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Cosmetic Ability of a selected Soil with Special Reference to Cation Exchange Capacity, pH, and Specific Surface Area

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Abstract: *Traditionally used clay-soil for cosmetic and medicinal applications has been found in different parts of the world. In the present experiment, five such samples of soil traditionally used as hair cleaner are analyzed to evaluate their cosmetic ability. For this purpose, eighteen physical and chemical parameters with special emphasis on pH, cation exchange capacity and specific surface area which are very much responsible for cosmetic ability of a soil sample were determined. Three commercial samples of cosmetic clays were also analyzed for comparison. For further confirmation of the results of the above experiments FTIR, XRD, EDAX analyzes and local application experiment on skin was also done. It has been found from chemical analysis that the experimental soil samples belong to smectite and illite classes of cosmetic clays. The experimental soil samples have values of the cited parameters similar to those of the standard cosmetic samples. FTIR, XRD and EDAX analysis support the findings of the chemical analysis. It indicates that the experimental soil samples are suitable to be developed for use in cosmetic purposes.*

Keywords: Cosmetic ability; Hair cleaner; Human beauty resource; Human health; Nadka Hasa

Soil is one of the most important natural resources and it has some special properties which make it suitable to have definite uses in various applied fields. Soil consists of air, water, organic compounds, microorganisms, and lots of minerals and nutrients. Clay, silt and sand are the three types of soil particles based on size. Every component of soil has its own role to play. One of the key ingredients used in cosmetic products for body beautification, cleansing, detoxification, mineralization and ultraviolet (UV) protection is clay. The ability of the particles of a substance to keep skin oil free; repair the pores of skin or scalp by exposing to air and transforming essential nutrients and ions, offering other beneficial activities are collectively known as its cosmetic ability (Sarma and Rabha, 2013) It can absorb UV radiations of sunlight; can absorb contaminants such as toxic elements due to ion exchange capacity of clay particles and also can absorb unhealthy microorganisms. Consequently, the cosmetic property of soil depends upon the clay particle content. Due to cosmetic ability, some clay soils have traditionally used in cosmetic purposes worldwide. Some clays are commercially available for the skin treatment

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which can be classified into three major classes on the basis of cation exchange capacity (CEC). Cation exchange capacity is the capacity of soil to hold or exchange positively charged ions viz. Ca^{2+}, Na^{+}, Mg^{2+}, K^{+}, Al^{3+}, NH_4^{+} and H^{+} in the colloidal clay particles present in the soil. It indicates that CEC enables clay to attract and remove positively charged toxins from skin. As such, a higher value of CEC more is the ability of the clay to remove toxins from the skin where it is applied. The three classes of clay as cited above are smectite having the highest CEC in the range of 60-70 cmol (+) kg^{-1} followed by illite with CEC around 25-40 cmol (+) kg^{-1}, and kaolinite with the lowest CEC at 3 – 15 cmol (+) kg^{-1}. Commonly known commercial clays are branded as sodium bentonite, French green clay, "*Multani mitti*", (fuller's earth), *Rassoul clay* etc. belong to smectite group of clay minerals. Specific Surface Area (SSA) is defined as the total surface area per unit volume or per unit mass of a soil. Early research in this field showed that higher percentages of clay and organic matter enhance the value of CEC and SSA SSA (Matike *et al*., 2011).

Increasing amount of clay particles increases the soil SSA, which indicates more pores with higher water adsorption/ absorption capacity. Increase in clay percent increases soil moisture or soil humidity or soil water holding capacity. Soil contains a lot of minerals; as such the soil water actually behaves as electrolyte which carries a large number of ions. Human hair consists of tiny shafts. These are made up of scales, called cuticles. The pH of hair shaft is 3.67 and that of scalp is 5.5. Water and alkaline hair cosmetics open the cuticles and expose the interior of hair shaft. This can lead to adverse effect caused by harmful elements. As such, an alkaline cosmetic increases the negative electrical charge of the hair fibers which leads to increase of friction between hair fibers. Consequently, materials used as hair and scalp cosmetic are desirable to be of acidic pH (MFRG Dias *et al*., 2014). Thus, comparison of pH, CEC and SSA of a clay (soil) sample with some commercial clay cosmetics informs about the possibility of exploring the cosmetic ability of the experimental sample up to a considerable extent.

Fourier Transform Infrared Spectroscopy (FTIR), X-ray diffraction (XRD) and Energy Dispersive X-Ray Analysis (EDX, EDS or EDAX) techniques can be used to examine the accuracy of the results obtained for the samples through CEC, SSA and pH analysis. Clay products used for health benefit are found in the topical form as ointments, masks, creams, gels etc., as well as oral supplement (Lopez-Galindo *et al*., 2007; Carretero, 2002). Due to some special properties of clays such as high SSA, CEC, stability of clay particles, near neutral and acidic pH, swelling property etc. they can adsorb and absorb many hazardous substances including heavy metals. New studies focused the safe application of different clays with slight modification for cleaning unwanted substances (Ismadji *et al*., 2013). In many parts of the world, there are uses of soil in these modes of application. '*Nadka Hasa'* is such a soil which is used traditionally as hair cleaner for generations by local '*Chantali*' people of Baksa district of Assam, India, nearing Indo-Bhutan border areas. Nadka means hair washing and hasa means soil in Santali language (Sarma and Rabha, 2013). This is a single example, but lots of such examples are there throughout different parts of the world. Scientific study may reveal the secrets of these soils and then the possibility of development of these into commercial products cannot be ruled out.

Materials and methods

Five samples of soils used as hair cleaner by local people of Baksa district (26.6935^{0}N; 91.5984^{0}E) of Assam, India in the Indo-Bhutan border areas were collected by adopting standard procedure of soil sampling. These samples were drawn from 1 to 1.5-meter depth from the soil surface from six different spots from each of the five places selected with the cooperation of local people. The samples drawn were air dried, ground with a mortar and pestle, the plant roots and stones were removed and finally sieved through 2-mm sieve. Quartering method is applied to get truly representative experimental soil samples of 1kg each. Resulting fine powders (samples) for each soil are preserved in airtight plastic container for physicochemical investigation and numbered as sample 1 to 5 in the order as appeared above. Three commercial clays widely used in cosmetic purposes are also taken for analysis to compare results with experimental samples (sample no. 6,7,8). Some important physicochemical parameters (eighteen numbers) for all the eight samples are evaluated. The parameters and their methods of determination are shown in the Table 1.

Soil Surface Area (*SSA) Calculation*

For the purpose of Soil Surface Area (SSA) calculation, the effective diameter of clay, silt and sand particles are taken as 0.002, 0.02 and 2 mm respectively in accordance to the International System of particle size analysis. They are considered for calculation to get the minimum surface area of each type of particles of the soil samples. The radii of the corresponding particles will be 0.001, 0.01 and 1mm respectively and these are applied as described in the equations in the following paragraph. The total surface area is then calculated by adding the separately calculated surface area for clay, silt and sand particles. In this calculation, experimentally determined particle densities ($\rho_{p)}$ are required to be used. The equations involved in the method are as follows:

Particle Density as per definition, $\rho_p = \frac{m}{v}$

Therefore, $V = m \div \rho_p$ —— equation (a)

Here, m = mass of soil particles, and V = volume occupied only by the soil particles, i.e.

The volume excludes the space occupied by soil air and or soil water. Assuming soil particles to be spherical in shape, the total volume occupied by all the soi particles (V) is:

$$V = n \times \frac{4\pi r^3}{3} \quad \text{—— equation (b)}$$

Here, n is the total number of particles and r is the radius of one particle.

Table1. Methods for measurement of different parameters

Sl. No.	Parameters	Experimental methods
1	Clay%	Bouyoucos Hydrometer method
2	Silt%	Bouyoucos Hydrometer method
3	Sand%	Bouyoucos Hydrometer method
4	Texture	Texture triangle
5	Bulk density (ρ_b)	Weight-volume measurement method
6	Particle density (ρ_p)	Weight-volume measurement method
7	Porosity	Calculation method, $[(1-(\rho_b \div \rho_p)]$ X 100 %
8	Electrical conductivity (EC)	Conductivity meter
9	pH	Digital pH meter
10	Soil organic carbon (SOC)	Titration method
11	Soil organic matter (SOM)	Using Von-Bemmelon factor (SOC X 1.724)%
12	Na^+,	Flame Photometer
13	K^+	Flame Photometer
14	SSA	Calculation method
15	CEC	Calculation method

Therefore, the total no. of particles,

$$n = \frac{3V}{4\pi r^3} \qquad \text{—— equation (c)}$$

So, total surface area of the particles

$$= n \times 4\pi r^2 \qquad \text{—— equation (d)}$$

Putting the value of n in equation (d), we get,

$$\text{total surface area} = \frac{3V}{r} \qquad \text{—— equation (e)}$$

Cation Exchange Capacity (CEC) calculation

The CEC values of the samples are estimated from their Soil Organic Carbon (SOC, %) and clay (%) contents by using regression equation as used by Subba *et al.* (1996). SOC is the mass of carbon atoms present in organic components of soil. It is generally expressed as mass per 100 g of soil.

The regression equation for determining CEC is as follows:

Estimated CEC [cmol (+) kg^{-1}] = $K + a_1x_1 + a_2x_2$

Here K is a constant and x_1 and x_2 are percentages of SOC and clay respectively; a_1 and a_2 are partial coefficients with respect to x_1 and x_2 respectively.

The regression equations used for different clay% and different SOC % to calculate CEC are as given in Table 2.

Characterization of the clay sample by FTIR, XRD and EDAX

FTIR is an analytical technique which has been used for decades in the identification of Clay minerals. The positions of transmittance bands due to structural OH and Si-O groups in the FTIR spectra help greatly in the differentiation of the clay minerals present in a naturally available sample (Madejova, 2003). For the sample preparation, KBr pellets were prepared by mixing clay sample and KBr in the ratio of 1:100 in a hydraulic pellet press machine. The spectra of the pellets in the wave number range 4000 cm^{-1} to 500 cm^{-1} were then recorded using an FTIR spectrometer (ALPHA BRUKER). Similarly, XRD study of the powder clay fraction gives information about the nature of the clays. EDAX is an x-ray technique widely used together with a scanning electron microscope (SEM) to identify the elemental composition of material. Here, a core shell electron is ejected out by applying energy, the vacancy of which is filled up by an outer shell electron releasing the extra energy as X-ray. This x-ray has a characteristic spectrum based on its atom of origin and thus the technique gives formation about the elemental composition of the sample.

Estimation of Swelling Property

Swelling property is expressed in terms of index - Free Swelling Index or Expensive Index. For estimation of swelling property, the apparatus used are a 10-ml graduated test tube and a 10-ml burette. In the 10-mL borosilicate graduated test tube, 2 ml(V) air dried powdered soil sample was taken and 5 mL distilled water added to it. The test tube was kept undisturbed for overnight. After 24 hours, the expanded volume (Vf) of the soil sample was recorded.

The Expansive Index, K=[(Vf-V)÷V]×100%.

Table 2. Equations used for calculation of CEC

Parameter	Quantity (%)	Applicable Regression equation	Equation Serial Number/ symbol
Clay	20 – 40	CEC= - 2.32 +2.97 x_1 +0.99 x_2	(f)
	>40	CEC= 7.51 -1.02 x_1 +0.77 x_2	(g)
SOC	<0.5	CEC= 4.37 +2.91 x_1 +0.79 x_2	(h)
	>0.5	CEC= 3.71 +1.09 x_1 +0.84 x_2	(i)

If free swelling index exceeds 35%, the soil is considered to have high expansion property and if K becomes greater than 50%, the soil is considered to have very high expansion property when comes in contact with water.

Application experiment on human skin

A topical application of the clay sample paste made in pure water was carried out physically in an experiment spread over four months. The clay paste was applied on the back side of the human palm to examine visual effects on the skin of the subject. Clay paste was applied once in 5 days for 10 minutes followed by washing with clean water which was repeated in the experimental period. The gradual effects were recorded by high resolution camera.

Results and Discussions

Physical and chemical Parameters

The average data obtained for all the observed physical and chemical parameters show resemblances of various parameters of the experimental soil samples with those of commercial cosmetic samples (Table 3). The entire experimental samples and the commercial samples were odorless in dry condition and all exhibit at least very high swelling property (40 to 55%) when mixed with water except sample 5. Sample 5 is least swelling (10%) (Table 4). More water absorption by soil indicates more SSA and so ions can easily be exchanged with skin and scalp increasing high CEC. This is due to the higher fraction of clay in the soils. The similarities can be explained by taking any parameter. Let us take potassium for example. In commercial samples, it varied from 4.35 to 41.17 mg l^{-1} and in experimental samples this range is 8.92 to 41.17 mg l^{-1}.

Calculation of CEC values

For CEC calculation, the applicable regression equation is shown in table 2. The calculation is done twice for each sample based on regression equation applicable on the basis of SOC percent and regression equation applicable based on clay percent. Both results were close to each other with a very small deviation (Table 5). CEC values of the reference samples (sample 6, 7, and 8) were in the range between 34.72 and 54.81 cmol (+) kg^{-1} and the CEC range for the local soils (sample 1 through sample 5) were between 32.45 and 60.82 cmol (+) kg^{-1}. It was inferred that samples 5 and 7 were dominated by illite (clays) and others were smectite or likely to be smectite clays on the basis of CEC data (Madejova, 2003). For the CEC calculation the regression coefficients (r) were 0.772 for clay and 4.39 for SOC. Due to the very small size of clay particles a small weight of a clay contains a large number of clay particles. A clay particle is a polar particle. The negative charges are spread over the large surface area of a flake shape particle and the positive charges are spread around the edges. Due to this polar nature of clay particles, they are also called clay minerals because the positive ions such as Al^{3+}, Ca^{2+}, Zn^{2+}, Na^{+}, K^{+}, Mg^{2+}, H^{+} are attached to the negative sides and anions such as silicates, NO^{3-} etc., are attached to the positive edges of the flakes. Since, water molecule is also polar; when water is added to clay, hydration sphere is formed in the clay colloid resulting extension of total surface area which is related to the swelling property of clay. When clay paste/mud is applied on the skin/scalp in a proper way, the toxins such as As^{3+}, Cd^{2+}, Hg_2^{2+}, Pb^{2+}, F^{-}, Cl^{-} are removed by ion exchange process, detoxify and provide essential nutrients. Again, the electric field in the clay colloid destroys the cell wall of bad bacteria in the skin/scalp by losing their hydration. So, clay also acts as antibacterial agent. When the clay paste gradually dries, the pores in the clay particles start absorbing extra oils from the scalp or skin. As such, rapid use of clays can cause dryness to skin. The toxins and unwanted wastes are removed when the dry clay is washed out.

Table 3. Experimental parameters and their values

Parameter	Nadka Hasa					Commercial clays		
	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8
Colour	Grey	Grey	Light grey	Light grey	Dark grey	Yellowish	White	Reddish brown
Odour	Earthy smell, becomes odourless on drying	Earthy smell, becomes odourless on drying	Earthy smell, becomes odourless on drying	Earthy smell, becomes odourless on drying	Earthy smell, becomes odourless on drying	Odourless	Odourless	Odourless
Swelling property	Moderate	Moderate	Enough	Enough	Mild	Enough	Enough	Moderate
pH	4.73±0.01	6.38±0.01	7.00±0.01	7.76±0.01	4.65±0.02	5.31±0.01	7.46±0.01	6.90±0.01
SOC (%)	0.80±0.01	0.86±0.01	0.40±0.02	0.47±0.01	0.60±0.01	0.37±0.01	0.37±0.02	0.96±0.01
SOM (%)	1.379±0.017	1.483±0.017	0.707±0.034	0.810±0.017	1.034±0.017	0.638±0.017	0.638±0.034	1.655±0.017
Textural class	Clay	Clay	Clay	Clay	Sandy clay loam	Clay	Sandy clay	Sandy clay
EC ($mScm^{-1}$)	0.664±0.001	0.048±0.002	1.734±0.001	0.202±0.001	1.340±0.001	4.440±0.001	0.370±0.001	3.458±0.001
ρ_b (gcm^{-3})	0.989	1.022	1.209	1.270	1.120	0.878	0.726	0.797
ρ_p (gcm^{-3})	2.195	2.256	2.854	2.602	2.080	2.810	2.010	2.720
Porosity (%)	54.94	54.70	57.32	51.19	46.29	68.90	63.90	70.70
Clay (%)	50.64	55.36	63.08	69.64	33.36	62.20	36.64	43.36
Silt (%)	16.56	11.56	2.28	3.84	33.56	2.56	7.40	5.56
Sand (%)	32.80	33.08	34.64	26.52	33.08	35.24	55.96	51.08
Na ($mg\ kg^{-1}$)	14.57	15.75	1343.75	2010.00	7.06	2788.75	90.15	5005.00
K ($mg\ kg^{-1}$)	9.44	8.92	41.17	41.17	10.39	41.17	4.35	41.06
CEC [cmol (+) kg^{-1}] from Table-(iv)	46.40±0. 72	50.20±0. 95	55.52±0.14	60.82±0.06	32.44±0.06	54.80±0.22	34.72±0.33	40.55±0.63
SSA (m^2g^{-1}) from Table-(viii)	0.72	0.75	0.67	0.81	0.53	0.67	0.56	0.48

Table 4. Swelling property of the samples

Sample No.	V	Vf	Vf-V	[(Vf-V)÷V]	K=[(Vf-V)÷V]×100	Remarks
-	mL	mL	mL	-	%	-
1	2	2.8	0.8	0.40	40	High expansion property
2	2	2.8	0.8	0.40	40	High expansion property
3	2	2.9	0.9	0,45	45	High expansion property
4	2	3.1	1.1	0.55	55	Very high expansion property
5	2	2.2	0.2	0.10	10	Low expansion property

Table 5. Calculation of CEC values (Calculated as per formula applicable to the experimental data in bold letters)

Sample No.	SOC, x_1 %	SOC range %	Clay, x_2 %	Clay range %	Applicable Regression Equation with number	CEC (Cal) cmol (+) kg^{-1}	Average CEC cmol (+) kg^{-1}
1	0.80	>0.5	50.64	>40	(i), CEC= 3.71 +1.09 x_1 +0.84 x_2	47.12	46.40±0. 72
	0.80	>0.5	50.64	>40	(g), CEC= 7.51 -1.02 x_1 +0.77 x_2	45.68	
2	0.86	>0.5	55.36	>40	(i), CEC= 3.71 +1.09 x_1 +0.84 x_2	51,15	50.20±0. 95
	0.86	>0.5	55.36	>40	(g), CEC= 7.51 -1.02 x_1 +0.77 x_2	49.26	
3	0.41	<0.5	63.08	>40	(h), CEC= 4.37 +2.91 x_1 +0.79 x_2	55.39	55.52±0.14
	0.41	<0.5	63.08	>40	(g), CEC= 7.51 -1.02 x_1 +0.77 x_2	55.66	
4	0.47	<0.5	69.64	>40	(h), CEC= 4.37 +2.91 x_1 +0.79 x_2	60.77	60.82±0.06
	0.47	<0.5	69.64	>40	(g), CEC= 7.51 -1.02 x_1 +0.77 x_2	61.84	
5	0.60	>0.5	33.36	<40	(i), CEC= 3.71 +1.09 x_1 +0.84 x_2	32.38	32.44±0.06
	0.60	>0.5	33.36	<40	(f), CEC= - 2.32 +2.97 x_1 +0.99 x_2	32.49	
6	0.37	<0.5	62.20	>40	(h), CEC= 4.37 +2.91 x_1 +0.79 x_2	54.59	54.81±0.22
	0.37	<0.5	62.20	>40	(g), CEC= 7.51 -1.02 x_1 +0.77 x_2	55.02	
7	0.37	<0.5	36.64	<40	(h), CEC= 4.37 +2.91 x_1 +0.79 x_2	34.40	34.72±0.33
	0.37	<0.5	36.64	<40	(f), CEC= - 2.32 +2.97 x_1 +0.99 x_2	35.05	
8	0.96	>0.5	43.36	>40	(i), CEC= 3.71 +1.09 x_1 +0.84 x_2	41.18	40.55±0.63
	0.96	>0.5	43.36	>40	(g), CEC= 7.51 -1.02 x_1 +0.77 x_2	39.92	

Table 6. Calculation of SSA values contributed by sand particles as per equations (a) and (e)

-1- Sample No.	-2- Particle Density ρ_p	-3- Sand quantity per 100g of soil	-4- Mass of sand per gram of soil	-5- Volume occupied by sand particles, as per equation (a)	-6- Total surface area of the sand particles as per equation (e)
–	gcm^{-3}	% from texture result	(m_1), g	mm^3 Values in column $(4÷2)x10^3$	(S_1), mm^2
1	2.19	33.80	0.3380	154.34	463.02
2	2.25	33.08	0.3308	147.02	441.06
3	2.85	34.64	0.3464	121.54	364.62
4	2.60	26.52	0.2652	102.00	306.00
5	2.08	33.08	0.3308	159.04	477.12
6	2.81	35.24	0.3524	125.41	376.23
7	2.01	55.96	0.5596	278.41	835.23
8	2.72	51.08	0.5108	187.79	563.37

Calculation for SSA values

Here m_1, m_2 and m_3 are the average mass of sand, silt and clay particles respectively. Also S_1, S_2 and S_3 are the surface area values for sand, silt and clay particles respectively. The maximum possible radii of sand, silt and clay particles equal to 1, 0.01 and 0.001mm respectively are taken for calculation of minimum value of surface area of the three different types of particles (Table 6, 7, 8). Overall SSA is calculated by summing up all the above three SSAs (Table 9). The minimum SSA values of the collected samples (minimum 0.53 m^2/g) resemble with the reference samples (minimum 0.56 m^2/g) as we considered the maximum diameter for the particles. In general, higher the SSA, higher is the CEC.

Comparison of pH

All the eight analyzed samples had pH values higher than the hair shaft pH of 3.6. Three of the samples and two of the commercial products have pH higher than the scalp pH of 5.5. It was observed that the pH values for experimental samples 1, 2 and 5 were acidic, and that of sample 3 was neutral and sample 4 was alkaline. Similar to those, pH values for reference samples 6, 8 were acidic and Sample 7 was alkaline. Recent clay research has shown that hair cleaner with pH higher than 5.5 exhibit adverse effect on hair. This includes increase friction, causing frizz,

Table 7. Calculation of SSA values contributed by silt particles as per equations (a) and (e)

-1- Sample No.	-2- Particle Densit ρ_p	-3- Silt quantity per 100g of soil	-4- Mass of silt per gram of soil	-5- Volume occupied by silt particles, as per equation (a)	-6- Total surface area of the silt particles as per equation (e)
—	Gcm^{-3}	% from texture result	(m_2), g	mm^3 Values in column $(4\div2)x10^3$	(S_2), mm^2
1	2.19	16.56	0.1656	75.62	22686
2	2.25	11.56	0.1156	51.38	15414
3	2.85	2.28	0.0228	8.00	2400
4	2.60	3.84	0.0384	14.77	4431
5	2.08	33.56	0.3356	161.35	48405
6	2.81	2.56	0.0256	9.11	2733
7	2.01	7.40	0.0740	36.82	11046
8	2.72	5.56	0.0556	20.44	6132

Table 8. Calculation of SSA values contributed by clay particles as per equations (a) and (e)

-1- Sample No.	-2- Particle Density ρ_p	-3- Clay quantity per 100g of soil	-4- Mass of clay per gram of soil	-5- Volume occupied by clay particles, as per equation (a)	-6- Total surface area of the clay particles as per equation (e)
—	gcm^{-3}	% from texture result	(m_3), g	mm^3 Values in column $(4\div2)x10^3$	(S_3), mm^2
1	2.19	50.64	0.5064	231.23	693690
2	2.25	55.36	0.5536	246.04	738120
3	2.85	63.08	0.6308	221.33	663990
4	2.60	69.64	0.6964	267.85	803550
5	2.08	33.36	0.3336	160.38	481140
6	2.81	62.20	0.6220	221.35	664050
7	2.01	36.64	0.3664	182.29	546870
8	2.72	43.36	0.4336	159.41	478230

Table 9. Calculation of total SSA values

-1- Sample Number	-2- Surface area of the sand particles as per Table v	-3- Surface area of the silt particles as per Table vi	-4- Surface area of the clay particles as per Table vii	-5- Total surface area [(Sum of column 2,3 and 4)÷10^6]
	(S_1), mm^2	(S_2), mm^2	(S_3), mm^2	m^2
1	463.02	22686	693690	0.72
2	441.06	15414	738120	0.75
3	364.62	2400	663990	0.67
4	306.00	4431	803550	0.81
5	477.12	48405	481140	0.53
6	376.23	2733	664050	0.67
7	835.23	11046	546870	0.56
8	563.37	6132	478230	0.48

enhance hair entangle and hair breakage. After use of such shampoo, a conditioner with lower pH needs to be applied so that besides lubricating, the electrostatic forces are neutralized, the frizz effect eliminated and cuticle scales may be sealed. As such the pH of a hair cleaner should not be alkaline. Here, except sample 4 and 7 all are having neutral or acidic pH. However, if the clay has much higher pH, the user needs to apply acidic treatment to help restore hair's natural pH balance after washing it with the clay. In the present experiment, it has been found that the experimental samples are in advantageous position to be used as a hair cleaner compared to the commercial samples.

Nature of the Clay

The result of FTIR analysis of sample 1 is given in Fig. 1.

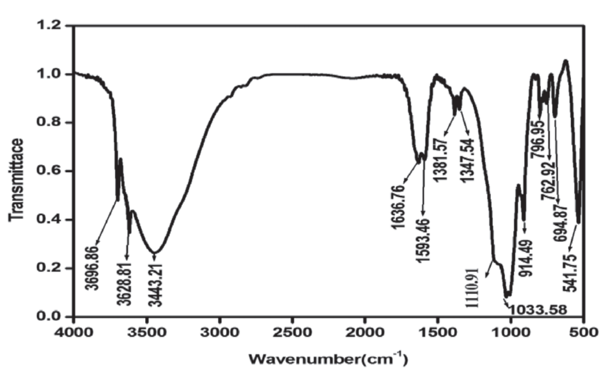

Fig. 1. Fourier Transform Infrared Spectra of sample1

The sharp band in the spectrum of the clay sample at 3696.86 cm^{-1} is due to Al—O-H stretching vibration of kaolinite. Another sharp band at 3628.81 cm^{-1} may be assigned to Al/Mg--O-H vibration (inter octahedral) in smectite (Wanyika *et al*., 2016). A broad band at 3443.21 cm^{-1} is indicative of H-O-H vibration in Kaolinite / Smectite (EHJ Lugwisha, 2011). The band at 1636.76 can be assigned to H-O-H bending vibration of water molecules (Katti *et al*., 2010). The bands at 1593.46cm^{-1}, 1381.57 cm^{-1} and 1347.54 cm^{-1} may be due to some nitrogen containing compound as impurity. The antisymmetric in-plane Si-O-Si stretching vibrations give two sharp bands at 1033.58 cm^{-1} and 1008.83 cm^{-1}. The high frequency band at 1110.91cm^{-1} can be assigned to the Symmetric stretching Si-O vibrations involving the basal oxygen atoms. The AlAlOH bending band at 914.49 cm^{-1} is the OH deformation band arising from vibration of the inner and inner surface OH groups. The weak bands at 796.95 cm^{-1}, 762.92 cm^{-1}, and 694.87 cm^{-1} are due to symmetric stretch of Si-O-Si vibration. The strong band at 541.75 cm^{-1} can be assigned to bending vibration of Si-O-Al group (Gates and Peti, 2017). Powder XRD study of <2μm fraction of the clay sample (Fig. 2) revealed that the clay is a mixture of Illite, Chlorite, Kaolinite and Smectite (Reynolds, 1997). The series of peaks at 2θ values 6.23°, 12.32°, 18.74°,24.79° and 32° gives the

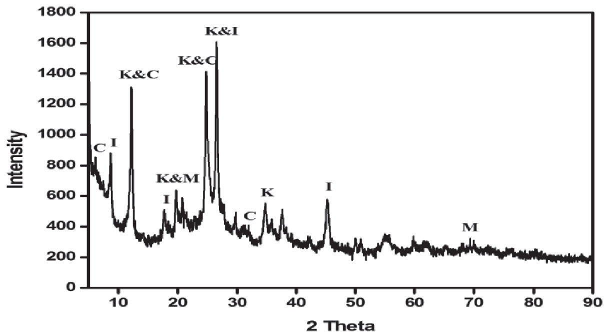

Fig. 2. Report of X-ray Diffraction analysis of sample 1

preliminary identification of Chlorite and can be assigned to 001, 002, 003, 004 and 005 planes. The sharp peaks corresponding 2θ values 12.32°, 19.73°, 24.79°, 26.62°, and 34.49° indicate the presence of Kaolinite. However, when Chlorite and kaolinite are present together in a clay sample, peaks at 2θ values 12.32° and 24.79° which are assigned to 002 and 004 planes of Chlorite overlap or nearly overlap on the peaks due to 001 and 002 planes of Kaolinite (Martin, 1954). Two peaks at 2θ values 19.73° and 69.37° can be assigned to Smectite and the peaks at 17.74°, 26.62° and 45.23° are due to presence of Illite in the sample. The report of EDAX analysis of sample 1 done at 200nm wave length are as given below (Fig. 3, 4; Table 10).

In an average, smectite clay contains oxygen, silicon and aluminium as the first three elements in decreasing order of their percentage by weight. In the experiment sample 1, the same order is followed.

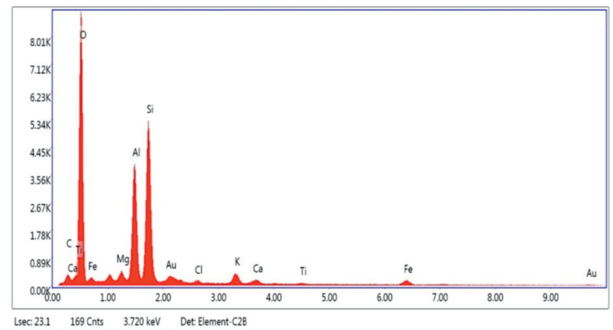

Fig. 4. Report of Scanning Electron Microscopy on elementary composition of sample-1

Results on application of clay on subject

That a clay sample has cosmetic abilityon application on human skin was clear from the application experiment. On completion of the experiment after application of aqueous clay paste

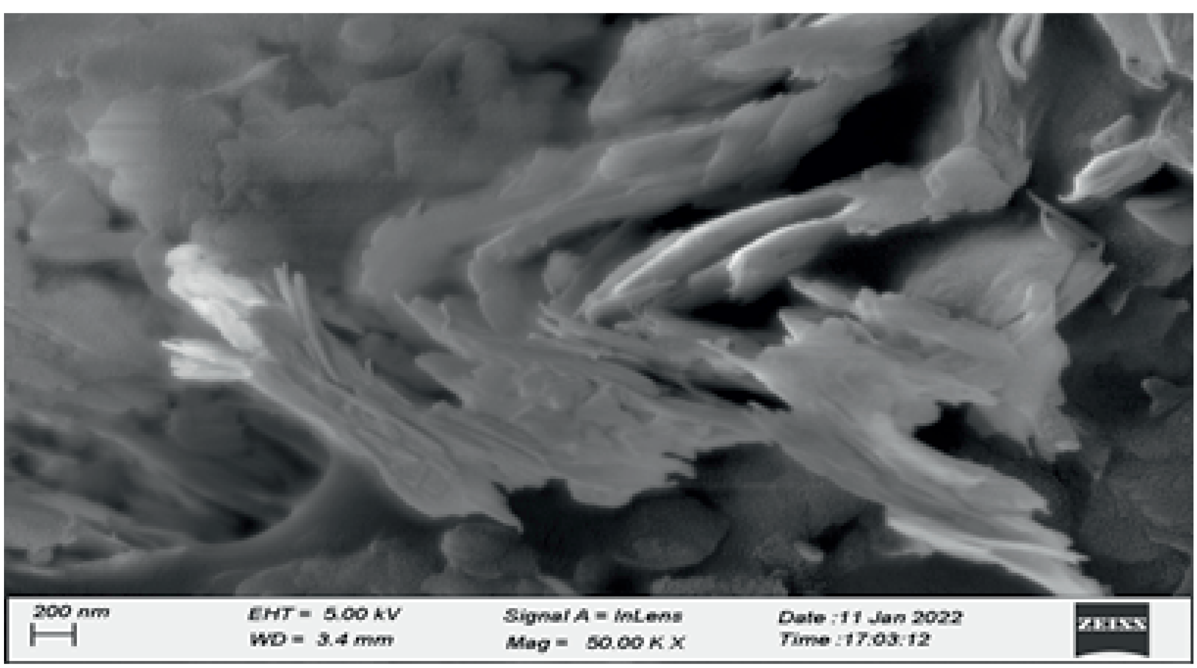

Fig. 3. Energy Dispersive X-ray Analysis report of the sample-1

Table 10. Elemental Composition of Sample -1

Element	Weight %	Atomic %	Net Int.	Error %	K ratio	Z	A	F
C K	2.80	4.80	49.72	16.89	0.0053	1.1176	0.1680	1.0000
O K	47.80	61.65	3075.82	7.49	0.2171	1.0653	0.4263	1.0000
MgK	1.55	1.32	151.86	8.06	0.0102	0.9783	0.6683	1.0032
AlK	15.76	12.05	1675.84	4.36	0.1158	0.9409	0.7780	1.0034
SiK	21.40	15.72	2221.12	4.59	0.1573	0.9604	0.7644	1.0015
AuM	1.06	0.11	36.14	34.88	0.0075	0.6042	1.1151	1.0518
ClK	0.45	0.26	31.87	23.57	0.0035	0.8919	0.8663	1.0041
K K	3.07	1.62	180.94	8.80	0.0257	0.8856	0.9406	1.0072
CaK	1.20	0.62	59.22	16.16	0.0104	0.9007	0.9543	1.0090
TiK	0.57	0.25	23.42	23.88	0.0047	0.8143	0.9805	1.0193
FeK	4.35	1.61	91.45	11.79	0.0357	0.7945	1.0009	1.0343

for a period spread over fourmonths, a noticeable change on the area is observed. The area of application is brighten, smoothen, exfoliated and also showing glow without any adverse symptoms (Fig. 5a, 5b).

1. **2.**

Fig. 5a & 5b. Changes in human skin
Before application At the end of the experiment after 4m

Conclusion

CEC studies have shown that the experimental sample No. 4 is a smectite clay. Non smectite clay includes almost all the existing commercial cosmetic clays (6 and 8). Sample no. 5 proves itself to be illite clay. The other samples i.e. sample no. 1, 2 and 3 seem to be smectite clay. This study reveals that the experimental samples of *Nadka Hasa* used as hair cleaner in raw form possesses properties of clay with high cosmetic ability. If it is properly processed, it can stand as a commercial cosmetic clay in the present market. The clays traditionally used in cosmetic purposes in different parts of the world deserve scientific study so that in near future many of them is likely to be developed to commercial cosmetic materials.

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Conflict of Interest

Authors have declared that none of them have any conflict of interest for the publication of this manuscript.

Contribution of Authors

The experimental works related to this manuscript has been performed by Ms Bidya Chakravorty. Mr Niranjan Phukon, Mr Ambarish Dev Goswami and Dr Sangeeta Agarwal helped in interpreting the experimental data. Dr Prahash

Chandra Sarma had supervised the experimental works and finalised the manuscript.

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