Mineralogical and chemical characterisation of clayey soils used traditionally for cosmetic in Flagstaff, Eastern Cape South Africa

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Abstract

Clayey soil samples used traditionally for cosmetic purposes in Flagstaff were characterized mineralogically and chemically, in an attempt to appreciate their role in the applications for which they were used. The colour of each sample was identified using the Munsell Soil Colour Charts; whereas mineralogical analysis was conducted using X-ray diffraction (XRD); and chemical analysis was determined using X-ray fluorescence (XRF) spectrometry. The colour of the samples were light brownish to reddish. Mineral phases identified in the clayey soils included *kaolinite, illite, goethite, quartz, albite, anorthite and muscovite*. Chemical analysis revealed high concentrations of SiO_2, Al_2O_3, Fe_2O_3 and lower concentrations of K_2O, Na_2O, MnO, MgO, CaO and TiO_2. The colours of the clayey soils were justified by the presence of goethite identified in most of them. Goethite and kaolinite, Fe_2O_3 and TiO_2 could influence the sunscreen ability of the clayey soils. Elements such as Ca and Mg penetrate the skin during application and influence their supplementation which is beneficial to the skin.

Keywords: *Kaolinite, goethite, quartz, X-ray diffractometry, X-ray fluorescence spectrometry*

1. Introduction

The use of clays in cosmetics dates back to more than 10 000 years, where they were first used by Egyptian women for cleansing purposes and later for body beautification (Lambert, 2001). Today, natural clays form an important component of several modern cosmetic products.

Cosmetic products are substances designed to be placed in contact with external parts of the human body including the epidermis, hair system, nails and lips, or with the teeth and the mucous membrane of the oral cavity (Viseras *et al.,* 2007; Pallington, 1998). They have been used by man since time immemorial (Carretero, 2002; Lopez-Galindo *et al.*, 2007) and constitute a significant aspect of health and hygiene in some societies. Reasons for the use of cosmetics include cleansing, changing physical appearance, perfuming, correcting odour, protecting and keeping these parts of the human body in good condition (European Community Directive 76/68/ECC, cited in Lopez-Galindo *et al.,* 2007). In recent times, clays and clay minerals have been widely used in the cosmetic industry as thickeners and emulsion stabilizers (Elmore, 2003) and as adsorptive materials. Choy *et al.* (2007) have reported the use of clays as antiperspirants to give the skin opacity and remove shine or blemishes. There are also records of the sunscreen ability of clays (Hoang-Minh *et al.,* 2010). Modern cosmetic products in which clays have been used include solar protectors (ilmenite), toothpaste (calcite), creams, powders and emulsions (palygorskite, sepiolite, kaolinite and smectites), bathroom salts (halite), eye shades and lipsticks (micas) and deodorants (Carretero & Pozo, 2010). Clays are also of great benefit to beauty spas around the world as they are used to perform pelotherapy and mud therapy (Veniale *et al.,* 2007). Further to this, many clay minerals have been included in face masks and bath oils which are used in beauty spas to soothe and cleanse the skin (Gomes & Silva, 2007). Though different types of clays exist, the clays used for cosmetics vary depending on their structural characteristics (Carretero, 2002; Droy-Lefaix & Tateo, 2006; Gomes & Silva, 2007; Lopez-Galindo *et al.,* 2007). These groups of clays include the phyllosilicates and hydrous oxides.

Their specific properties such as high specific surface area, high sorption capacity and high cation exchange capacity have been positively exploited in the cosmetic industry by including some of these clays in products designed for absorption of toxins, grease and other unwanted substances from the skin (Carretero, 2002; Carretero *et al.,* 2006; Carretero & Pozo, 2009; Juch *et al.,* 1994). The hydrous oxides such as goethite (FeOOH) and hematite (Fe_2O_3) commonly known as yellow and red ochre respectively have also been widely used for cosmetic purposes especially in traditional African societies.

Whereas modern cosmetic products have been widely available to western and civilized societies, the less exposed and more culture-oriented communities in Africa and other developing countries in the world have relied on traditional methods to beautify their body. Although these indigenous groups may not have the scientific background related to the use of clays in cosmetics, they have through indigenous knowledge been able to identify clays that could be used for different cosmetic purposes. These communities use physical properties of clays like colour, texture, smell, taste and feel to identify clays which can be used to satisfy their cosmetic desires. Mpuchane *et al.* (2008) highlighted the use of colour in the identification of traditionally used cosmetic clays in communities in Southern Africa. In the Eastern Cape Province of South Africa, different clays are still in use for cosmetic purposes and their colour play an important role in their identification. However, performance of clays in cosmetic products is justified not by their colour alone, but also by their physicochemical, mineralogical and chemical properties (Cara *et al.,* 2000; Lopez-Galindo *et al.,* 2007; Carretero & Pozo, 2009; Carretero & Pozo, 2010), which greatly depend on the structure of the clays (Carretero, 2002; Gomes & Silva, 2007). There is very limited documented research on the properties of traditionally used cosmetic clays in Eastern Cape Province. This study therefore aimed at mineralogically and chemically characterising clays used traditionally for cosmetic purposes in Flagstaff, Eastern Cape Province.

2. Materials and methods

2.1. Survey

A survey was conducted in Flagstaff to identify clayey soils used for cosmetic purposes in the area. Flagstaff (29^{o}29' E and latitude 31^{o}5' S) is found in the Oliver Reginald (OR) Tambo District in Eastern Cape Province of South Africa. Twenty one questionnaires were distributed in Flagstaff to obtain background information on the types of clayey soils used in the area and reasons for their application. Snowball sampling technique (Babbie & Mouton, 2005) was used to identify individuals in the study area to whom the questionnaires were administered. No more than five respondents were identified through any one initial subject. Purposive sampling (Babbie & Mouton, 2005) was then used to identify another initial subject who formed the initial respondent through whom others were identified.

The respondents identified eight different cosmetic clayey soils used for sunscreen and body beautification; and also provided the traditional name of each clayey soil type. Among the eight clayey soil types, three were used for sunscreen and five for body beautification. From the identified clayey soils, 12 representative samples (Table 1) were collected and analysed for their mineralogical and chemical properties. The samples were collected from areas identified by respondents (Table 1). All samples were sealed in air tight sampling bags, labelled 1 – 12 and transported to the laboratory for analyses.

Table 1: Traditional name, usage and source of cosmetic clayey soils in Flagstaff.

Sample No.	Sample Name	Sample Application	Area from where sample was collected
1	*Icumse*	Sunscreen	Yard
2	*Umthoba*	Body beautification	Roadside
3	*Umthoba*	Body beautification	Roadside
4	*Umkhuma*	Body beautification	Roadside
5	*Isibindi*	Body beautification	Roadside
6	*Ibomvu*	Sunscreen	Yard
7	*Imbola*	Sunscreen	Yard
8	*Umkhuma*	Body beautification	Roadside
9	*Isabunge*	Body beautification	Roadside
10	*Isabunge khaki*	Body beautification	Roadside
11	*Umkhuma*	Body beautification	Yard
12	*Ibomvu*	Sunscreen	Yard

2.2 Laboratory analyses

Three main analyses were conducted on the samples, and they included colour determination minerals identification and determination of major elements concentration. Prior to analyses, the samples were air-dried for 24 hours and gently disaggregated using an agate mortar and pestle (Tan, 1996; van Reeuwijk, 2002).

2.2.1 Clayey soil colour determination

Using a spatula a small amount of each sample was mounted on white cardboard sheets provided by the Munsell Colour Company Inc., MD 21218, USA. The samples were visually compared with the soil colours chips recorded in the Munsell Soil Colour Book to obtain their hue, value, chroma and colour (Munsell Soil Colour Book, 2002; Ekosse *et al.,* 2007).

2.2.2. Clay and non clay minerals identification

For identification of minerals, 1 g of each powdered samples was loaded in Al specimen holders and mounted in a Philips PW 1710 X-ray diffractometer system for qualitative identification of mineral phases contained in them (Nkoma and Ekosse, 1999). The Philips XRD equipment had a Cu-K_α radiation and a graphite monochromator, and was set to operate at 40kV and 45 mA. Samples were scanned at a speed of $1^o 2\ \theta$ / min; from $2^o 2\ \theta - 70^o\ 2\ \theta$. The raw data was captured with the aid of a PW 1877 Automated Powder Diffraction (APD) X'PERT Data Collector software package. A 2001 Version of the Philips X'PERT Graphics & Identify software package was used for qualitative identification of the minerals from both the data and patterns obtained. Diffractograms showing XRD peaks in each sample were constructed using Sigma Plots 11.0 Software. The mineral peaks produced by the software package were compared with those in the Mineral Powder Diffraction File Data Book ICDD (2002) for identification of clay and non clay minerals in each sample.

2.2.3. Major elements identification

In determining major elements in the clayey soils, 2.5 g of powdered samples were placed in 30 ml tarred porcelain crucibles and placed in an oven overnight at 700 ^{o}C. After cooling, they were placed in a furnace and heated at 900 ^{o}C for four hours. Each ignited sample and 2.4 g of $Li_2B_4O_7$ were placed in a flat bottomed Pt crucible to make beads at approximately 1200 ^{o}C in an oven. The beads were loaded on a Phillips X'Unique II XRF Spectrometer, and analysed for oxides of major elements including Si, Al, Fe, Mn, Mg, Ca, Na, K, Ti, P, Cr and Ni. The lower limits of detection were from 0.01 - 0.03 %.

3. Results

3.1 Colour of cosmetic clayey soils from Flagstaff

The samples had a variety of colours which included shades of red, yellow and brown (Table 2). Whereas the samples used for sunscreen were reddish in colour, those used for body beautification were yellowish and brownish. The hue was between 2.5YR and 10YR. Values ranged from five to seven; and the chroma ranged between four and eight. The values of samples used for body beautification were higher (6-7) than those used for sunscreen (5) (Table 2).

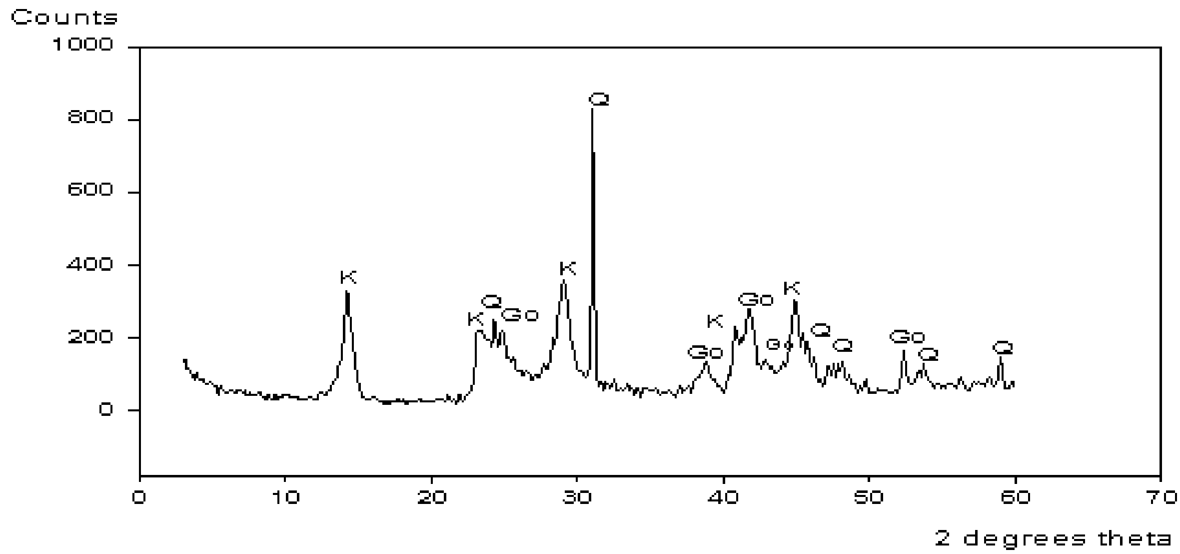
Table 2: Colour of cosmetic clayey samples from Flagstaff.

Sample no.	Sample name	Sample application	Hue/value/ chroma	colour
1	*Icumse*	Sunscreen	5YR / 5 / 8	Yellowish red
2	*Umthoba*	Body beautification	10YR / 6 / 8	Brownish yellow
3	*Umthoba*	Body beautification	10YR / 6 / 8	Brownish yellow
4	*Umkhuma*	Body beautification	7.5YR / 6 / 4	Light brown
5	*Isibindi*	Body beautification	5YR / 6 / 8	Reddish yellow
6	*Ibomvu*	Sunscreen	2.5YR / 5 / 8	Red
7	*Imbola*	Sunscreen	5YR / 5 / 4	Reddish brown
8	*Umkhuma*	Body beautification	2.5Y / 7 / 4	Pale yellow
9	*Isabunge*	Body beautification	7.5YR / 5 / 8	Strong brown
10	*Isabunge khaki*	Body beautification	7.5YR / 6 / 4	Light brown
11	*Umkhuma*	Body beautification	2.5Y / 7 / 4	Pale yellow
12	*Ibomvu*	Sunscreen	2.5YR / 5 / 8	Red

Note: YR = yellow-red; Y = yellow; and R = red.

3.2 Minerals identified by XRD in cosmetic clayey soils from Flagstaff

Mineral assemblages in the samples included kaolinite, $Al_2Si_2O_5(OH)_4$; goethite, $Fe^{+3}O(OH)$; muscovite, $KAl_2(Si_3Al)O_{10}(OH,F)_2$; illite, $(KAl_2(Si_3Al)O_{10}(OH)_2$; albite, $(Na,Ca)(Si,Al)_4O_8$; anorthite, $(Ca,Na)(Si,Al)_4O_8$ and quartz, SiO_2. Kaolinite and quartz were identified in all the samples, and goethite was detected in 50 % of the samples (Table 3). Representative X-ray diffractogram showing identified mineral peaks in the samples is shown on Figure 2.



Note: K = kaolinite; Go = goethite; Q = quartz

Figure 2: Representative X-Ray diffractogram of clayey soils used for sunscreen in Flagstaff.

Table 3: Minerals identified by XRD in cosmetic clayey soil samples from Flagstaff.

Sample No.	Sample Name	Primary minerals				Secondary minerals		Oxides
		Albite	Anorthite	Quartz	Muscovite	Illite	Kaolinite	Goethite
1	*Icumse*	-	-	√	-	-	√	√
2	*Umthoba*	√	-	√	-	-	√	√
3	*Umthoba*	√	√	√	-	-	√	√
4	*Umkhuma*	-	-	√	√	-	√	-
5	*Isibindi*	√	-	√	-	-	√	-
6	*Ibomvu*	-	-	√	-	-	√	√
7	*Imbola*	-	-	√	-	√	√	√
8	*Umkhuma*	-	-	√	√	-	√	-
9	*Isabunge*	√	-	√	-	-	√	-
10	*Isabunge khaki*	-	-	√	√	-	√	-
11	*Umkhuma*	-	-	√	√	-	√	-
12	*Ibomvu*	-	-	√	-	-	√	√

3.3 **Chemical** ***composition of cosmetic clayey soils from Flagstaff***

High concentrations of SiO_2, Al_2O_3 and Fe_2O_3 were detected in the samples (Table 4). The concentrations differed with the usage of the samples. Whereas SiO_2 was higher in samples used for body beautification, Fe_2O_3 was slightly higher in the samples used for sunscreen; and Al_2O_3 concentrations were slightly higher in samples used for sunscreen than in body beautification. MgO, CaO, K_2O, TiO_2, P_2O_5, Na_2O, MnO, Cr_2O_3 and NiO recorded varied wt %. Samples used for body beautification recorded higher wt % of MgO, CaO, K_2O and Na_2O than those used for sunscreen. P_2O_5, MnO, Cr_2O_3 and NiO were very low in all the (Table 4).

Table 4: Chemical composition of cosmetic clayey soil samples from Flagstaff (oxides wt %).

Sample No	SiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	Cr_2O_3	NiO	TOTAL	LOI
1	45.11	33.10	19.57	0.08	0.37	0.12	0.18	0.31	1.44	0.08	0.06	0.002	100.45	16.83
2	51.90	25.47	16.22	0.21	2.06	1.18	0.89	0.45	1.27	0.03	0.06	0.01	99.76	16.79
3	52.68	23.81	15.26	0.23	2.29	2.65	1.57	0.44	1.17	0.03	0.05	0.01	100.19	13.32
4	65.16	21.74	8.30	0.07	0.17	0.28	0.26	2.90	0.91	0.16	0.02	0.006	100.00	6.72
5	51.83	23.66	15.48	0.23	2.22	3.12	1.65	0.39	1.12	0.08	0.05	0.02	99.86	13.87
6	45.54	31.09	20.61	0.09	0.50	0.09	0.24	0.03	1.58	0.09	0.06	0.02	100.22	15.29
7	25.81	18.34	42.97	0.16	0.22	0.04	0.35	0.95	0.64	0.28	0.04	0.02	99.82	11.94
8	66.24	23.25	6.83	0.05	0.22	0.01	0.14	2.31	0.92	0.06	0.05	0.01	100.11	8.40
9	52.36	23.44	16.10	0.23	2.27	2.85	1.34	0.39	1.21	0.07	0.06	0.01	100.36	12.83
10	64.26	22.10	8.88	0.07	0.16	0.28	0.38	2.90	0.91	0.14	0.05	0.008	100.15	6.63
11	66.00	24.02	6.72	0.04	0.23	0.01	0.19	2.24	0.85	0.06	0.02	0.006	100.39	8.30
12	45.44	31.69	20.16	0.08	0.49	0.10	0.19	0.29	1.48	0.08	0.03	00.01	100.05	15.44

4. Discussion

4.1 Influence of mineralogical and chemical compositions of the clayey soils on sunscreen

Goethite identified in samples 1, 2, 3, 6, 7 and 12, may have formed from the hydratation of hematite, and may be responsible for the reddish and yellowish colour in the samples (Ekosse & Jumbam, 2010; Ekosse *et al.,* 2010; Ngole *et al.,* 2010). The presence of kaolinite in all the samples used for sunscreen may influence sunscreening as the small particle size of kaolinite provides a platform for scattering of ultraviolet (UV) radiation (Juch *et al.,* 1994), as well as a refractive index (RI) value of $n_\alpha = 1.55$ (mindat.org/min-2156.html) which enables it to shield the skin from the effects of UV radiation. Usage of kaolinite-rich clayey soils for sunscreen by the Pondo people from Flagstaff is common knowledge to other African tribes such as the Ilha women of Mozambique (Indwe, 2010).

High concentrations of Fe_2O_3 detected in the samples justify the presence of goethite which was identified in majority of them. Goethite may influence the sunscreening potential of the clayey soils due to the structural influence of Fe^{3+} in the absorption of photons unto a vacant orbital (Hoang-Minh *et al.,* 2010). Goethite also has RI value of $n_\alpha = 2.39$ (mindat.org/min-1719.html), which shows low levels of UV radiation transmission. The lack of Fe bearing minerals in samples 4, 8, 10 and 11 is justified by the low concentrations of Fe_2O_3 in them; and may also account for the reason why the clayey soils are not used for sunscreen in the study site. Usage of Fe-rich clayey soils for sunscreen has also been observed in other indigenous African communities such as the Himbas of Namibia; who are known for covering their entire bodies with reddish clay mixture (Namibia Direct, 2006).

Furthermore, TiO_2 detected in the clayey soils may also contribute to their sunscreening potential. Titanium dioxide has been reported to have RI value of $n_\alpha = 2.7$ (Hewitt, 1992; Carretero and Pozo, 2010), and as a result has been included in several modern sunscreen cosmetic products. Therefore the presence of TiO_2 together with Fe_2O_3 in the clayey soils can facilitate enable solar protection by the clayey soils when applied on the skin.

4.2 Influence of mineralogical and chemical compositions of the clayey soils on body beautification

Body beautification may be greatly influenced by colour of the clayey soils. The reddish, yellowish and brownish colours identified compare well with those of cosmetic clayey soils used in other parts of Southern Africa (Mpuchane *et al.,* 2008). The presence of Fe bearing minerals in the clayey soils justifies their reported hue (2.5YR and 10YR) and yellowish and reddish brown colours. Also the high values recorded by the clayey soils may contribute to bright colourful patterns which can be painted on different parts of the body to ensure beautification and decoration. The use of yellowish and reddish shades of clayey soils for body decoration is also reported among the Wodaabe tribe of Niger (Wood, 2000), the Samburu and Karo tribes of Ethiopia (Ettagale, 1999). These colours are also common colours of several modern cosmetic products such as face powders, lip glosses, blushers and eye shadows, which are meant to augment the beauty of the body.

The presence of quartz (SiO_2) in all the clayey soils can influence body beautification by the soils. Quartz is a hard mineral measuring 7 on the Mohr scale (Ngole *et al.,* 2010), and may cause the resulting clay mixture to be gritty and abrasive when smeared on the body. Beneficiation of the clayey soils through elimination of quartz before application on the skin is recommended. Furthermore, the hazardous or beneficial effects of the transfer of the identified chemical elements from the clay on the skin will depend on the concentration of the element in the clays and their frequency of application.

The chemical elements such as Ca and Mg in the clayey soils may also contribute towards beautifying the skin when used for cosmetic application. According to Carretero *et al.,* (2011), chemical elements can move from clay to the skin and vice versa, and such mobility of chemical elements from clay to the skin can improve the appearance and beauty of the skin (Szanto and Papp, 1998). In-vitro studies on percutaneous migration (Gomes and Silva, 2007; Veniale *et al.,* 2007; Tateo *et al.,* 2009; Carretero *et al.,* 2010) indicate that after 20 minutes of clay application, several chemical elements such as Ca and Mg cross the skin barrier (Tateo *et al.,* 2009). Calcium has been reported to facilitate skin cell renewal by stimulating protein kinase C enzyme which ultimately results in fresher looking skin (Meltdown, 2001). Calcium also stimulates the production of catalase and superoxide dismutase, which are the skin's age-protective and cancer-protective antioxidants (Meltdown, 2001). Magnesium also ensures skin care by penetrating the lower layers of the skin to heal damaged cells (LIVESTRONG.com, 2011). Applications of the clayey soils on the skin can thus ensure Ca and Mg supplementation which have beneficial effects on the skin.

5. Conclusion

This paper has presented the mineralogical and chemical characteristics of clayey soils used traditionally for cosmetic purposes in Flagstaff, South Africa. The clayey samples exhibited brownish, yellowish and reddish colours. The colours may have resulted from the presence of Fe bearing mineral goethite which was identified in majority of the samples. Mineral constituents of the samples included kaolinite, goethite, quartz, microcline, and micas. Whereas kaolinite was the most dominant clay mineral in the samples, quartz was the most dominant non clay mineral. SiO_2, Al_2O_3 and Fe_2O_3 were most concentrated in the samples.

With increasing quest for beauty on the one hand, and high levels of poverty in the Eastern Cape Province on the other hand, clayey soils from Flagstaff could gain recognition as alternative cosmetic products. Their mineralogical and chemical compositions can contribute to their sunscreening and body beautifying functions.

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