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X-RFS and AAS Estimation of Heavy Metals and Trace Elements in Kenyan Cosmetic Clays

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Abstract

Heavy metals are natural components of the earth's crust. Some are essential in maintaining the metabolism of the human body, others are toxic even in small quantities while others are xenobiotics. Some communities in Kenya apply clay on their skins on several occasions like initiation or for cosmetic purposes. In this study, heavy metals like Cd, Pd, Si, Ti, V, and trace (micronutrients) element like Cu, Cr, P, K, Ca, Mn, Fe, Al, Ni and Zn were qualitatively and quantitatively estimated using X-ray fluorescence spectroscopy and flame atomic absorption spectrometry (FAAS) in 12 cosmetic clays used by some communities in Kenya. The results indicate that among the toxic heavy metals, none were exceeding the European Union (EU), Canada and Germany permissible exposure limits (PEL) fixed for cosmetic preparations. Fe was found in a higher concentration than Cu, Cr and Zn. The presence of trace elements can prove to be beneficial but the presence of toxic heavy metals in small amounts may have adverse effects on the consumer health that always use the cosmetic clays in an impression of being safe because of the natural origin. However, repeated exposure over a long period to poisonous heavy metals like cadmium and lead can pose a health risk. This study presents the status of heavy metals and trace elements in cosmetic clays.

Keywords: AAS, X-RFS, Heavy metals, Cosmetic clays

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1 Introduction

The existence of man and many other organisms are connected to clay similar to air and water. Clay has been largely utilized in daily life for example in cosmetics (Morekhure-Mphahlele, Focke, & Grote, 2017; Morekhure-Mphahlele et al., 2018; Mpuchane, Ekosse, Gashe, Morobe, & Coetzee, 2008; Thuadaij, Duangkham, & Hobanthad, 2020). Other fields are in industrial products utilizing clay raw materials, such as porcelain, various ceramic goods, plastics, rubber goods, innumerable sorts of paper, paints and varnishes where they act as a filler among other products that influence the environment of mankind (Konta, 1995). Evidence on human exploitation of clay dates back to the Middle Stone Age has was found at Klein Kliphuis, the Blombos Cave, Diepklo of Rock Shelter and the Sibudu Cave as cited by (Morekhure-Mphahlele et al., 2017). In Kenya several communities' still practice topical application of clays on skin for example the Maasai apply red ochre commonly known as *Ereko,* the Kalenjins and the Luhyas apply white clay during initiation period of boys into adulthood.

For clays to be suitable for pharmaceutical or cosmetic applications, they must comply with a number of chemical (stability, purity, chemical inertia), physical (texture, water content, particle size) and toxicological (toxicity, safety and microbiological purity) requirements. Specifically, they must have zero or very low toxicity (Mattioli, Giardini, Roselli, & Desideri, 2016). Metal contamination is one area of concern since soil and clay in general is a major sink of metal contaminants released into the environment by pedogenetic processes of weathering of parent materials at levels that are regarded as *trace* (<1000 mg kg^{-1}) and rarely toxic (R. Wuana, Okieimen, & Imborvungu, 2010) and anthropogenic activities (Kirpichtchikova et al., 2006).

Metals, a major category of globally-distributed contaminants, are natural elements that have been extracted from the earth and harnessed for human industry and products for a millennium (Zhang, Liu, & Wang, 2010). Metals are notable for their wide environmental dispersion from such activity; their tendency to accumulate in select tissues of the human body; and their overall potential to be toxic even at relatively minor levels of exposure (R. A. Wuana & Okieimen, 2011). Some metals, such as copper and iron, are essential to life and play irreplaceable roles in, for example, the functioning of critical enzyme systems. Other metals are *xenobiotics,* i.e., they have no useful role in human physiology and most other living organisms. As in the case of lead and mercury, may be toxic even at trace levels of exposure. Even those metals that are essential, however, have the potential to turn harmful at very high levels of exposure, a reflection of a very basic tenet of toxicology (R. A. Wuana & Okieimen, 2011)

Heavy metal contamination of clay may pose a risk to humans who use it. Many heavy metals are found in cosmetics as impurities due to their persistent nature though not acceptable ingredients, which is in contravention of the general prohibition found in section 16 of the Food and Drugs Act of 2006 (Sahu, Saxena, Johnson, Mathur, & Agarwal, 2014). Exposure to metals can occur through a variety of routes. Dermal exposure is the most significant route for cosmetic products (Sahu et al., 2014; Sainio, Jolanki, Hakala, & Kanerva, 2000). Involuntary oral exposure can occur for cosmetics used in and around the mouth, as well as from hand-to-mouth contact after exposure to cosmetics containing heavy metal impurities. Metals may be inhaled as dust or fume (tiny particulate matter, such as the lead oxide particles produced by the combustion

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of leaded gasoline) (R. Wuana et al., 2010). Once a metal is absorbed, it distributes in tissues and organs. Excretion typically occurs primarily through the kidneys and digestive tract, but metals tend to persist in some storage sites, like the liver, bones, and kidneys, for years or decades.

In this study, the heavy metal and trace elements composition of clay used as cosmetics from selected communities living in Kenya was analysed to inform on its use.

2 Materials and methods

2.1 Materials and sample preparation

Clay samples from three counties namely; Bomet, Bungoma and Narok counties were collected. Indigenous communities in these Counties apply clays on their skins on several occasions for different purposes and occasions. Collection of samples were from specific sites normally used by communities living in this region. A paint less shovel was used to dig out a sample and transferred to a labelled clear polyethylene bag. The clay samples were air-dried in shallow trays in well-ventilated area until a constant mass was achieve. Any clay clods present were broken, plant materials and any stones present were also removed. The dry clay samples were crushed using a mortar and pestle and sieved using a 2 mm sieve.

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3 Characterization

Fluorescence spectroscopic analysis was determined using a Philips PANAlytical axios, wavelength dispersive X-ray fluorescence (WDXRF) spectrometer. About 15 g of ≤ 2mm clay samples was weighed and subjected to further grinding and sieved at ≤ 250 μm. Exactly 10 g of the sample was weighed, 5 g of starch added, and the two were homogenized before being pressed hydraulically to form a sample disc. These pellets were then analysed directly to determine the spectrum of heavy metals present and their semi quantitative mass in percentage with the help of Minipal Minimate software.

Quantification of heavy metals was achieved by weighing 1 g of homogenized soil sample ≤ 2 mm into a 250 mL reaction vessel dried in an oven at a temperature of 105 °C. A digestion mixture (30 mL) made by mixing Nitric acid (HNO_3) and Hydrochloric acid (HCl) in the ratio of 1:2 was added. With the help of a hot plate in a fume chamber and the temperature of the contents was raised to 360^0 C for 30 minutes or just almost to dryness (Nnorom *et al*., 2005;

Omolaoye *et al*., 2010). The samples were cooled before addition of 10 mL distilled water followed by filtration to a 50 mL volumetric flask, and topped to the mark with distilled water ready for analysis. The heavy metal concentration were calculated with the help of a straight-line equation of standards. The absorbance of metal standards were determined in triplicates in ascending concentration order using an atomic absorption spectrophotometer (AAS). The standards used were prepared by diluting a 1000ppm stock solution of pure metal using the dilution formula $C_1V_1 = C_2V_2$ standards in the order of 1 ppm, 2 ppm, 3 ppm, 4 ppm, 5 ppm, 10 ppm, 15 ppm and 20 ppm or as per the sensitivity of the lamp used.

4 Results and Discussion

4.1 X-Ray Flourescence Spectroscopy Analysis

Fifteen oxides were identified from the cosmetic clay samples obtained from the three counties. Data obtained for semi quantitative compositions reported as mass percentage are given in tables 1, 2, and 3. Silicon oxide from cosmetic clay samples from Narok County had a mean of 46.00 ± 16.15, the lowest value was 31%, and the highest was 66%, TiO_2 ranged from 0.10 to 3.40% with a mean of 1.83 ± 1.47. Iron oxide had a mean of 19.10 ± 12.51 and a range of 1.70 to 28.80. The ZnO percentage composition was between the range of 0.01 and 0.03 with a mean of 0.02 ± 0.01. Copper oxide had a mean of 0.04 ± 0.02 and it ranged from 0.02 to 0.06. Cadmium oxide had a mean of 1.88 ± 0.88 and a range of 0.74 to 2.90, Cr_2O_3 ranged from 0.03 to 0.04 and a mean of 0.04 ± 0.01 sample N3 and N4 were below detectable levels. Lead oxide ranged from 0.02 to 0.07 and a mean of 0.05 ± 0.02.

In Bomet County, the cosmetic clay samples were silicaceous. Silicon oxide had the highest mean of 49.00 ± 13.64 and a range of 42 to 69 %. Titanium oxide was ranging at 1.10 to 1.50 with a mean of 1.28. Iron oxide had a mean of 12.16 ± 6.39 and a range of 4.44 to 19.70. Cadmium oxide was between 1.70 to 2.60 and a mean of 2.10 ± 0.39. Zinc oxide, Copper oxide, chromium oxide, and lead oxide had means below 1.00. The total mean of all oxides identified was 98.92 ± 2.78.

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Table 1: Qualitative Analysis of Element Oxides Present In Clays from Narok County Using X-RFS Technique

Sample code	Mass % compositions														
	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	V_2O_5	Cr_2O_3	MnO	Fe_2O_3	NiO	CuO	ZnO	CdO	PbO	Total
N1	31.00	35.00	BDL	BDL	0.03	3.40	0.10	0.03	0.07	28.80	0.03	0.05	0.02	0.74	0.07	99.3
N2	26.00	52.00	BDL	0.50	0.04	1.20	0.03	0.04	0.13	18.30	0.02	0.04	0.03	1.80	0.03	100.2
N3	25.00	66.00	0.40	4.00	0.10	0.10	BDL	BDL	0.02	1.70	BDL	0.02	0.01	2.90	0.03	100.3
N4	35.00	31.00	1.00	0.50	0.10	2.60	0.25	BDL	0.17	27.60	0.02	0.06	0.03	1.90	0.06	100.3
Mean±	29.25±	46.00±	0.70±	1.67±	0.07±	1.83±	0.13±	0.04±	0.10±	19.10±	0.02±	0.04±	0.02±	1.84±	0.05±	100.0
SD	4.65	16.15	0.42	2.02	0.04	1.47	0.11	0.01	0.07	12.51	0.01	0.02	0.01	0.88	0.02	0.45

Table 2: Qualitative Analysis of Heavy Metals Present In Clays from Bomet County Using X-RFS Technique

Sample code	Mass % compositions														
	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	V_2O_5	Cr_2O_3	MnO	Fe_2O_3	NiO	CuO	ZnO	CdO	PbO	Total
BT1	41.00	39.00	0.30	1.70	0.04	1.20	0.06	0.11	0.11	14.00	0.02	0.04	0.02	2.20	0.05	99.85
BT2	42.00	42.00	0.20	2.50	BDL	1.50	0.08	0.07	0.05	4.44	0.01	0.02	0.02	1.90	BDL	94.79
BT3	15.00	69.00	0.50	2.20	0.30	1.10	0.04	BDL	0.31	10.50	0.02	0.03	0.04	1.70	BDL	100.74
BT4	21.00	46.00	0.30	1.50	6.20	1.30	BDL	0.03	1.50	19.70	BDL	0.10	0.07	2.60	BDL	100.30
Mean	29.75	49.00	0.33	1.98	2.18	1.28	0.06	0.07	0.49	12.16	0.02	0.05	0.04	2.10	0.05	98.92
±SD	±13.79	±13.64	±0.13	±0.46	±3.48	±0.17	±0.02	±0.04	±0.68	±6.39	±0.01	±0.04	±0.02	±0.39	±0.00	±2.78

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Table 3: Qualitative Analysis of Heavy Metals Present In Clays from Bungoma County Using X-RFS Technique

Sample code	Mass % compositions															
	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	V_2O_5	Cr_2O_3	MnO	Fe_2O_3	NiO	CuO	ZnO	CdO	PbO	Total
9	21.00	59.00	0.30	0.75	1.20	1.90	0.05	0.04	0.15	13.10	0.02	0.10	0.06	2.20	BDL	99.87
10	21.00	60.00	0.60	0.86	1.00	1.90	0.04	0.04	0.10	11.80	0.04	0.10	0.04	1.90	BDL	99.42
11	22.00	22.00	BDL	0.52	0.86	1.60	0.06	0.07	0.21	26.00	0.06	0.20	0.09	1.90	BDL	75.57
12	26.00	41.00	0.20	0.57	0.92	1.50	0.10	0.03	0.25	27.40	0.02	0.20	0.08	1.60	BDL	99.87
Mean	22.50	45.50	0.37	0.68	1.00	1.73	0.06	0.05	0.18	19.58	0.04	0.15	0.07	1.90	0.00	93.68
±SD	±2.38	±17.94	±0.21	±0.16	±0.15	±0.21	±0.03	±0.02	±0.07	±8.26	±0.02	±0.06	±0.02	±0.24	±0.00	±12.08

Key: BDL means Below Detectable level.

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Silica (SiO_2) was also dominant in samples from Bungoma County with a mean of 45.50 ± 17.94 and a range of 22 to 60 %. Fe_2O_3 ranged between 13.10 to 27.40 and a mean of 19.58 ± 8.26. TiO_2 composition was between 1.50 to that of sample 9 and 10 of 1.90; its mean was 1.73 ± 0.21. CdO had a mean of 1.90 ±0.24 and a range of 1.60 to 2.20. CuO and ZnO had means below 1.00 while PbO were all below detectable levels in the clay samples. The total mean concentration was 93.28 ±12.08.

4.2 Quantitative Analysis of Heavy Metals in the Clays Using AAS

Absorbance of the Standards and clay samples was determined using a Thermo Ash Jarell 1978 AAS model. This was done by aspiration of the standards to the FAAS burner followed by the clay sample filtrates. Triplicate absorbance readings were made to increase accuracy of standards and a calibration curve plotted for each heavy metal using Ms. Excel 2010 software.

Triplicate absorbance readings of clay samples were used to determine the actual concentration of elements in ppm, using the linear regression equations obtained from calibration curves of the respective metals and data reported with standard deviation (SD).

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Table 4: Concentration in ppm of heavy metals of cosmetic clay samples from Narok County

Sample code	Cd	Cu	Fe	Zn	Pb	Cr
	Mean±SD	**Mean±SD**	**Mean±SD**	**Mean±SD**	**Mean±SD**	**Mean±SD**
N1	0.06±0.02	1.79±0.06	76.39±6.01	0.64±0.01	1.29±0.20	0.16±0.11
N2	0.07±0.01	1.73±0.00	143.61±7.00	2.52±0.09	2.28±0.18	2.92±0.06
N3	0.03±0.01	0.04±0.04	54.17±7.86	0.08±0.03	0.64±0.27	0.09±0.10
N4	0.09±0.01	0.47±0.04	173.75±4.64	2.63±0.04	0.82±0.37	0.06±0.06
Mean±SD	0.06±0.02	1.10±0.77	111.98±56.05	1.47±1.30	1.26±0.73	0.85±1.38

Among the heavy metals, data presented in table 4 reveals that iron had the highest concentration and varied from 54.17 ± 7.86 to 173 ± 4.64 and a mean of 111.98 ± 56.05. Zn had a mean of 1.47 ± 1.30 and ranged from 0.08 ± 0.03 to 2.63 ± 0.04. Cu and Pb had comparable means of 1.10 ± 0.77 and 1.26 ± 0.73. Cr varied from 0.06 ± 0.06 to 2.92 ± 0.06 and a mean of 0.085 ± 1.38. Cd had a mean lower than 0.10. Sample N3 registered the lowest concentrations of all the metals analysed.

Table 5: Concentration in ppm of heavy metals of cosmetic clay samples from Bomet County.

Sample code	Cd	Cu	Fe	Zn	Pb	Cr
	Mean±SD	**Mean±SD**	**Mean±SD**	**Mean±SD**	**Mean ± SD**	**Mean±SD**
BT1	0.03±0.01	0.11±0.04	98.61±6.36	0.35±0.02	0.35±0.18	0.32±0.06
BT2	0.03±0.01	0.02±0.04	12.92±0.95	0.07±0.02	0.70±0.18	0.08±0.03
BT3	0.06±0.01	0.13±0.00	80.56±4.81	1.42±0.05	0.58±0.20	0.19±0.10
BT4	0.12±0.02	0.19±0.00	62.50±7.22	2.07±0.06	0.35±0.18	0.32±0.11
Mean±SD	0.06±0.04	0.17±0.07	63.65±36.89	0.98±0.93	0.50±0.18	0.28±0.06

The contents of trace elements from Bomet County in the screened preparations as a mean of triplicate described in table 5 shows that iron had a high mean and standard deviation of 63.65 ± 36.89 and varied from 12.92 ± 0.95 to 80.56 ± 4.81. Sample BT3 had the lowest zinc concentration of 0.07 ± 0.02 while sample BT4 had the highest at 2.07 ± 0.06**.** Lead concentration ranged from 0.35 ± 0.18 to 0.70 ± 0.18 and a mean of 0.50 ± 0.18. Chromium varied from 0.08 ± 0.03 to 0.32 ± 0.11 and a mean of 0.28 ± 0.06, sample BT1 and BT 4 had comparable concentration of 0.32 ppm. Cadmium and Copper had means that were below 0.20.

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Table 1: Concentration in ppm of heavy metals of cosmetic clay samples from Bungoma County

Sample code	Cd	Cu	Fe	Zn	Pb	Cr
	Mean±SD	**Mean±SD**	**Mean±SD**	**Mean±SD**	**Mean±SD**	**Mean±SD**
BM1	0.09±0.03	1.60±0.00	36.11±4.81	0.74±0.08	0.58±0.10	0.54±0.06
BM2	0.06±0.01	1.69±0.04	43.06±9.62	0.53±0.04	1.17±0.44	0.48±0.00
BM3	0.01±0.01	4.29±0.06	130.83±6.88	0.76±0.04	1.40±0.30	0.96±0.10
BM4	0.04±0.01	4.68±0.13	94.44±4.81	0.75±0.03	2.11±0.53	0.74±0.24
Mean±SD	0.05±0.03	3.07±1.65	76.11±44.81	0.69±0.11	1.32±0.63	0.68±0.22

Iron was found to be high in Bungoma County at a mean of 76.11 ± 44.81 and it ranged from 36.11 ± 4.81 to 130.83 ± 6.88. Cu varied from 1.60 ± 0.00 to 4.68 ± 0.13 and a mean of 3.07 ± 1.67. Pb was from 0.58 ± 0.10 to 2.11 ± 0.53 with a mean of 1.32 ± 0.63. Zinc and Chromium had comparable means of 0.69 ± 0.11 and 0.68 ± 0.22 respectively. Cadmium registered a low mean concentration of 0.05 ± 0.03.

The heavy metal concentrations were subjected to ANOVA test to ascertain the significance levels and results reported in table 7. Copper was found to vary significantly at 0.010 ($p \leq 0.05$) in the three counties. However when counties were compared individually copper and lead significantly differ in Bomet and Narok at 0.021 and 0.042 ($P \leq 0.05$). The means of copper were subjected to kruskal wallis test to show how they varied and it was shown that Bungoma county had the highest copper concentration while Narok registered a large value for lead.

Table 7: Significance tests of heavy metals in clay cosmetic samples from Bomet, Narok and Bungoma Counties

Element	Sum of squares	Df	Mean square	F	Sig.
Cadmium	0.000	2	0.000	0.085	0.920
Copper	17.497	2	8.749	7.908	0.010
Iron	5037.349	2	2518.674	1.161	0.356
Zinc	1.227	2	0.613	0.717	0.514
Lead	1.669	2	0.834	2.593	0.129
Chromium	0.683	2	0.342	0.524	0.609

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The high concentration of lead in cosmetic samples from Narok County might be attributed to deposition of anthropogenic lead due to vehicular emissions since two of the samples were collected by the roadsides. Clay samples from Bungoma County were collected from swamps hence their probable source might be attributed to ground water that has percolated through rocks containing disseminated pyrite and chalcopyrite.

Table 8: A table showing standard maximum amounts of metals in cosmetics

Metal	European Union (EU) (ppm)	Canada (ppm)	Germany (ppm)
Cadmium	0.5	3	5
Chromium	0.1	-	-
Pb	0.5	10	0.1

The maximum amounts of some heavy metals in cosmetics has been brought forward in very few countries among them are the European Union, Canada and Germany.

Conclusion

Clay samples from three counties in kenya namely Bomet, Bungoma and Narok were analysed. These clays are usually used for topical application. XRFS analysis revealed high levels of SiO_2, Al_2O_3, and Fe_2O_3. There were no radioactive materials present. However, heavy metals i.e. Cu, Zn, Ni, Cd and Pb were shown to be present. The heavy metal contents were below permissible levels in almost all clays. However, it must be noted that repeated exposure over long period to these poisonous heavy metals e.g. Cadmium and Lead may pose a health risk. Cu, Cr, Fe, and Zn on the other hand are essential to the well-being of human health in trace amounts. The Fe_2O_3 high levels can also be advantageous as it blocks the harmful UV rays. Other minerals such as TiO_2, K_2O, and CaO, which are essential minerals for cosmetics field, were present in small amount.

Declaration of competing interest

The author declares that they is no known competing financial interests and personal relationships that could have appeared to influence the work reported in this paper.

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