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Determination of Some Selected Heavy Metals in Effluents from Lubricants Manufacturing and Associated Contamination of Oysters (*Crassostrea cucullata*) and Soil near Discharge Points in Mombasa, Kenya

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Abstract

Purpose: Available reports on heavy metal contamination along the Kenyan coastal zone focused on concentration levels in marine and coastal waters, sediments and biota, with indications of potential sources. However, information on contribution of specific sources to the heavy metal load into the marine and coastal environment is scanty or unavailable. Thus, this study addressed the need to assess specific potential sources and their contribution to heavy metal contamination. The generated data can be applied for monitoring discharge loads of heavy metals from lubricant manufacturing processes into the environment, its level of contamination to the environment and the potential risk to man through food chain.

Materials and Methods: Concentrations of selected heavy metals Cd, Cr, Cu, Pb, and Hg in effluents filtrates, residues (SPM), Oysters (*Crassostrea cucullata*) and soil samples were determined using ICP-OES. The procedures of the analytical methods and instruments used were validated with analysis of a multi-element certified reference material (CRM IAEA-452) for the elements Cd, Cr, Cu, Pb and Hg in the effluent samples and standard reference material (SRM 1566b) from National institute standards and technology for the elements Cd, Cr, Cu, Pb and Hg in the marine bivalve tissues. Physico – chemical parameters Temperature, P^H, Conductivity, Dissolved oxygen and Total organic carbon were also determined in the effluent samples. Sampling was done in the year 2021 and 2022. Effluent samples were collected from OLA energy Kenya limited, VIVO energy Kenya limited and TOTAL energy Kenya limited. Oyster samples and soil sediment samples were collected from Makupa creek. Data analysis was carried out using (SPSS) for Windows (Version 16) and one way analysis of variance (ANOVA) at 95% confidence level. The difference in mean concentrations of heavy metals within and between groups were considered significant at $P<0.05$. The interrelationships of heavy metals contamination in the samples were determined using the Pearson correlation coefficient. It was considered significant at P value <0.05.

Findings: This study showed presence of heavy metals that varied in concentrations in effluents samples, soils samples and Oyster samples. The mean concentrations of heavy metals in the effluents filtrates ranged as follows Pb = 0.0038±0.0004 mg/l – 0.0025 ± 0.003 mg/l, Cu = 0.0054±0.0038 mg/l – 0.0019±0.0002 mg/l, Cr = 0.0071±0.0021mg/l – 0.0045±0.0022 mg/l, Cd = 0.0041±0.0026 mg/l – 0.0013±0.0001 mg/l and Hg = 0.0063±0.0018 mg/l – 0.0026±0.0004 mg/l. The range of values for the mean concentrations of the heavy metals in the Oysters showed that Pb = 0.0227±0.0091mg/kg – 0.019 ± 0.006 mg/kg, Cu = 0.0618±0.0171mg/kg – 0.0601±0.0168 mg/kg, Cr = 0.0265 ± 0.0277 mg/kg – 0.0152 ± 0.0009 mg/kg, Cd = 0.0464±0.0282 mg/kg – 0.0273 ± 0.0117 mg/kg and Hg = 0.0264±0.0158 mg/kg – 0.025±0.0080 mg/kg. The range of values for the mean concentrations of the heavy metals in the soil samples showed that Pb = 0.0523 ± 0.0189 mg/kg – 0.0447±0.0181 mg/kg, Cu = 0.3010 ± 0.0282 mg/kg – 0.2437 ± 0.2690 mg/kg, Cr = 0.6564 ± 0.1468 mg/kg – 0.5862 ± 0.3073 mg/kg, Cd = 0.5434 ± 0.2207 mg/kg – 0.3506±0.2157 mg/kg and Hg = 0.2679 ± 0.1923 mg/kg – 0.2366±0.1584 mg/kg. The range values for the Physico-chemical parameters in the effluent samples were P^H = 7.31±0.198 – 6.53±0.459, Temperature = 27.53±0.378ºC – 23.3±0.2ºC, Dissolved oxygen = 6.81±0.191mg/l – 3.11±0.242 mg/l, conductivity = 2740.0±901.041 uS/cm – 1523.33±55.075 uS/cm, Total organic carbon = 15.18±1.2750 mg/l – 0.92±0.1026 mg/l. Concentrations of heavy metals were at safe limits set by WHO, US – EPA and NEMA (2017) except Hg in soil samples was slightly above permissible limits. Concentration of mercury was lower in effluent filtrates and effluents residues samples than in soil and Oysters samples.

Implications to Theory, Practice and Policy: This study recommends that frequent inspection be carried out on wastes emanating from industries, at its disposal sites and in living organisms at the vicinity of the wastes disposal sites. Industrialists should adopt best practices of wastes disposal and management to reduce emissions of harmful substances into the environment. Public education and awareness should be carried out to enlighten the residents about the impacts on health upon interaction with the polluted environment and feeding on organisms that reside in such contaminated environments.

Keywords: *Heavy Metals, Physico-Chemical Properties, Effluents, Lubricants, Soil, Crassostrea Cucullata.*

1.0 INTRODUCTION

Along with expanding urbanization and industrialization, environmental pollution which negatively affects the surroundings, has been rising quickly. As a result, induces heavy metal contamination which poses a serious threat to living organisms of aquatic and soil ecosystems (29). In developed countries such as India, industrialization played a significant impact on the development of the economies. Their effects were environmental pollution resulting from dumping of industrial wastes, home trash and synthetic agricultural inputs. Information on contribution of specific sources to the heavy metal load into the environment is scanty or unavailable. In developing countries such as Ghana, elemental analysis of heavy metals in soils around oil filling and service stations revealed a significant concentration of heavy metals such as Cr, Cu, Cd, Pb, Hg, Zn and Mn. This was attributed to common anthropogenic source such as the oil filling activities and Spillage of corroded vehicle engine oils during oil change (30).

Heavy metals are naturally occurring elements that have a high atomic weight and a density at least 5 times greater than that of water (19)(8). Human activities such as mining and smelting operations; industrial production and use; domestic, agricultural and technological applications have led to their wide distribution in the environment raising concerns over their potential effects on human health, wildlife, plants and the environment at large.

According to Don M. Pirro, Martin Webster and Ekkehard Daschner (2016) (3), a lubricant is a material that lowers friction between surfaces in contact, hence reducing the heat produced when the surfaces move. Ricardo Aucelio, Roseli Martins de Souza, Reinaldo Calixto de Campos and Norbert Miekeley (2007)(13) reported that a high-performance lubricant frequently contains additional components that have high concentrations of heavy metals like Pb, Cd, Cu, Cr, and Hg. Lubricants manufacturing processes such as handling of bulk raw materials, blending, testing of fresh lubes and used oils, packaging and warehousing are responsible for the generating effluents of which when discharged into the environment without proper monitoring becomes a source of environmental pollution (28).

At the economic growth rate of 10% desired in order to achieve Vision 2030, it is projected that lubricants and petroleum products annual consumption will grow from 4.5 million metric tonnes recorded in the year 2017 to 12 million metric tonnes by the year 2030. Because of this tremendous growth, more industrial effluents will be generated and if not properly managed they may end up in the environment. In the environment, heavy metals do not dissipate through microbial or chemical processes; as a result, they frequently bio-accumulate and spread throughout the food chain (6) (12). Heavy metals in edible aquatic organisms can directly threaten human health causing neurological, reproductive and respiratory illnesses. They can accumulate in important organs of human beings and animals causing serious health problems (10) (22) (24). Thus, there is need to assess specific potential sources and their contribution to the heavy metal contamination in the environment and provide appropriate recommendations for mitigation. Therefore, this study assessed the level of heavy metals concentration in the lubricants industrial effluents and associated contamination of Oysters (*Crassostrea cucullata*) and soil near discharge Points in Mombasa, Kenya.

2.0 MATERIALS AND METHODS

The Study Area

The main study sites were three leading lubricants manufacturing industries and the Makupa creek where effluents are discharged. The three industries are OLA Energy Kenya limited, VIVO Energy Kenya limited and Total energy Kenya limited. These three industries are located along Shimanzi - Kismayu circuit road near the Kenya Ports Authority (KPA) on Mombasa Island.

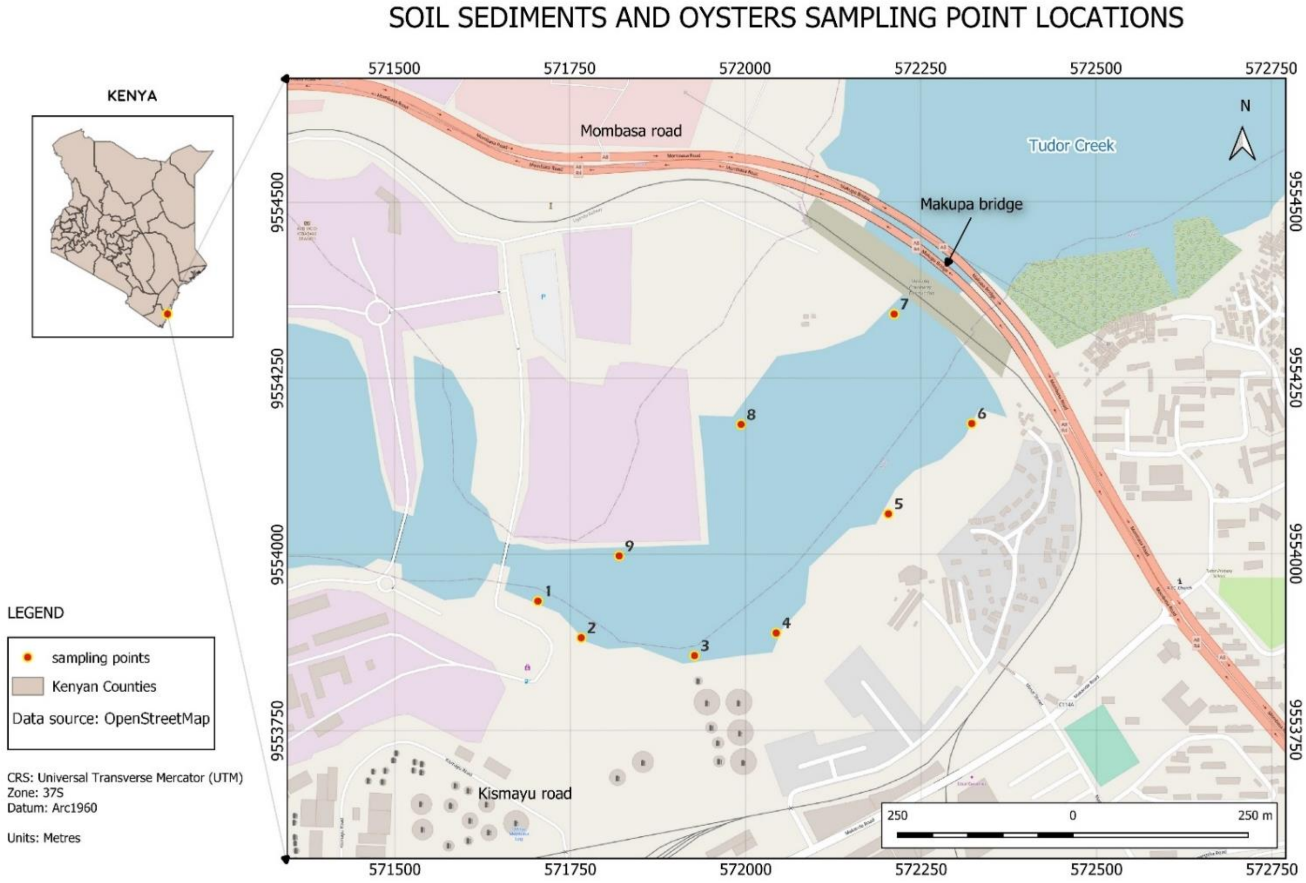

Fig 1: Geographical Map Showing Study Site

Samples Collection

Samples of the wastewater were taken from Total Energy Kenya Limited, OLA, and VIVO. A total of 18 effluent samples were collected and examined. Soil samples and oysters (*Crassostrea cucullata*) samples were collected from the Makupa Creek effluent discharge site. Eight oyster samples and a total of 18 soil samples were picked. At low tide, soil samples were taken at a depth of 20 cm and at a distance of 30 meters from each sampling point. Fewer samples of oysters were picked. This is due to their unavailability in the study location.

Samples Processing

Effluent Samples

Homogenized water samples were filtered using pre-weighed 0.4 µm pore size cellulose filter papers. A 25 mL of aliquot of each homogenized water sample was transferred to 100 ml Pyrex glass beakers. 2ml of 69% concentrated HNO_3 and 6ml of 30% concentrated HCl all of which are trace metal grade acids were added to acidify each sample. The mixtures were heated for 3 hours on a hot plate at 95^{o}C until the solution became clear and transparent. After cooling to room temperature 25^{o}C, each of the digested samples was transferred to a 50ml volumetric flasks. The samples were then brought to the 50ml mark with ultrapure water (25)

Soil Samples

10g of each soil sample was dried at 105^{o}C in an oven for 24 hours. The dried soil sample was cooled to room temperature at 25^{o}C, grounded, sieved and homogenized. 5g of each homogenized sample was transferred into the labelled volumetric flasks. 20ml of 65% concentrated nitric acid and 10ml of 70% perchloric acid of which all are trace metal grade were added in each sample for acid digestion. The acid digested samples were then heated for 1 hour at 120° C for hot plate heat digestion. The mixture was filtered using Whatman filter papers Merck, 0.45 μm. Deionized water was added to each filtrate to a final volume of 50 ml (4).

Oyster Samples

Each fresh Oyster sample was washed with tap water followed by distilled water and then immediately dissected. The soft tissues obtained were frozen at -40 °C for 12 hours. 10g of each frozen sample was dried at 105^{o}C ± 2^{o}C in an oven for 24 hours, cooled to room temperature at 25^{o}C, grinded to powder and sieved through a 250-micrometer sieve. The sieved particles were homogenized for 6 hours at a temperature of 25^{o}C and stored in the desiccator for 12 hours to avoid contamination with humidity. 5.0g of each of the homogenized sample was digested using a mixture of Nitric acid (70%, Spectrosol) and hydrogen peroxide (35%, Riedel-de Haen) prepared in a 1:1 (v/v) ratio followed by microwave digestion for 15 minutes at a temperature of 95^{o}C until a clear solution was obtained (21).

Physico – Chemical Parameters of Effluent Samples

Temperature, P^{H}, electrical conductivity and dissolved Oxygen were measured at the effluents collection site. Digital thermometer, portable P^{H} meter, Conductivity meter and a potable MW600 dissolved oxygen meter were used. Total Organic Carbon of the effluent samples was measured using TOC portable 450TOC 110/240VAC analyzer.

Analysis of Heavy Metals

After calibration. The sample solutions were aspirated using the ICP-OES 5110 from Agilent Technologies. Pb, Cu, Cd, Cr, and Hg concentrations in effluent samples, soil samples, and oyster samples were evaluated using test method US EPA 6010D. The average of each sample component was calculated after the samples were evaluated in triplicate. Additionally, the blank samples were used in triplicates (4)

Data Analysis

Statistical Package for Social Sciences (SPSS) for Windows (Version 16) and one way analysis of variance (ANOVA) at 95% confidence level was used to examine the statistical significance and differences in the mean concentration of heavy metals in the effluent samples, soil samples and Oyster samples. The difference in mean concentrations of heavy metals within and between groups were considered significant at $P<0.05$. The interrelationships of heavy metals contamination in the samples were determined using the Pearson correlation coefficient. The Pearson correlation coefficient was considered significant where the P value was <0.05 (26)

3.0 FINDINGS

Physico-Chemical Properties

Table 1: Mean and Standard Deviation of Physico-Chemical Parameters of Effluent Samples

Sample Location	Season	pH	Temp (°C)	Conductivity (µS /cm)	DO (mg/l)	TOC (mg/l)
WHO/US-EPA permissible limits		6.5-8.5	20-35°C	200 - 2500	4.0-6.0	≤0.1- 25
Vivo energy	Wet	6.95±0.1757	23.43±0.0577	1723.33±58.5932	3.17±0.4517	1.19±0.2650
	Dry	6.53±0.4590	27.53±0.3785	2275±30.6648	6.09±0.7308	12.82±1.5497
Total energy	Wet	7.31±0.1985	23.5±0.1	1563.33±66.5832	3.11±0.2421	0.92±0.1026
	Dry	6.78±0.0057	26.73±0.2081	2740.0±901.0416	4.89±1.3267	7.48±0.9360
Ola energy	Wet	7.6±0.2722	23.3±0.2	1523.33±55.0757	3.26±0.1913	1.17±0.1552
	Dry	6.84±0.0585	26.2±0.4	2548.33±859.2964	6.81±0.19 15	15.18±1.2750

The study's findings (table 3.1) revealed that the P^H, temperature, dissolved oxygen, and total organic carbon levels were within the acceptable limits established by the WHO and US-EPA throughout both the dry and wet seasons. During the dry season, a sample from Total Energy reported the highest electrical conductivity value (2740±901.0416 S/cm), which was above the permissible limits. Samples taken from different places revealed conductivity values that are within the acceptable range. The mobility and total concentrations of heavy metal ions released into the effluents are higher at high temperatures and low P^H. As a result, electrical conductivity increases (20)

Effluent Samples

Table 2: Mean and Standard Deviation of Heavy Metals in Effluents Filtrates Samples During Wet and Dry Season

	Parameter		**Cu(mg/l)**	**Cr(mg/l)**	**Cd(mg/l)**	**Pb(mg/l)**	**Hg(mg/l)**
	WHO/ EPA (mg/l)		10.0	1.6	0.8	2.0	0.05
Sampling location and seasons	Vivo energy	Wet	0.0019±0.0002	0.0071±0.0021	0.0014±0.0003	0.0032±0.0004	0.0026±0.0004
		Dry	0.0021±0.0006	0.0052±0.0015	0.0041±0.0026	0.0036±0.0017	0.0063±0.0018
	Total energy	Wet	0.0019±0.0005	0.0067±0.0011	0.0038±0.0006	0.0038±0.0004	0.0037±0.0003
		Dry	0.0022±0.0005	0.0045±0.0022	0.0029±0.0020	0.0027±0.0002	0.0062±0.0013
	Ola energy	Wet	0.0054±0.0038	0.0067±0.0010	0.0013±0.0001	0.0025±0.0003	0.0029±0.0005
		Dry	0.0021±0.0001	0.0059±0.0022	0.0029±0.0015	0.0033±0.0004	0.0045±0.0006

Both the wet and dry seasons, the concentration of heavy metals in the effluent filtrates samples was below the US-EPA and WHO-recommended levels. The highest value of 0.0071± 0.0021 mg/l for the concentration of Cr was found in the effluent samples from Vivo Energy during the wet season, followed by Total Energy and Ola Energy with values of 0.0067± 0.0011 mg/l and 0.0067 ± 0.0010 mg/l, respectively (table 3.2). The lowest Cd value was found in effluent samples from Ola Energy, at 0.0013 ± 0.001 mg/l. Generally, wet seasons had higher concentrations of heavy metals than dry seasons. This could be attributed to the increased rainfall that occurs together with surface runoffs during the wet season compared to the dry season (7)

Effluent Residues (SPM)

Table 3: Mean and Standard Deviation of Heavy Metals in Suspended Particulate Matter During Wet and Dry Season

	Parameter		**Cu(mg/kg)**	**Cr(mg/kg)**	**Cd(mg/kg)**	**Pb(mg/kg)**	**Hg(mg/kg)**
	WHO/ EPA (mg/l)		10.0	1.6	0.8	2.0	0.05
Sampling location and seasons	Vivo energy	Wet	0.002±0.001	0.0026±0.0020	0.004±0.0043	0.002±0.0017	0.004±0.0026
		Dry	0.005±0.0026	0.0036±0.0015	0.0036±0.0030	0.0033±0.0032	0.0016±0.0005
	Total energy	Wet	0.002±0.001	0.0046±0.0063	0.0086±0.0058	0.0023±0.0005	0.002±0.001
		Dry	0.0036±0.0028	0.0053±0.0035	0.0033±0.0005	0.0013±0.0005	0.0016±0.0011
	Ola energy	Wet	0.0046±0.0028	0.0033±0.0015	0.0036±0.0015	0.0016±0.0005	0.002±0
		Dry	0.0016±0.0005	0.0053±0.0030	0.003±0.002	0.0046±0.0037	0.0013±0.0005

In both the wet and dry seasons, the concentrations of heavy metals in the effluent residue samples were lower than those in the filtrate (table 3.3). This could be due higher solubility of heavy metal ions into the effluent water at high temperature and low PH. This results in the residues having a low release of heavy metals (23)

Soils

Table 4: Mean and Standard Deviation of Heavy Metals in Soils During Wet and Dry Season

Metals	**Cd (mg/kg)**	**Cr (mg/kg)**	**Cu (mg/kg)**	**Pb (mg/kg)**	**Hg (mg/kg)**
WHO/EPA (mg/kg)	0.8	1.6	10.0	2.0	0.05
Wet season	0.5434±0.2207	0.5862±0.3073	0.2437±0.2690	0.0447±0.0181	0.2366±0.1584
Dry season	0.3506±0.2157	0.6564±0.1468	0.3010±0.2820	0.0523±0.0189	0.2678±0.1923

The mean and standard deviation of the concentration of heavy metals in soil samples during wet season and dry season was found to be within the permissible limits of WHO and US- EPA except Hg (0.2366±0.1584mg/kg – 0.2678±0.1923mg/kg) which was slightly above the limit (table 3.4). The mean concentration and standard deviation for Pb was lowest in both wet and dry seasons (0.0447 ± 0.0181 mg/kg – 0.0523 ± 0.0189 mg/kg). The mean concentration of heavy metals was higher in soils than in effluent filtrate samples and effluent residues. This is in line with a research report by Kinuthia and his colleagues (2020) which explained that contaminated waste water can lead to a build-up of heavy metals in soils (4). Availability of heavy metals in soils is influenced by environmental conditions, industrial activities and the weathering processes of the underlying rocks (15)

Oysters

Table 5: Mean and Standard Deviation of Heavy Metals in Oysters During Wet and Dry Season

Metals	Cd(mg/kg)	Cr(mg/kg)	Cu(mg/kg)	Pb(mg/kg)	Hg(mg/kg)
WHO/FAO (mg/kg)	2.0	2.0	73.3	6.0	0.5
Wet season	0.0272±0.0117	0.0152±0.0009	0.0618±0.0171	0.019±0.0062	0.0264±0.0158
Dry season	0.0464±0.0282	0.0265±0.0277	0.0601±0.0168	0.0227±0.0091	0.025±0.0080

The mean and standard deviation of the concentration of heavy metals in Oyster samples during wet season and dry season was found to be lower than the concentration of heavy metals in soil samples but higher than in the effluents filtrates and effluents residues. Results demonstrate that small quantities of the metals are accumulated in Oysters tissues than in the underlying soils (18)

Discussion

Conductivity is a measure of the ability of aqueous solution to carry an electric current that depends on the presence and total concentrations of ions, their mobility, valance and on the temperature (20). Electrical conductivity values were found to be slightly above the WHO and US-EPA (200 µS/cm - 2500 µS/cm) acceptable limits. Samples from Total energy reported the highest mean value of electrical conductivity (2740±901.0416 µS /cm) during dry season. This indicates high mobility and greater concentrations of heavy metal ions in the samples. Temperature ranged from 27.53°C during dry season to 23.3°C during wet season. At higher temperature (25°C – 35°C) the release rates of metals ions in effluents is greater than at low temperature (5)

TOC values were higher during dry season than in wet season. It ranged between 15.18±1.2750 mg/l in samples from Ola to 12.82±1.5497 mg/l in samples from Vivo energy. These values are below the maximum allowable limit of 2000 mg/l (27). Presence of TOC could be attributed to the addition of biodegradable organic waste materials from terminal food canteens, retention of effluents in the interceptors and its slow flow rate out of the interceptors. This leads to high solubility of organic matter resulting to the increase TOC (17).

In comparison to the dry season, the pH value was greater during the wet season. The pH readings during the wet season ranged from 7.31 ± 0.1985 to 7.6±0.2722 and between 6.53±0.4590 to 6.84±0.0585 during the dry season. At higher temperatures, the effluent water readily dissociates into ions, resulting in more concentration of hydrogen ions in solution. This causes the effluents' pH to decrease (1).

The current study detected the presence of heavy metals in effluents, soils and Oysters samples. The mean concentration of heavy metals was higher in soils than in effluent samples and Oysters. Concentration of Cr was highest in soil during dry season while Cd was highest in the wet season. Environmental conditions, weathering processes of the underlying rocks, industrial activities and the remnants of heavy metal ions in the reclaimed Makupa dumpsite are associated with higher elemental concentrations in Makupa creek. This is in line with a research report by Kinuthia and his colleagues (2020) which explained that contaminated waste water can lead to a build-up of heavy metals in soils (4). Similar results were reported by Ann et al (2003)(2), Philip et al (2016)(11) and Mwatsahu et al (2020)(14) where heavy metals were detected in the soils samples and fauna samples from Makupa and Port-Reitz creeks.

4.0 CONCLUSION AND RECOMMENDATIONS

Conclusion

This study showed presence of heavy metals that varied in concentrations across the seasons in effluents, soils and Oyster samples. Concentration levels of heavy metals were higher in the soil and Oysters than in the effluent samples. Concentration of Hg in soil samples were slightly above the permissible limits recommended by WHO and US- EPA. This study therefore proposes that frequent inspection be carried out on wastes emanating from industries, at its disposal sites and in living organisms at the vicinity of the wastes disposal sites.

Recommendation

Industrialists should adopt best practices of wastes disposal and management to reduce emissions of harmful substances into the environment. Public education and awareness should be carried out to enlighten the residents about the impacts on health upon interaction with the polluted environment and feeding on organisms that reside in such contaminated environments.

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