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PEDOLOGY DEPARTMENT

By A. MUIR

During the year Mr. J. R. Butler was appointed to the spectrographic section. Mr. K. Norrish of the Waite Institute who has come for two years' study and Mr. J. Garcia Vincente from Madrid, are carrying out X-ray studies on soils from their respective countries. Dr. E. M. Chenery completed his period of research under the Colonial Development and Welfare Research Scheme and has now taken up a post in Uganda.

Two members of the department (Dr. D. M. C. MacEwan and G. Brown) have between them taken a considerable share in the compilation of the Clay Minerals Group monograph on "The X-ray Identification and Structure of Clay Minerals," which is to appear in 1951.

WEATHERING OF ROCKS AND MINERALS

The study of the weathering of the crystalline rocks of the Malvern complex is now essentially complete and has shown strikingly the effect of parent bed-rock in determining the secondary products, i.e. the mineralogical composition of the clays of the derived soils. The results show the dominance of clay minerals of the chlorite-vermiculite crystallization associated with the ultra-basic rocks rich in hornblende and biotite, and of an illitic clay mineral with the granitic rocks. In the "Ivy Scar Rock" (a granophyric quartz diorite) of intermediate composition both types of clay minerals are present in the soil clay. The other crystalline constituents of the soil clays are kaolin and quartz, which are particularly associated with the more acid rocks. Iron and aluminium oxides are present in all the clays, but are mainly in the amorphous state.

A similar comparative study of the mineralogical characteristics of soils from the Gold Coast derived from acid and basic rocks has been started.

CLAY MINERAL STUDIES

Techniques

There has been considerable call on the X-ray apparatus for use as an auxiliary to petrological and other studies. It has thus been necessary to give much attention to the question of reducing exposure times for X-ray photographs. The question has been approached from several different directions.

(1) The use of a larger tube current with the usual size of focus is precluded by the rate of heat dissipation at the anode, but with the slit collimation we employ it has been found that perfectly sharp photographs can be obtained from a much larger focus than usual, which permits passage of a larger current and hence shorter exposures.

(2) Substitution of detachable windows sealed with rubber O-rings enables the filter material, which is in any case necessary, to be used as a window instead of aluminium and so eliminates

absorption from this source. The camera windows have been replaced by 0.3 mm. polythene, which, unlike cellophane, seems to be unaffected by X-rays.

(3) Experiments have been made with a camera slit system made of metallized glass plates. Such a system reflects X-rays which strike it at a narrow angle and so gives an enhanced intensity. The system we use gives a gain of about 2x, but appears to give rather broader lines than the ordinary collimating system, which may be due to the poor optical quality of the glass used, together with inaccuracies in assembly.

(4) The use of Zelger's copper intensifier on the film gives an improvement in contrast as well as intensity. The complications of the procedure, however, are such that it will not be applied unless a number of films are processed together, or it is essential to have an exposure of minimum length.

The total reduction of exposure time by a factor of 6 may be achieved by these methods, and it is now possible to obtain a well-exposed photograph in 10 minutes.

Quantitative determination of clay minerals

Preliminary calculations have been made on a method based on the use of oriented aggregates for the accurate determination of clay minerals in mixtures by X-rays, and experiments are being carried out. The problem is not a simple one, but this method seems to hold out considerable hope of an eventual solution. It has been described in a preliminary communication to the Clay Minerals Group. Arising partly out of this, extensive calculations have been made on the effects of orientation and absorption on the intensities of X-ray reflections from oriented aggregates of clays.

Clay mineralogy of rocks and soils

(1) In collaboration with W. N. Croft of the Imperial Institute an investigation has been made of the clay mineral content of Devonian siltstones from North Wales. The problem was to find if the break between Upper and Lower Devonian (several million years) was reflected in the mineral composition. A break was found, there being chloritic material present in the Lower Devonian which was almost absent in the Upper Devonian. A series of Brecon siltstones is now being examined to see if a similar break can be traced.

(2) A number of soils from the Kongwa groundnut area have been examined to see if some indication could be got of the reason for the strong compaction occurring after rain. The general properties of the soils that show their characteristics, the red loam in particular, correspond to those of comparable soils from other parts of Africa, i.e., low silt content, low base exchange capacity. The clay minerals in the red loam and upland pallid soils proved to consist mainly of halloysite with a little mica, quartz and iron oxides. The dark valley soil clay consisted mainly of mica with some halloysite as accessory. The sand fractions consisted almost entirely of angular quartz fragments. There is nothing in the results to suggest that there is anything inherently peculiar about

the Kongwa soils, but samples from other groundnut areas are now available so that the investigation can be extended.

(3) A wide range of soil clays from Syria have been studied with interesting results. It was found that the mineral attapulgite was a common and sometimes abundant constituent of the soils derived from the sedimentary rocks of the desert. The *terra rossa* soils of the Mediterranean coastal area contained mainly a "mellorsitic" type of clay mineral. The clay minerals of the basalt soils are apparently connected with the degree of crystallinity of the rock; the highly crystalline rocks yield halloysite on weathering, whereas the glassy types of basalt give beidellite.

(4) The department has now a considerable collection of bentonites, and alleged bentonites, from various sources, as well as other clay materials, and X-ray photographs have been taken with a view to establishing a series of standard patterns likely to be of use in dealing with soil clays.

The examination of a series of Punjab clays from Dr. J. N. Mukherjee showed that some were kaolinites of quite exceptional purity, the others containing a montmorillonite impurity.

Among other materials examined by X-ray methods were soil clays from Uganda, Iraq, Persia and Ireland.

Location of the sorbed ions and molecules in montmorillonoids

This investigation has now been completed for both montmorillonite, the dioctahedral montmorillonoid, and hectorite, the trioctahedral member. The results for both of these support the Hofmann structure for the minerals and show that the exchangeable cations take up a position midway between the inorganic layers of the mineral in their glycerol complexes.

Complexes of montmorillonoid clays with proteins

Using oriented flakes of clay particles ($< 1\mu$) a new phenomenon has been observed: one-, two- and four-layer complexes have been detected with gelatin, edestin and pepsin suspensions according to the concentration and pH of the suspension: each layer has a thickness of 4.3 to 4.5 Å, which corresponds very closely to the calculated and directly observed thickness of single polypeptide chains in proteins. This implies a competition between the surface energy of the inter-lamellar spaces of montmorillonite and the lattice energy of the hydrated protein crystals. Furthermore, the most stable two- and four-layer complexes are formed at nearly 2 pH units below the pI of the protein, which strongly suggests the mechanism of proton transfer being necessary for these complexes.

Complexes of montmorillonoids and pyridine

A systematic investigation of the interaction of pyridine and other polar molecules with montmorillonite is in progress. Three main methods are being used. First the measurement of the sorption isotherms in the vapour and the liquid phases. Secondly, the study of the resultant complexes by X-ray diffraction and in certain favourable cases the application of one dimensional Fourier synthesis.

Thirdly, the use of the differential thermal method to study the evaporation of the sorbed material.

The results so far obtained indicate that the sorption isotherm of pyridine is dependent on the water content of the system, the nature of the base exchange ion in montmorillonite, and the previous heat treatment of the mineral.

Several other complexes likely to be of interest have been examined by X-ray diffraction. The blue and green colours formed by certain amines, notably dimethylaniline, with montmorillonite are being investigated. Results suggest that the colour formation is independent of the extent of interlaminar sorption.

Autunite

The mineral autunite, a calcium uranyl phosphate $\text{Ca}(\text{UO}_2)_2(\text{PO}_4)_2 \cdot n \text{H}_2\text{O}$ is a layer type mineral which shows reversible hydration properties between the states $\text{Ca}(\text{UO}_2)_2(\text{PO}_4)_2 \cdot 10\text{H}_2\text{O}$ called autunite and $\text{Ca}(\text{UO}_2)_2(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$ called meta-autunite-1. The spacing of autunite is $10 \cdot 32 \text{ \AA}$ and of meta-autunite-1 $8 \cdot 42 \text{ \AA}$. The structure of the layers of oxygen atoms in autunite is similar to the structure of montmorillonite suggested by Edelman. It was thought that the autunite might be made to expand with organic liquids such as glycerol in the same way as montmorillonite.

The mineral was therefore synthesized and various treatments were tried, either adding the organic liquid to the freshly prepared autunite or precipitating the autunite in the presence of the organic liquids. No expansion was shown in any case. In fact, most of the treatments merely dehydrated autunite (the $10\text{H}_2\text{O}$ state) to meta-autunite-1 (the $8\text{H}_2\text{O}$ state). These changes were all followed by means of X-ray photographs.

SPECTROGRAPHIC WORK

Work has been continued on the quantitative determination of molybdenum in soils and methods have been studied for other elements. The Mannkopff spectrograph has been found suitable for the quantitative estimation of potassium in solid specimens. Quantitative estimations have also been made of the major elements (i.e. Al, Ca, Fe, K, Mg, Mn, Na) in minerals, rocks and plant ashes. In addition, semi-quantitative methods are in use for about thirty minor elements.

The geochemistry of clays and soils is being investigated by these methods and it is hoped eventually to supplement the results by micro-chemical determinations of, more especially, zinc.

In the determination of exchangeable cations in soils, an alternative has been adopted by several workers to the standard method of leaching the soil on a filter with successive portions of ammonium acetate. This consists of allowing the solution to percolate through the soil in an automatic apparatus. The method is being investigated and an apparatus has been devised which is considerably simpler in design than some that have been described. The results obtained are in good agreement with those obtained by the leaching method and it is hoped to adopt the method for routine use.

GLEYING IN SOILS

As reported previously, it has been found possible to induce gleying artificially in soil material by the action of anaerobically fermenting plant material. Under these conditions ferric oxide undergoes extensive solution and reduction, leaving an essentially iron-free residue. In this respect the laboratory experiments do not suggest a close parallel with the natural process since such analytical data as are available in the literature suggest that, in the field, gleying does not result in a marked diminution of the total iron content. However, it has been found that ferric oxide, either alone or as present in soil, has the property of abstracting ferrous iron from solution in an irreversible manner; if the ferric oxide with its associated "fixed" ferrous iron is completely dissolved in acid it is found that little or none of the "fixed" iron is present in the ferrous state. It is suggested that the explanation of this is as follows: an initial adsorption of ferrous iron as the ferric oxide takes place, and in the adsorbed state the Fe^{2+} is sufficiently readily oxidised for this to be effected rapidly by dissolved oxygen in the system. It seems probable that this phenomenon is responsible for the differences between the laboratory and natural processes mentioned above.

It has been demonstrated that gleying is not necessarily caused by direct microbial action; by treating a clay suspension with a fermented grass extract under sterile, anaerobic conditions, a gleying action is produced which, although less extensive than in the fermentation experiments, is nevertheless appreciable.

Extracts of fermented grass have been shown to cause an almost instantaneous reduction of dissolved ferric iron; determination of the ferric-reducing capacities of such extracts gives theoretical values for the extent of gleying action which exceed the experimental results by a factor of 3 to 4. This, it is felt, indicates that the rate-determining stage in the overall process of solution and reduction is the initial solution of ferric oxide to give soluble ferric compounds, reduction of which takes place almost immediately.

Fermentation of grass in the presence of ferric oxide gives solutions in which the iron appears to be present entirely as complex ferrous compounds. More than one type of complex combination appears to be involved. Part of the iron is readily precipitated by atmospheric oxidation of the solution, or by an increase in the pH; the iron remaining in solutions is not precipitated even after prolonged exposure to the air at pH9, and under these conditions oxidation of the Fe^{2+} is so slow that it is not possible at present to say with certainty whether oxidation does indeed take place.

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