2014).Web. https://www.tandfonline.com/doi/abs/10.3184/095422914X13887685052506

manganese showed highest percentage rise in leaching over 4 weeks at pH 3.0, i.e. acid rain scenario while copper showed lowest percentage rise under same conditions. Then, in acid rain scenario, chromium was retained the most by soil while manganese the least. Using a soil prototype, that resembled the actual soil structure, helped in analyzing the heavy metal retention in soil. Further, the time period of 4 weeks was used to effectively measure the change in heavy metal retention over a month. And the pH 3.0 and 6.0 were used in the part 2 of the experimentation, as one represented the acid rain scenario whereas the other was close to the optimum rain pH i.e. 5.6.

To verify if the results obtained are faulty or not, standard deviation of absorbance was calculated which ranged from 0.180 to 0.201 for chromium; 0.363 to 0.463 for copper and 0.148 to 0.387 for manganese (Table 32, 33 and 34). As standard deviations were not very large, it was concluded that their effect on results was negligible.

Uncertainty analysis was done for concentrations of chromium, copper and manganese. The uncertainty in concentration was in range of 0.0010 to 0.0045 for chromium, 0.0010 to 0.0090 for copper and 0.0005 to 0.0035 for manganese (Table 35, 36 and 37). As these were minute too, their effect was assumed to be insignificant on the final results.

Conclusion

The investigation **aimed at finding change in leaching potential of heavy metals with change in pH and contact time**, supported the actual hypothesis as it was found that **leaching potential increases with decrease in pH and increase in contact time**. Moreover, samples with **pH 3.0 (acid rain scenario) showed an increment in leaching into groundwater**. The reason for this was that **when H^+ ion concentration increased**, chromium, copper and manganese ions that were **chemically bonded to the surface of coal ash** (chemisorption) were **released due to displacement by H^+ ions**. Hence, desorption of heavy metal cations increased along with their concentration in samples and leachate.

Chromium, copper, and manganese not only leached into underground water but also got retained by soil (Table 34, Page 33), thus, heavy metals from coal ash pollute both underground water and soil. Retention by soil might be due to stable complexes formed by heavy metal cations with organic and inorganic ligands. Moreover, calculations showed that **chromium was retained the most** due to its electrochemical properties causing complexes formation with organic matter, whereas **manganese was retained the least** due to its multiple redox mechanisms that make it highly soluble. Also manganese leached most over time while copper the least, because of manganese's high solubility while copper formed coordinate complexes with strong electrostatic attraction so took longer to leach. Presence of heavy metals in soil makes growing conditions for plants toxic, as they absorb polluted groundwater and bioaccumulation of heavy metals may take place. When animals and humans consume these plants, heavy metals migrate from producers to consumers; which is a threat to human and animal health. In this way, trace level heavy metals start from producers and reach tertiary consumers in food chain (Figure 3), thus making thermal power plants pollution emanating industries. Hence, a shift of world pollution focus from chimneys to underground water is required. So that water pollution can be controlled without affecting the power plants production.

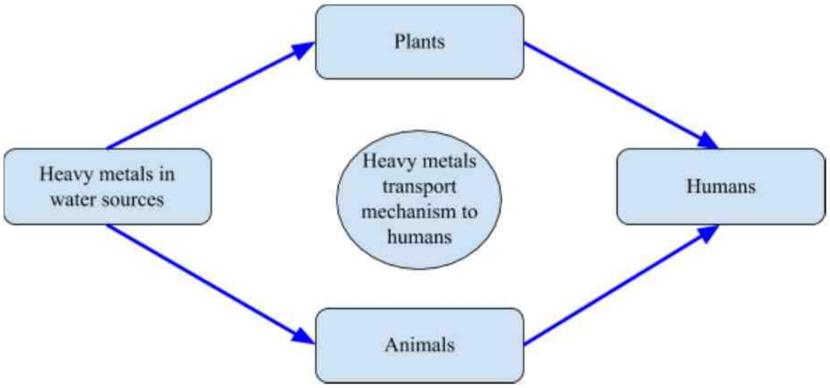

Figure 3: Flow chart showing migration of heavy metals from water sources to humans

Limitations and improvements

The sample of coal ash obtained had big chunks of coal that were removed by hand, leaving relatively same size particles. However, there still might have been some big particles left which were difficult to remove as compared to other finely divided particles. These might cause an error in the final readings. Hence, pestle and mortar was used to grind all big chunks into powder. Furthermore, coal ash was stored in a sealed container to avoid any contamination from dust particles.

During pre-lab for manganese analysis, no colour was produced when 1.0g potassium periodate was added in the solution while heating. Thus, 1.0g potassium periodate was added again and then a colour change was observed. Hence, in the actual lab 2.0g potassium periodate was used.

Further, another reason for error in part 2 was identified in pre-lab, as there was little difference in concentrations at pH 3.0 and 6.0, allowing pH 6.0 sample to flow right after pH 3.0 samples caused interference by existing heavy metals which could hinder final results. Hence, in the actual lab, before letting pH 6.0 samples flow through the earth prototype, distilled water was allowed to flow through twice to wash out any traces of previous sample.

The UV-vis spectrophotometer has a low sensitivity that might not detect very low concentrations of heavy metals. Hence, the scope of detecting a wider range of heavy metals was reduced (heavy metals which would have been in very low concentration). UV-vis spectrophotometers detects a broad band of light and other substances present in the solution might also absorb light and lead to false readings. To eliminate these limitations, more sophisticated instruments like **atomic absorption spectrophotometers** or **flame absorption spectrophotometers** can be used.

Instrumental errors in calibration of electronic balance or other devices that were used along with human errors could have hampered the readings. Hence, three trials were performed to eliminate random error but more trials can also be performed.

Future scope

Coal has many heavy metals, thus scope can be broadened by analyzing a wider range of heavy metals. Moreover, sensitive and sophisticated instruments like atomic absorption spectrophotometer can be used to ensure better results. Additionally, conditions affecting leaching like temperature can be varied to see the effect of global warming on leaching potential. The local crops grown in Kota, Rajasthan (or regions around other power plants in India) like **jowar** (English name: Sorghum/ Milo), **rice, wheat and oil seeds** can be analyzed for heavy metals to see the effect of leaching as the supposedly polluted underground water is the major source of irrigation for them.[61] The extent of migration of heavy metals in the food chain can also be seen, as manganese is used in photosynthesis and is useful for the plant to an extent[62], but the cell sap in leaves can be analyzed for the concentration of manganese that goes above the beneficial limit using sensitive techniques like **graphite furnace atomic absorption spectroscopy** or **inductively coupled plasma mass spectroscopy**.[63] Based on it a better technique for removal of heavy metals from coal, pre and post combustion, can also be devised.

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[61] *CENTRAL GROUND WATER BOARD."Ground Water Scenario of Kota District."DISTRICT GROUND WATER BROCHURE.GOVERNMENT OF INDIA, MINISTRY OF WATER RESOURCES, 2013.Web. 28 Dec. 2018.* *http://cgwb.gov.in/District_Profile/Rajasthan/Kota.pdf*

[62] *GAVALAS, NIKOS A., and HAROLD E. CLARK."On the Role of Manganese in Photosynthesis."On the Role of Manganese in Photosynthesis (1971): 139-43. Web.28 Dec. 2018* *https://www.ncbi.nlm.nih.gov/pmc/articles/PMC365827/*

[63] *Álvarez-Fernández, Ana, Pablo Díaz-Benito, AnunciaciónAbadia, Ana-FlorLópez-Millán, and Javier Abadia. "Metal Species Involved in Long Distance Metal Transport in Plants." Frontiers. Frontiers, 04 Mar. 2014. Web. 31 Dec. 2018.* *https://www.frontiersin.org/articles/10.3389/fpls.2014.00105/full*

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EE/RPPF

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Extended essay - Reflections on planning and progress form

Candidate: *This form is to be completed by the candidate during the course and completion of their EE. This document records reflections on your planning and progress, and the nature of your discussions with your supervisor. You must undertake three formal reflection sessions with your supervisor: The first formal reflection session should focus on your initial ideas and how you plan to undertake your research; the interim reflection session is once a significant amount of your research has been completed, and the final session will be in the form of a viva voce once you have completed and handed in your EE. This document acts as a record in supporting the authenticity of your work. The three reflections combined must amount to no more than 500 words.*

The completion of this form is a mandatory requirement of the EE. It must be submitted together with the completed EE for assessment under Criterion E. As per the 'Protocols for completing and submitting the Reflections on planning and progress form' section of the EE guide, a mark of 0 will be awarded by the examiner for criterion E if the RPPF is blank or the comments are written in a language other than that of the accompanying essay.

Supervisor: *You must have three reflection sessions with each candidate, one early on in the process, an interim meeting and then the final viva voce. Other check-in sessions are permitted but do not need to be recorded on this sheet. After each reflection session candidates must record their reflections and as the supervisor you must sign and date this form.*

First reflection session

Candidate comments:

As my father is an engineer in thermal power-stations, part of my childhood was spent in Tiroda, Maharashtra where there was a problem of deformed limbs in kids which was due to consumption of contaminated water that got me thinking how groundwater contaminates. From scholarly article in The Logical Indian, "In areas around Sterlite copper plant, groundwater contaminated with metals", I got to know that heavy metals from coal ash, in case of thermal power plants, contaminate groundwater by leaching into soil with rainwater. The research papers, "Heavy metals leaching in Indian fly ash" and "Effects of acid rainfall and heavy metals on forest ecosystem", led my research on the extent to which rain water's pH affects leaching potential of heavy metals like chromium. The challenge I faced was to find rainwater of different pH. Hence, as per "Transformation of heavy metals' speciation in soil receiving simulated acid rain"; I decided to adapt the method used to prepare artificial rainwater of required pH.

Date: 18th July'18

Supervisor initials:

Interim reflection

Candidate comments:

The background reading of my EE included "A Study on Acid Rain: Effects and Control Measures" and "Methods of Sampling and Test (Physical and Chemical) For Water and Wastewater" which provided the base for adapting the methodologies to prepare water samples of varying pH and to analyze the heavy metal concentration in waste water. While carrying out the experiment, I had to measure the absorbance of heavy metals like lead, nickel, arsenic, chromium, zinc, iron and copper. However, the final concentrations of nickel and arsenic turned out to be very less as compared to other heavy metals and it was doubtful whether there was an error in the values or the concentrations were actually very low. So I had to carry out the experiment again, particularly for nickel and arsenic to eliminate any doubt as my EE supervisor said that even slight errors can result in huge deviations. The second time I got some acceptable results, but I had difficulty in finding a way to present the results in a way that would make each step clear but also keep it concise. Finally I decided to make a tabular representation supported by the calibration graphs of each heavy metal. Although this process was time consuming the final outcomes were satisfactory.

Date: 10th Oct.'18

Supervisor initials:

Final reflection - Viva voce

Candidate comments:

My EE was directed towards determining the extent to which pH change caused by acid rain affects leaching potential of chromium, copper, and manganese over 4 weeks. Final results showed that heavy metal leaching potential increased with increase in pH and contact time. Moreover, concentration of heavy metals retained by soil indicated soil pollution. The completion of my EE was satisfying as I was introduced to extensive experimenting and my problem solving and time management skills were honed, starting from the pre-lab, to finally obtaining results that were in line with conclusions of scholarly papers and within limits of accuracy. The results would make people living around power plants aware of existing contamination and pollution, thus stirring them towards better methods of purification. Through further research, I discovered that heavy metal concentration in crops increases via bioaccumulation. Hence, with prior knowledge of my EE, I can lead my research further to determine the extent to which crops are affected by heavy metals as food crops are another direct source of heavy metals for humans.

Date: 9th Jan.'19

Supervisor initials: