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Pedology Department

A. Muir

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PEDOLOGY DEPARTMENT

A. MUIR

J. H. Rayner attended a colloquium on Clay Mineral Genesis and Synthesis held in Paris. A. Muir visited India on behalf of UNESCO to consider the setting up of a Central Institute for Pedology and Soil Mechanics in relation to existing institutes. Mr. C. B. Wells (Commonwealth Scientific and Industrial Research Organisation) returned to Australia. Dr. M. Covadonga Rodriguez Pascual completed her study of swelling properties of soils with R. Greenekelly and has joined the Soils Department in Oxford. Mr. Ismail Tunckale (Istanbul University) is working with both the Soil Survey and the Pedology Department.

The following visitors came to gain experience in X-ray techniques: Professor R. Lounsbury, Purdue University, Indiana; Dr. A. Lapa, University of Oporto; Mr. I. Babiker, University of Khartoum; Mr. D. Carroll, University of Oxford; Mr. E. Robinson, University of Queensland; Mr. P. Watson, University College of Rhodesia and Nyasaland. Their training occupied much time and detracted from the ordinary work of the department.

G. Brown edited, on behalf of the Mineralogical Society, the second edition of *The X-ray identification and crystal structures of clay minerals*, which has now been published and should be as successful internationally as the first edition.

SOIL FORMATION

Sols lessivés (gray-brown podzolic soils)

In the definition of this group of soils, referred to as brown earths, Robinson allowed some mechanical translocation of clay. The profiles of such soils have an increased clay content in the subsoil and in Europe are known as *sols lessivés*. We have not attempted to characterise such soils in Britain in any detail, but if they are to be recognised here as elsewhere as an international group it is obvious that soils chosen for comparison should be developed on homogeneous parent materials of which loess-like drifts are the most suitable. The Soil Survey and Pedology Department were invited to co-operate in the detailed study of profiles of *lessivé* type derived from "brickearth". The samples were collected by Mr. P. Askew (Wye College), and the results recorded below are the joint work of the Pedology Department and the Soil Survey.

The site, at Kennington, Kent, is 165 feet above sea-level on a very slightly sloping terrace. The average rainfall is about 29 inches per annum; the average maximum and minimum temperatures (° F.) in winter are 53.8 and 22.0 and in summer 83.4 and 43.6 respectively. The vegetation is mainly of oak with beech, sycamore, holly and elder; ivy is the only ground flora. Drainage of both site and profile is satisfactory.

The profile consists of thin ($\frac{1}{4}$ inch) F layers overlying a very

E

dark brown, finely granular loam A₁₁ horizon (2 inches) which changes abruptly to a brown, structureless, very friable, loam to silt loam A₁₂ horizon (12 inches) that merges into a pale brown, structureless, silt loam A₂ horizon about 6 inches thick. A transitional B₁ horizon (8 inches) separates the A horizons from the B₂ horizon, which is over 36 inches thick, and is characterised by dark-brown and reddish-brown colours, prismatic structure and a slightly finer texture than the upper layers. Between 66 and 72 inches this merges into C horizon material of yellowish-brown, structureless silt loam, which persists to about 96 inches and shows gley features along old root channels.

Thin sections were examined, and the disposition of the oriented clay indicates that it has been removed from the two upper mineral layers and accumulated in maximum amounts between about 35 and 50 inches. Evidence of clay deposition in voids is visible even at 96 inches. The maximum frequency of secondary iron concretions at about 22 inches suggests not only segregation at a single level but also movement of iron down the profile. Darker segregations of iron and manganese oxides become prominent near the base of the profile, and their deposition may be associated with the higher pH values and the presence of small calcite grains at 66–84 inches. The rarity of glauconite, muscovite, feldspars and calcite in the upper layers might suggest a different parent material from that

Sample depth (in.)	2½–13½	13½–19	19–27	30–36	39–45	66–72	78–84
Horizon	A ₁₂	A ₂	B ₁	B ₂	B ₂	C ₁	C ₁
Moisture 100–105° C.	2.28	2.05	2.10	2.69	2.80	3.07	2.89
<i>Mechanical Fractions</i>							
600–2,000 μ	0.9	1.0	1.2	0.1	0.1	0.1	0.1
200– 600 μ	2.7	2.3	2.6	0.8	0.6	1.7	2.1
60– 200 μ	6.7	6.3	6.0	5.9	5.9	9.0	6.6
20– 60 μ	45.7	45.3	45.0	41.2	38.2	45.5	47.0
6– 20 μ	20.6	20.0	20.3	22.5	20.1	19.4	16.4
2– 6 μ	5.1	5.4	4.5	5.5	6.6	5.0	4.3
1– 2 μ	2.4	3.9	5.4	2.8	5.3	2.6	2.3
<1 μ	13.6	14.2	15.5	21.2	24.6	19.3	18.6
% CaCO ₃						0.2	0.3
Dissolved R ₂ O ₃ (included in <2 μ)	0.3	0.2	0.2	0.1	0.1	0.1	0.1
Loss on ignition, %	5.4	3.6	3.4	3.5	3.4	2.9	2.7
Organic carbon, % C.	2.26	0.97	0.72	0.27	0.20	0.10	0.12
pH in water (1 : 2.5)	4.2	4.4	4.5	5.1	5.2	7.5	7.8
Exchangeable Ca	0.70	0.65	1.60	6.09	6.22		
m.e./100 g. Mg	0.09	0.11	0.28	1.59	1.46		
„ K	0.04	0.05	0.10	0.13	0.12		
„ Na	0.03	0.03	0.11	0.26	0.31		
„ Total	0.9	0.8	2.1	8.1	8.1		
„ H+	15.8	11.5	10.5	7.2	6.6		
C.E.C. (by addition)	16.7	12.3	12.6	15.3	14.7		
Total Bases (Jackson)	2.5	3.3	4.5	10.3	10.6		
Soluble Fe ₂ O ₃ , % (KCl-EDTA)	0.67	0.76	0.74	0.59	0.46	0.50	0.59
Soluble Al ₂ O ₃ , % (KCl-EDTA)	0.25	0.26	0.26	0.20	0.18	0.16	0.19
<i>Clay Analysis</i>							
SiO ₂ /R ₂ O ₃	2.64	2.63	2.58	2.73	2.72	2.74	2.69
SiO ₂ /Fe ₂ O ₃	9.47	10.70	10.15	10.00	9.61	10.65	10.10
SiO ₂ /Al ₂ O ₃	3.70	3.49	3.47	3.76	3.79	3.68	3.65

below, but it is more probable that they have been weathered out and the decomposition products removed from higher levels by leaching. X-ray analysis of the clay minerals reveals that vermiculite-montmorillonite are dominant below 2.5 inches, with mica dominant in the top mineral soil. This is a common feature in some soils and suggests that the potash released by the vegetation is adsorbed and fixed by the clay.

The table presenting analytical data of selected layers shows that the mechanical analyses support the idea that the profile is homogeneous, for there is no striking difference between horizons in the amount of any fraction except of the clay. This increases greatly at about 30 inches and maintains similar high levels to about 66 inches, below which it decreases. The rise in the amount of clay, combined with the thin-section observations, confirms that clay has been mechanically transferred into the B horizon. That little chemical breakdown has occurred is revealed by the ratios of silica to sesquioxides which, except for slight decreases in $\text{SiO}_2/\text{Fe}_2\text{O}_3$ between 19 and 45 inches, are almost constant down the profile. Thin sections confirm the segregation of iron at these levels. As might be expected, the exchangeable bases increase with the increased clay content of the B horizon, in which the exchangeable hydrogen decreases from the higher values, associated with lower pH values, in the upper layers.

All the evidence suggests that this profile falls into the class of "sols bruns lessivés" or "gray-brown podzolic soils" developed on silty material which becomes slightly calcareous at depth. (Bascomb, Du Feu, Rudeforth, Stephen.)

MINERALOGY

Clay mineralogy

The normal crystallographic work was hampered by the record number of visiting workers, all of whom required instruction in X-ray techniques, and most of whom examined their own material to incorporate the results in theses. (Brown.)

A total of 214 samples from home and overseas were analysed for clay minerals. The common presence of palygorskite in soils from the Middle East has previously been reported. This mineral was again found in some Persian soils. Unless it is present in large amounts, it is more simply detected by the electron microscope than by X-rays. (Du Feu and Brown.)

PHYSICAL CHEMISTRY OF SOIL MINERALS

Surface studies

It is a common observation that clay soils of similar clay contents differ greatly in drainage characteristics, which are evidently correlated with the development of structure in the subsoil. An examination of the factors responsible was started with a study of a range of $(\text{Ca}, \text{Mg})^{2+}$ saturated subsoils with clay contents about 50%, but with widely differing nitrogen specific surfaces (27–103 m^2/g . external surface). The physical properties of these soils are being compared; so far the British Standard plastic and liquid limits,

suction curves and shrinkage characteristics have been measured. The plasticity index (liquid limit–plastic limit) was nearly independent of the specific surface, in agreement with the results of Odell, Thornburn and McKenzie (*Proc. Soil Sci. Soc. Amer.* 1960, **24**, 297–300), who found that it could be highly correlated with clay content and less so with mineral composition of the clay, silt percentage, exchange capacity and carbon content. The maximum shrinkage, i.e., the difference between the liquid limit and shrinkage limit, was remarkably constant for the remoulded samples, although the field soils had very different characteristics. For example, gilgaied tropical black earths from Australia have exceptionally high specific surfaces (relative to clay content) and have great shrinkage with detrimental effects on foundations, fences, and telegraph posts. In this connection, Stephen, Bellis and Muir (*J. Soil Sci.*, 1956, **7**, 1–9) investigated a puff and shelf phenomenon in soils of the Athi plain (Kenya) and concluded, despite evidence of termite activity, that the soils were gilgai. The specific surface of the soils was 150 m.²/g. (for 100% clay), the highest specific surface yet measured on a soil. The natural inference is that the soils would be gilgai if the conditions were suitable.

The effect of removing free iron oxides and organic matter on the specific surfaces of selected soils was investigated. Removing organic matter increased the specific surface, whereas removing iron oxides usually slightly decreased it. (Rodriguez and Greene-Kelly.)

Apparatus

A high-speed differential thermal analysis apparatus was made to the design of Foldvari-Volg and Klibursky. It is manually operated during the fifteen-minute run, and the differential temperature is recorded photographically. The apparatus has excellent resolving power; it has been demonstrated for the first time that the 50–200° endotherm of illites has the same double-peak formation (depending on the exchange cation) as montmorillonite.

A vacuum thermobalance using a silica spring was built. The products of decomposition can be condensed in traps or the apparatus filled with a particular gas depending on the information required.

Both these new instruments will be used to study soil minerals. (Greene-Kelly.)

Clay mineral synthesis

The series of experiments on the rate of transformation of silica–alumina gel to kaolin described last year was continued at the lowest of the temperatures used before (180°), but allowing longer reaction times. At 180° the time for half conversion is at least six weeks.

Brindley and De Kimpe (*Nature, Lond.* 1961, **190**, 254) claimed to have prepared kaolin at 150° in one to three weeks from gibbsite and “Ludox” silica sol or ethyl silicate in *N*/10-HCl. Similar experiments in a furnace built by G. C. Dibley, to heat six sealed glass tubes to this temperature while turning them end-over-end,

gave only a small yield of kaolin. The difference is explained by the wrong estimates made of reaction tube temperature, for De Kimpe (private communication) now states that the temperature for complete transformation should be 165–175°. In the first experiment done here the maximum tube temperature was 160°, and running the furnace 10° higher gave 33% conversion of gibbsite and silica sol to kaolin in 8 days and 75% conversion in 16 days. This is a very much faster reaction than that using silica–alumina gel.

Advantage was taken of the technique developed to prepare kaolin from silica–alumina gel in which deuterium would replace most of the hydrogen. The silica–alumina gel was dried at 500° and treated in the furnace with 99.9% D₂O at 300°. The infra-red absorption of the product was measured by Dr. V. C. Farmer (Macaulay Institute) as part of his work on the assignment of infra-red bands in sheet silicate structures. He found that replacement was nearly complete and the sample contained very little hydrogen. (Rayner.)

SPECTROCHEMISTRY

Trace elements in soil separates

Studies of the manner in which trace metals are combined in soils showed they are held in one or more of the following ways: (1) as water-soluble or exchangeable ions; (2) as integral constituents of primary and secondary minerals (including association with the sesquioxides); (3) as a non-exchangeable part of the organic matter.

Two well-drained brown earths (Charity and Hatfield series) were used, from which the samples were taken from the surface horizons enriched in organic matter and from the clay-enriched horizons. When the soils were dispersed in dilute ammonia solution and the fractions analysed the sums of the trace elements Ba, Co, Cr, Cu, Ni, Ti, V and Zr in the sands, silts and clays very nearly equalled the totals in the untreated soils. This indicated that only small proportions of the elements occurred in the soils as water-soluble or exchangeable ions. Dispersion with ammonia was incomplete, for much of the sand- and silt-size fractions obviously consisted of clay-size material cemented with iron and aluminium oxides. To remove these oxides before mechanical analysis, Tamm's oxalate method, as modified by A. S. de Endredy, was used, in which the extraction is done under ultra-violet irradiation. For trace-element work the method has the great advantage that the oxalate can be highly purified and is readily removable, so that both the extracted sesquioxides and the cleaned sands, silts and clays can be readily analysed. Unfortunately, part of the soil organic matter is also extracted, and it has not yet been possible to estimate directly the trace elements associated with it.

The experiments show that less than 1% of the silica of the soil was extracted, from which it was concluded that the treatment does not cause appreciable breakdown of the silicate minerals. Approximately 60% of the total iron and 10% of the total aluminium in the soil were removed as sesquioxides, and, as repeated extractions removed no more, the remaining iron and aluminium were assumed

to be constituents of the silicate minerals. The sesquioxide extract contained more than 90% of the Co, Cu and Mn, approximately 60% of the Ni, Pb and V, but less than 10% of the Ba, Cr, Ga, Sr, Ti, Y and Zr. Of the elements remaining in the soil after treatment, Co, Cu, Ga, Ni, Pb and V were greatly concentrated in the clay fraction. (Le Riche and Weir.)

Spectrophotometric determination of magnesium

As mentioned in last year's Report, a spectrophotometer for determining magnesium by the porous-cup technique was built in the laboratory by G. C. Dibley and R. Farrow. The machine is a copy of one at the Macaulay Institute, and we acknowledge the very generous help given by Drs. R. L. Mitchell, R. O. Scott, A. Ure and others at the Institute. In twelve months more than 3,000 determinations in soils and plants were made for the Soil Survey and the Chemistry Department. The apparatus gives a precision of $\pm 2\%$ per determination at an even temperature, but tedious recalibrations with standards are often needed in the winter when the laboratory temperature fluctuates widely. Attempts to stabilise the temperature have so far failed.

The rate of production of porous-cup electrodes was increased by using a machine for cutting carbon rods. It was built by the Workshop from plans drawn by B. Edwards from photographs of a similar machine at the Macaulay Institute. The new machine gives a smooth and reproducible cut and is quiet and dust-free in operation. It will handle all the carbon rods for spectrographic work. (Weir.)

The many soil and plant samples analysed for semi-major and trace elements included some for members of the department, other workers at Rothamsted, the Forestry Commission and the Colonial Office. Discoloured and stunted seedlings of Sitka Spruce and Lawson's Cypress growing near wire netting surrounding the seedbeds contained abnormally high zinc.

Samples of forest soils sent by the North Borneo Department of Agriculture contained high levels of total Co, Cr and Ni. (Burnett.)

SOIL CHEMISTRY

Determination of total sulphur in soils

The combustion method for determining total sulphur (*Rep. Rothamst. exp. Sta.* for 1960, p. 82) was adapted to the micro-scale appropriate for soils. The soil is heated with V_2O_5 in a current of nitrogen and oxidation of volatile organic compounds is completed by contact with hot CuO. The combustion products are passed over hot metallic copper to reduce oxides of nitrogen and to convert SO_3 to SO_2 , which is absorbed in a solution of sodium tetrachloromercurate and determined colorimetrically with *p*-rosaniline.

Methods based on the reduction of sulphate to sulphide are subject to interference from copper, but this appears to be without influence in the present method. 100 p.p.m. S may be determined accurately in the presence of a tenfold excess of Cl^- , PO_4^{3-} , NH_4^+ or NO_3^- .

Combustion with V_2O_5 also appears to offer a more rapid and convenient method than distillation with HBr/Br_2 for separating Se from soils. Preliminary results were encouraging, and the method is being further investigated.

Copper fixation by organic matter

The formation of a surface organic mat in an orchard with a long history of heavy spraying with copper fungicides was described in earlier Reports. The mat is 1–2 inches thick, and a bulked sample had a total copper content of 1,870 p.p.m. The following figures relate to the mineral soil immediately below the mat, and show that the copper content falls off very sharply:

0– $\frac{1}{2}$ inch	130 p.p.m. Cu
$\frac{1}{2}$ –2 inches	70 p.p.m. Cu
2–5 inches	45 p.p.m. Cu

About 5% of the total copper was extractable from the mat as collected, at pH 7, and some 30% was removed at pH 3; these values decreased when the mat was air-dried. The material can take up considerably more copper, but this is completely removed on extraction at pH 3, so that the copper-fixing capacity of the mat is saturated.

In an investigation of the effect of cultivation on the release of copper from the mat, aerobic incubation for one month did not change the amount of copper extracted at pH 7, but the amount removed at pH 3 increased by about 200 p.p.m. Incubation for a further period of one month slightly decreased the amount of acid-extractable copper, but the value remained higher than for the original material. The addition of subsoil, nitrogen and green manure slightly increased the amount of extractable copper, but in view of the low density of the mat, about 0.28 g. per cubic cm., it seems unlikely that ploughing it in would make the soil phytotoxic. Experiments to study this point are in progress.

More than half the copper contained in the mat is removed by extraction with a mixture of ethanol and benzene, and the copper thus extracted includes essentially all that removed by ammonium acetate at pH 3. An artificially copper-saturated phragmites peat behaved similarly. As peat waxes are acidic in nature, the solvent-soluble copper is probably combined with these compounds and is present as a metallic soap. (Bloomfield and Pruden.)

APPARATUS

Electron probe

The preliminary work on the electron probe reached a stage which justified the design and development of a practical model. It has been decided to accept an electron beam of 3 μ until the need for a smaller one becomes apparent; the larger beam is likely to be more stable and permit steadier operating conditions. Geiger-counter equipment and X-ray crystal plates were obtained, and the apparatus is being constructed to enable samples to be examined by one of several techniques—emission, absorption or microscopy. The specimen chamber is designed to take 3 specimens, 18 standards and 18

X-ray transmission targets, which can be interchanged, without releasing the vacuum, from outside the chamber. The specimen can be moved under the beam in the horizontal plane in steps of 5μ , enabling any part of the specimen of $\frac{3}{4}$ inch diameter to be examined. The vacuum system was modified to increase pumping speed and to improve the vacuum in the radiation chamber.

An evaporating plant was built for making X-ray transmission targets for use with the electron probe. It is designed to enable targets to be made by evaporating material on to "Melinex" sheet, and the target material is of the order of 1μ thick.

Several prototypes of Cartesian divers were made for C. L. Bascomb, as well as a tank to determine the density of clay suspensions by means of a liquid density gradient.

The quartz crystal monochromator was completely redesigned to make adjustments easier and more effective, and also to enable the monochromator to be used with an ordinary 9-cm. powder camera. (Dibley.)