ACTIVATION AND CLARIFYING PROPERTIES OF FULLER'S EARTH.

PART V. DEHYDRATION OF FULLER'S EARTH.

By B. S. Kulkarni and S. K. K. Jatkar.

INTRODUCTION.

192.

It was shown in Part I (*This Journal,* 1936, **19A**, 15–23) that a change in the hydration of the earth would materially alter its properties. The various processes, to which a raw earth is subjected during activation, are accompanied by changes in the water-content. In the process of revivification, *i.e.,* reactivation of the earth which has been used for decolorisation or clarification, the earth is usually subjected to high temperature in order to get rid of the adhering organic matter. The temperature employed has to be high enough to carbonise the organic impurities, but should not be so high as to reduce its water-content to the point of impairing its activity. A precise knowledge about the influence of water-content on the activity of fuller's earth is also necessary for the production of high grade earths.

These technical aspects involve considerations not only with regard to fuller's earth alone, but also with regard to clay materials and ceramic products in general, as fuller's earth is but a peculiar member of a vast family of clay materials. Recently extensive investigations have been made regarding hydration and ultimate constitution of clay materials and ceramic products of known composition, by crystal structure. The cumulative work classifies the minerals into three broad divisions, namely, kaolin, montmorillonite [including montmorillonite $(Mg, Ca)O\ Al_2O_3.\ 5SiO_2\ nH_2O$, Beidellite $Al_2O_3\ 3SiO_2\ nH_2O$, Nontronite $(Al, Fe)O_3\ 3SiO_2.\ nH_2O$, Pyrophyllite $Al_2O_3\ 4SiO_2\ 2H_2O$ and Saponite $2MgO\ 3SiO_2\ nH_2O$] and the alkali-bearing metabentonite and clay mica. The work of Hofmann and co-workers (*Angew. Chem.,* 1934, **47**, 541) with fifty bleaching earths, has established the identity of the essential mineralogical constituent of the fuller's earth, with the minerals of the montmorillonite group, in which the structural type corresponds to the formula $Al_2O_3\ 4SiO_2\ nH_2O$. Fuller's earth is often found to contain minerals of the different groups in very large proportions, but only those earths belonging to the montmorillonite group possess the highest activity.

Each of these different minerals possesses a well-defined and characteristic dehydration curve (loss of water against temperature), which can be used for identification of unknown constituents in clay bodies. Kelly and co-workers (*Soil Science,* 1936, **41**, 259) have recently used such curves for identification of the soil colloids. The dehydration of montmorillonite, has furnished the most interesting study among the clay minerals. Such studies have been made by Kelly and Jenny (*Ibid.*, 1936, **41**, 376–82), Mehmel (*Chem. Erde*, 1937, **11**, 1–16) and others. These curves have been found useful in the classification of the earths used in our investigations.

All hydrous alumino-silicates are characterised by a substantial proportion of combined water. The fact that during dehydration, the hydrous silicates behave both as colloidal gels and as definite compounds, has led many investigators to study the precise manner in which water molecules are united to the remainder of the substance. The three different types of water so far recognised, namely, (1) the colloidal water which controls plasticity; (2) water of hydration or crystallisation (which is reversible); and (3) the water of constitution (which is irreversible), have been interpreted by Kelly and co-workers (*Soil Science,* 1936, **41**, 259) from crystal structure studies of the various minerals by X-ray analysis. The total water-content, according to them, can be divided into two main groups, (1) adsorbed water, and (2) crystal lattice water. The adsorbed water is attached molecularly and can be distinguished as "planar watar" or "broken-bond water", depending on the strength of the forces with which it is held. Considering the crystals of montmorillonite, in which the planar arrangement in the lattice is as follows:—

Montmorillonite
$6\ O^-$
$4\ Si^{+4}$
$4\ O^- + 2\ OH^-$
$4\ Al^{+3}$
$4\ O^- + 2\ OH^-$
$4\ Si^{+4}$
$6\ O^-$

they conclude that each of the exposed ions in the lattice (Al, Si, O) possess a free bond which is a centre of strong electric fields, and that the water molecules being dipoles, are strongly attracted by, and attached to, these exposed or broken-bond ions, through the processes of polarisation and orientation. This they term as 'broken-bond water'. The "planar water" is loosely attached to the Si-O-Si and OH-Al-OH planes, which, although having a large area, are characterised by absence of either unsaturated valence, free electric field or broken-bonds. The union is only through weak electric stray fields and therefore this type of adsorbed water is easily given off at low temperatures. On the other hand, crystal-lattice water is entirely derived from the breakage of the O^- and OH^- planes in the lattice. This corresponds to what was previously described as water of constitution. The significance of these different types of water in fuller's earth in dehydration and activity, is discussed in the later paragraphs.

One important aspect in which fuller's earth differs from the clay minerals, is its activation by acids and consequent changes in its hydration capacity. The relation between activity of the earth and its mode of dehydration, has not yet received sufficient attention. It was believed by Eckart (*Angew. Chem.*, 1934, **47**, 822) that the activation of the earth was brought about by the dissolution of alumina and other bases and the resultant increase of the surface. Hofmann and co-workers (*loc. cit., Angew. Chem.* 1935, **48**, 187) on the other hand attributed the decolorising activity to *the hydrogen and aluminium ions* in the earth. They did not study the relationship between activity and dehydration. It has been shown by us previously (*This Journal,* **20A**, 111–23) that the decolorising action depends solely on the zeolitic hydrogen ion content of the earth. As the dehydration losses of an activated earth would include losses of the hydrogen ion from the crystal planes, there should exist a close relationship between the manner of dehydration of the earth, and its activity.

The dehydration of fuller's earth itself has been studied by Newmann and Kober (*Angew. Chem.*, 1929, **40**, 337), Schultze (*Ibid.*, 1936, **49**, 74) and by Alexanian (*Ann. Combustibles Liquides*, 1936, **11**, 471–86). It was the object of the present investigation to study the dehydration of some of the Indian fuller's earths in order to identify the active constituents, and to study the relationship between hydration, hydrogen zeolite content and decolorising activity.

EXPERIMENTAL.

The earths used in these experiments were samples from some of the major deposits in India: three earths from Jodhpur, one local earth and one from C.P. Kaolin and kieselguhr were used for comparative purposes. As raw earths contain impurities and ballast matter and are likely to give misleading results, only earths previously activated by acid treatment, were subjected to dehydration studies.

Activation.—To 150 gms. of the earth, were added 75 c.c. of concentrated hydrochloric acid (sp. gr. 1.19) and 100 c.c. of distilled water, and the mixture digested under influx for three hours, The earths were then washed free from chlorides and dried at 80 ° –90° The dried products were sieved through 100-mesh and kept over water at room temperature for five days to allow them to attain hydration equilibrium at 25° C.

Dehydration.—The ventilation chimney of the electric furnace used in this work, ensured a steady circulation of air during dehydration. The samples to be hydrated were weighed in platinum crucibles and heated at the desired temperatures for varying periods and weighed to constant weight.

Hydrogen exchange capacity was determined by shaking 2 gms. of the dehydrated earth with 100 c.c. of N NaCl solution, and by determining the pH of the resultant mixture with a quinhydrone electrode.

Decolorising experiments were carried out with ordinary safflower oil, by treating 100 gms. of the oil with 2 gms. of the dehydrated earth at 90°–95°, and measuring the colour of the treated oil on a Lovibond Tintometer.

Results.—Table I gives the per cent. loss in weight, due to dehydration at various temperatures of the earths, and the accompanying figures (1, 2, 3) illustrate the manner of dehydration in each case.

The curves for the three Jodhpur earths, one of which (No. III) is bentonite, run closely parallel to each other. The similarity of these curves with the dehydration curves of some of the typical members

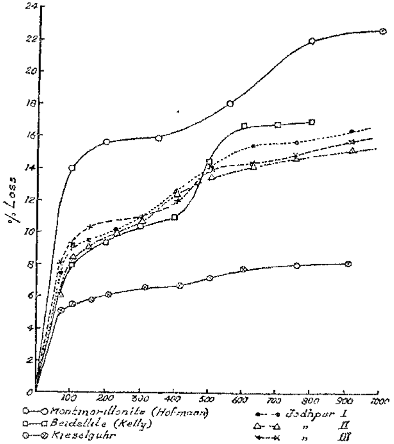


FIG. 1.

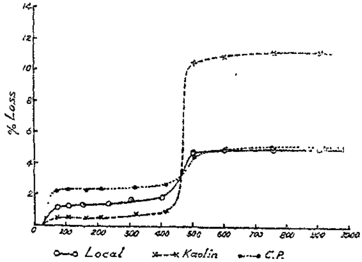


FIG. 2.

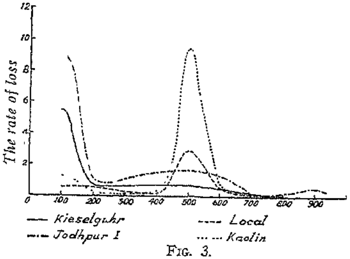


FIG. 3.

of the montmorinollite family [Montmorinolitte, from Hofmann (*Z. Kristallogr,* 1933, **86**, 346) and Beidellite, from Kelly (*Soil Science,* 1936, **41**, 261)] is evident by comparison. These are characterised by high total water-content, most of which is given off below 500°. The inflexions in the curves occur at separate stages, at which the three different types of water, *viz.,* planar water, broken-bond water and lattice water are given off. This is illustrated in Fig. 2, in which, the rate of loss of water is plotted against temperature. The curves show, how at some stages the rate is slow, while at others it is very high within a narrow range of temperature.

Entirely different kinds of curves are given by the local earth, C.P. earth and Kaolin. From the manner of dehydration, they appear to belong to the same group. The total water-content is smaller than that found in the montmorinollite family, all the adsorbed water (which forms less than 2% of the mineral) is given out below 80°. Between 430° and 500° there is not much loss, indicating an absence of broken-bond water. The whole of the crystal-lattice water is lost

TABLE I.

Hydration Loss and Temperature.

Jodhpur earth I		Jodhpur earth II		Jodhpur earth III		Local earth		C.P. earth		Kaolin		Kieselguhr	
Temp. °C.	% Loss	Temp. °C.	% Loss	Temp. °C.	% Loss	Temp. °C.	% Loss	Temp. °C.	% Loss	Temp. °C.	% Loss	Temp. °C.	% Loss
70	7·45	70	6·02	70	8·08	70	1·17	70	2·19	70	0·48	70	5·15
107	9·10	107	8·40	107	9·29	107	1·24	107	2·29	107	0·52	107	5·53
150	9·71	150	9·06	150	9·91	150	1·28	160	2·24	160	0·54	160	5·83
232	10·16	232	10·06	232	10·48	232	1·45	210	2·40	210	0·57	210	6·12
305	11·02	305	10·70	305	10·96	305	1·71	316	2·56	316	0·73	316	6·77
406	12·59	406	12·42	406	12·13	406	1·84	417	2·74	417	0·93	417	6·78
503	14·22	503	13·52	503	14·02	503	4·71	504	4·48	504	10·29	504	7·38
628	15·51	613	14·23	613	14·36	613	4·99	607	4·93	607	10·96	607	7·89
757	15·85	757	14·75	757	14·93	757	5·18	760	5·04	760	11·26	760	8·13
915	16·44	915	15·21	922	15·78	922	5·18	910	5·15	910	11·36	910	8·25

at about 500°. The local and the C.P. earths show a large proportion of the kaolin content, which accounts for theer low bleaching power (*see* below).

A third type of dehydration curve is the one given by kieselguhr, and is characteristic of a substance which acts as a purely capillary adsorbent. The amount of adsorbed water, which is about 5%, is given out below 80°, after which, the remaining 3% is lost in regular manner up to 800°, without showing any inflexions. It is difficult to say whether kieselguhr contains any crystal-lattice water, because the manner of dehydration, resembles drying of an amorphous material or of a colloidal gel. A comparison of this curve with that obtained for fuller's earths, lends support to the view that in decolorisation or clarification, kieselguhr and fuller's earth act entirely differently.

It will be seen, that the curves for the three Jodhpur earths do not show complete identity with any of the pure mineral, nor do the curves for local and C.P. earths are similar to that of kaolin. This is because, the activated earths, in addition, contain certain proportions of the minerals belonging to other classes, especially free silica, which has the effect of lowering the per cent. of hydration.

Although it is difficult to precisely mark the stages at which the three different types of water are evolved, the method suggested by Kelly and co-workers (*loc. cit.*) appears to be fairly satisfactory. A comparison of the amounts of the different types of water in the earth, is tabulated in Table II which brings forth the identity of their constituent material. Montmorillonites are characterised by high total water contents, high absorbed water content but low lattice water content, while the kaolinic groups are characterised by low adsorbed water content and relatively high lattice water content.

Table II.

Earth	Total water %	Adsorbed water %	Crystal lattice water %
Jodhpur earth I .	16·69	12·59	4·10
,, ,, II ..	15·81	12·42	3·39
,, ,, III ..	17·09	12·13	4·96
Montmorillonite[1] ..	23·2	19·0	4·2
Ca-Beidellite[2]	19·65	14·59	5·06
Local earth	5·18	1·84	3·27
C.P. earth	5·15	2·74	3·41
Kaolin .. .	11·36	0·93	10·43
Kaolinite[2]	16·86	2·80	14·00
Kieselguhr	8·25	8·25	..

[1] Mehmel. [2] Kelly and Jenny.

Effect of activation.—As stated previously, raw earth may ontain, in addition to free oxides and carbonates, minerals of both ontmorillonite and kaolin groups, the quality of the earth depending n the proportion of the minerals of the former group. On treating ch product with a sufficient quantity of a mineral acid, all the solu-le free oxides and carbonates are dissolved, and simple silicates are ecomposed with precipitation of free silica. A portion of the mine-ls having low silica-alumina ratio (kaolins) are also broken up ith deposition of free silica, and finally, the hydrogen from the id replaces most of the other basic elements like Ca, Mg, Na, K, e, etc. The acid treatment thus serves both as a process of purifica-on and activation, the treated products being mainly composed of e minerals of the montmorillonite group, heavily charged with the ydrogen ions in the crystal lattice. As a result, the water-content the activated earth is always found to be higher than the raw ones, rticularly in the case of the montmorillonite earths. Chemical nalysis, although incapable of yielding information worth the labour. ves sufficient qualitative information in support of what has been id. For illustration the chemical composition of the original Jodh-r earths I and II (*montmorillonite*) is given in the following table:

TABLE III.

Analysis of the original earth.

	Moisture	Ignition loss	% on Dry earth					
			SiO_2	Fe_2O_3	Al_2O_3	CaO	MgO	Na, K
lhpur I ..	0·9	9·2	54·1	12·0	20·8	0·32	3·18	0·42
,, II ..	7·9	11·9	55·7	6·5	15·0	0·94	6·41	4·63

The losses on activation of the earths are given in Table IV.

TABLE IV.

Earth	% Loss
Jodhpur I	25·3
,, II	28·0
,, III	32·5
Local earth	6·5
C.P. earth	52·4

The quantities of the three main constituents in the activated products found on analysis, are tabulated below (Table V):—

TABLE V.

Analysis of the activated earths.

Earth	Total H_2O %	SiO_2 %	Al_2O_3 %	Silica Alumina
Jodhpur I	16·7	60·4	22·9	2·63
,, II ..	15·8	69·6	14·6	4·77
,, III ..	17·1	59·9	23·1	2·60
Local	5·2	74·1	20·9	3·54
C.P. earth ..	5·2	83·2	11·6	7·17

It will be noticed from the above results, that there is a considerable increase in the water content on activation. The silica alumina ratio shows an increase, although, on treatment of montmorillonite, with acids, the ratio should decrease. In the naturally occurring montmorillonite, the alumina silica ratio is 3.09, by weight, but if the bases are removed and it is reduced to pure hydrous alumino-silicate ($Al_2O_3 . 4\ SiO . nH_2O$) the silica alumina ratio reduces to 2.36. The dehydration curves have shown that, the three Jodhpur earths belong to the montmorillonite group. From the ratios given in the last column, it may appear that they are wholly composed of pure montmorillonite and silica. We intend to subject this mineral to X-ray analysis in order to confirm this finding.

TABLE VI.

	25° C.			70° C.		
	pH	(H·)	Change in Lovibond y	pH	(H·)	Change in Lovibond y
1. Jodhpur earth I ..	3·11	$7·8 \times 10^{-4}$	15·9	3·10	$7·9 \times 10^{-4}$	16·2
2. ,, ,, II ..	3·57	$2·7 \times 10^{-4}$	14·9	3·55	$2·8 \times 10^{-4}$	15·3
3. ,, ,, III ..	3·01	$9·9 \times 10^{-4}$	16·0	2·95	$1·1 \times 10^{-3}$	16·1
4. Local earth ..	6·07	$8·5 \times 10^{-7}$	3·6	6·05	$8·9 \times 10^{-7}$	3·6
5. C.P. earth	4·03	$9·3 \times 10^{-5}$	9·0	3·94	$1·2 \times 10^{-4}$	9·4
6. Kaolin	5·95	$1·1 \times 10^{-6}$	2·2	5·92	$1·2 \times 10^{-6}$	2·1
7. Kieselguhr	4·42	$3·8 \times 10^{-5}$	7·9	4·32	$4·8 \times 10^{-5}$	7·9

	100° C.			150° C.		
	pH	(H·)	Change in Lovibond y	pH	(H·)	Change in Lovibond y
1. Jodhpur earth I ..	3·06	$8{\cdot}7 \times 10^{-4}$	16·0	3·04	$9{\cdot}1 \times 10^{-4}$	16·5
2. ,, ,, II ..	3·53	$2{\cdot}9 \times 10^{-4}$	15·3	3·55	$2{\cdot}9 \times 10^{-4}$	15·8
3. ,, ,, III ..	2·92	$1{\cdot}2 \times 10^{-3}$	16·1	2·91	$1{\cdot}2 \times 10^{-3}$	16·2
4. Local earth ..	6·27	$5{\cdot}4 \times 10^{-7}$	4·7	6·24	$5{\cdot}8 \times 10^{-7}$	4·7
5. C.P. earth .. .	3·99	$1{\cdot}1 \times 10^{-4}$	9·6	4·06	$8{\cdot}6 \times 10^{-5}$	10·0
6. Kaolin	5·99	$1{\cdot}0 \times 10^{-6}$	2·1	5·83	$1{\cdot}5 \times 10^{-6}$	2·1
7. Kieselguhr	4·31	$4{\cdot}9 \times 10^{-5}$	8·1	4·23	$5{\cdot}9 \times 10^{-5}$	8·6

	200° C.			300° C.		
	pH	(H·)	Change in Lovibond y	pH	(H·)	Change in Lovibond y
1. Jodhpur earth I ..	3·05	$8{\cdot}9 \times 10^{-4}$	16·2	3·13	$7{\cdot}4 \times 10^{-4}$	16·0
2. ,, ,, II ..	3·58	$2{\cdot}6 \times 10^{-4}$	15·6	3·54	$2{\cdot}9 \times 10^{-4}$	5·5
3. ,, ,, III ..	2·94	$1{\cdot}1 \times 10^{-3}$	15·9	3·06	$8{\cdot}7 \times 10^{-4}$	15·9
4. Local earth ..	6·15	$7{\cdot}1 \times 10^{-7}$	4·6	5·94	$1{\cdot}1 \times 10^{-6}$	4·4
5. C.P. earth	4·15	$7{\cdot}3 \times 10^{-5}$	9·8	4·16	$7{\cdot}0 \times 10^{-6}$	9·4
6. Kaolin	6·05	$8{\cdot}9 \times 10^{-7}$	2·1	6·28	$5{\cdot}2 \times 10^{-7}$	1·6
7. Kieselguhr	4·30	$5{\cdot}0 \times 10^{-5}$	8·1	4·29	$5{\cdot}2 \times 10^{-5}$	8·1

	400° C.			500° C.		
	pH	(H·)	Change in Lovibond y	pH	(H·)	Change in Lovibond y
1. Jodhpur earth I	3·57	$2{\cdot}7 \times 10^{-4}$	15·8	3·68	$2{\cdot}1 \times 10^{-4}$	16·1
2. ,, ,, II	3·91	$1{\cdot}2 \times 10^{-4}$	13·7	3·70	$2{\cdot}0 \times 10^{-4}$	15·2
3. ,, ,, III	3·34	$4{\cdot}6 \times 10^{-4}$	15·9	3·47	$3{\cdot}4 \times 10^{-4}$	15·8
4. Local earth	5·9	$1{\cdot}2 \times 10^{-6}$	4·4	5·92	$1{\cdot}2 \times 10^{-6}$	4·2
5. C.P. earth	4·31	$5{\cdot}0 \times 10^{-5}$	9·2	4·39	$4{\cdot}1 \times 10^{-5}$	9·0
6. Kaolin	5·91	$1{\cdot}2 \times 10^{-6}$	1·6	4·87	$1{\cdot}3 \times 10^{-5}$	1·6
7. Kieselguhr	4·30	$5{\cdot}0 \times 10^{-5}$	7·9	4·29	$5{\cdot}1 \times 10^{-5}$	7·9

	600° C.			750° C.		
	pH	(H·)	Change in Lovibond y	pH	(H·)	Change in Lovibond y
1. Jodhpur earth I	3·84	$1{\cdot}4 \times 10^{-4}$	15·7	4·79	$1{\cdot}6 \times 10^{-5}$	14·6
2. ,, ,, II	3·79	$1{\cdot}6 \times 10^{-4}$	14·9	4·99	$1{\cdot}0 \times 10^{-5}$	14·4
3. ,, ,, III	3·47	$3{\cdot}4 \times 10^{-4}$	15·8	3·55	$2{\cdot}8 \times 10^{-4}$	15·7
4. Local earth	5·91	$1{\cdot}2 \times 10^{-6}$	3·9	5·88	$1{\cdot}3 \times 10^{-6}$	2·1
5. C.P. earth	4·48	$3{\cdot}2 \times 10^{-5}$	8·8	4·79	$1{\cdot}6 \times 10^{-5}$	7·6
6. Kaolin	5·12	$7{\cdot}6 \times 10^{-6}$	1·6	5·75	$1{\cdot}8 \times 10^{-6}$	7·6
7. Kieselguhr	4·70	$2{\cdot}0 \times 10^{-5}$	7·6	5·84	$1{\cdot}5 \times 10^{-6}$	6·6

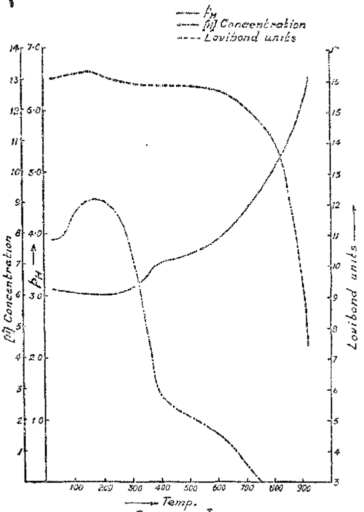
	900° C.		
	pH	(H·)	Change in Lovibond y
1. Jodhpur earth I ..	6·66	$2·2 \times 10^{-7}$	7·4
2. ,, ,, II ..	6·79	$1·6 \times 10^{-7}$	4·5
3. ,, ,, III .	5·45	$3·5 \times 10^{-6}$	11·1
4. Local earth ..	6·59	$2·6 \times 10^{-7}$	2·1
5. C.P. earth	6·38	$4·2 \times 10^{-7}$	6·2
6. Kaolin	6·42	$3·8 \times 10^{-7}$	1·1
7. Kieselguhr	6·56	$2·7 \times 10^{-7}$	5·6

In some of the earths which contain a large proportion of the kaolinic matter, the alumina-silica ratio is higher than the ratio for pure kaolin. These earths, therefore, contain a very high proportion of free silica.

Hydrogen exchange capacity.—Table VI gives the hydrogen exchangeable capacities of the dehydrated earths in terms of both pH and of the hydrogen-ion concentration.

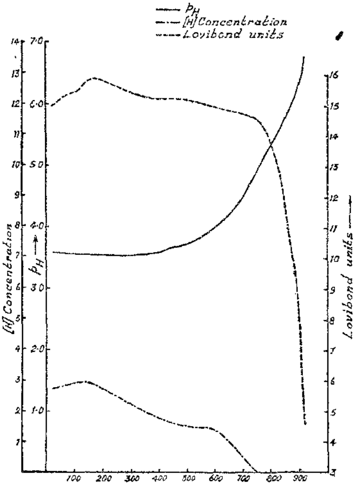
The pH values are plotted against temperatures in Figs. 4, 5, 6 and 7. As in the case of dehydration experiments, the earths appear to fall into different groups in exchange capacities also. The earths generally show higher exchangeability at lower temperature, the activity being completely destroyed at 900°. Considering the pH values at the lower temperatures, the three Jodhpur earths (Montmorillonite group) show the lowest pH among the earths. The C.P. earth stands midway between the Jodhpur earths on one hand, and the local earth and kaolin on the other. The exchange capacity is moderate, and the results indicate that the earth is an uneven mixture of montmorillonite and kaolin. In the local earth, the pH is of the order observed in kaolin itself. It also contains a high proportion of silica. The earth does not indicate the presence of any montmorillonite at all.

The significance of the pH measurements is best shown by plotting the exchanged hydrogen-ion concentration against the temperature, in case of the three Jodhpur earths, the highest values for exchangeable hydrogen being observed between 150° and 200 °, above which there is precipitate decrease in the available hydrogen ions.



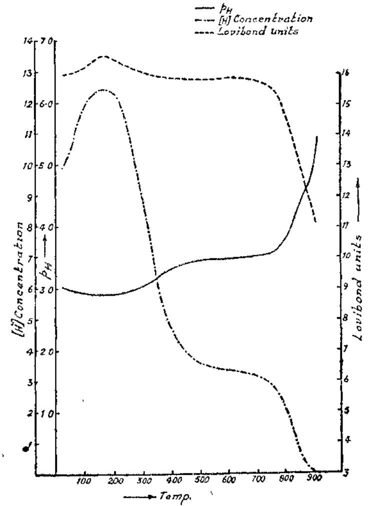
JODHPUR I

FIG. 4.



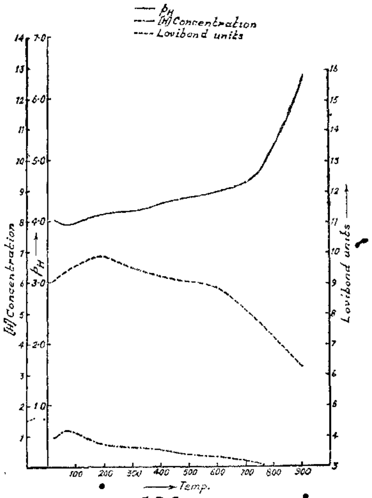
JODHPUR II

FIG. 5.



JODHPUR III

FIG. 6.



C.P. EARTH

FIG. 7.

The seat of ionic exchange in the clay material is the crystal lattice. According to Kelly and Jenny (*loc. cit.*) two types of OH planes are significant from the standpoint of H-ion replacement, first, a plane of OH ions immediately on the exposed surface of the lattice package, which is arranged in the following manner:—

H H H H H H
O O O O O O (hydroxyl plane),

second the subsurface OH plane which is a network of O ions:

O O O O O O (Oxygen plane)
H H H H H
O O O O O (Hydroxyl plane).

In both the types, H ions are available for replacement. In activated earths, the most abundant source of H ion for replacement is those ions situated on the broken edges of the Si-O planes, *i.e.*, those which have replaced the various cations on activation.

Considering the results in light of this, it appears that the planar water, which is loosely attached and is driven out at about 150°, envelopes the replaceable ions, making their exchange difficult. After the expulsion of this water, hydrogen exchange-capacity is uniformly increased. When the temperature is sufficiently high to break up the OH and O-OH planes, the H ions on the broken edges appear to be the first to be driven out along with the nearest O and OH ions. This is shown by the precipitate loss of hydrogen exchange capacity between 200° and 400°. The hydrogen ions exchanged at temperatures higher than 450°, are very few and are entirely derived from the innermost still unbroken OH planes, and also by mere physical adsorption by the free silica and alumina. The exchange capacity at high temperatures is thus very low, and nearly constant.

Dehydration and decolorising activity.—In the previous papers the direct relationship between the decolorising ability of fuller's earth and its exchangeable hydrogen ion was established. It was found that higher the exchange capacity, the higher was the decolorising power. In the present experiments the object was to study whether the same relationship holds good in case of the same earth which has been dehydrated at different temperatures and has thus suffered different losses in its exchangeable hydrogen ion. The amount of decolorisation was studied by the change in Lovibond units of yellow colour with safflower oil. In Table VI each of the third columns under temperature, gives the change (in yellow Lovibond units) produced by 2% earth, in the safflower oil. In Figs. 4, 5, 6 and 7 the pH and the colour units removed by the earth are plotted against temperature.

Highest decolorising activity is shown by the Jodhpur earths, next in order, comes the C.P. earth, while kieselguhr, local earth and kaolin show the lowest activity. The same order was also shown, regarding the exchangeable hydrogen ion. The fact that kieselguhr shows lower decolorising power, lends support to the view that the earths which decolorise by virtue of their hydrogen content are usually more powerful decolourisers than those which act merely by surface adsorption.

Although the reciprocal relationship between the pH of the salt solution treated by the earth and the decolorising power is well established, and there is a general parallelism between the exchangeable hydrogen ion of the dehydrated earth and its decolorising power, the proportionality is not exact, with earths heated at higher temperatures. This can be seen by comparing the figures for the exchanged hydrogen ion and the change in the Lovibond units shown in figures 4, 5, 6 and 7 and Table VI. It appears that, here, the activated earth decolorises by simultaneous *adsorption and zeolitic action*. This can be explained if we assume that the planes at the broken edges in the lattices occupied by the hydrogen ions, act as strong adsorption centres. A detailed study of this aspect of the problem will be published in the next part of this series.

A study of the adsorption isotherm of the fuller's earth is in progress.

SUMMARY.

The identity of the active mineralogical constituent of some Indian fuller's earths, with montmorillonite, has been established by a study of the comparative dehydration curves.

The difference between the kaolinitic clays and those of the montmorillonite group, is shown by the different types of water given out during dehydration.

The hydrogen exchange capacities of the earths are maximum when dehydrated between 150°–200°, the activity being destroyed at higher temperatures.

The decolorising power of the various earths is also maximum when heated at 150°–200°, and is found to be inversely proportional to pH, in agreement with the results obtained previously. It appears that the decolorising action of fuller's earth is due to adsorption by the hydrogen zeolite.

Department of General Chemistry,
Indian Institute of Science,
Bangalore (India).

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