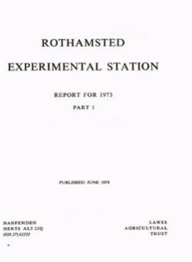
Report for 1973 - Part1

Full Table of Content



ROTHAMSTED

EXPERIMENTAL STATION

REPORT FOR 1973

PART 1

PUBLISHED JUNE 1974

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Pedology Department

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C. Bloomfield (1974) *Pedology Department ;* Report For 1973 - Part1, pp 67 - 78 **- DOI: https://doi.org/10.23637/ERADOC-1-130**

PEDOLOGY DEPARTMENT

C. BLOOMFIELD

This year our mineralogical work included studies of soils from western Nigeria, and sediments from the Nile Delta. An interstratified expanding clay from Belgium and a saponite-like mineral from South African mining slimes were also studied. Our study of loess was extended to eastern Yorkshire and Lincolnshire. Existing chemical methods for determining basic aluminium layers in soil clays were shown to be unsatisfactory.

Although storm run-off from the M1 in Leicestershire was found to contain appreciable amounts of lead and zinc (up to 3000 and 880 ppm respectively), the metals are almost entirely in particulate form, and very small amounts are in true solution. It is possible that some of the particulate matter is deposited in the Blackbrook reservoir, and the possibility of its subsequent dissolution causing the contamination of drinking water remains to be investigated. Soils subjected to frequent flooding by the Trent contained significantly larger amounts of chromium, copper, nickel, lead and zinc than similar unflooded land.

Work on the geochemistry of soils of the Denchworth series and of north Pembrokeshire was continued, and advantage was taken of Rothamsted's collection of soils sampled during the last 100 years to assess the effect of road traffic on the lead contents of our soils. Although the lead content of Rothamsted soils has increased, there is no apparent increase in the amount contained in the herbage.

Our work on the effects of fumigation on soil metabolism is almost completed. Much of our effort in this field has been devoted to developing a new method of estimating the amount of living tissue in soil, and adapting the method for routine use. Work on the radiocarbon dating of soil organic matter continues; measurements made this year show that in Rothamsted soils the concentration of radiocarbon derived from the bomb tests of the early 1960s has now passed its maximum.

We have continued our study of trace metal–organic matter interactions, and have made an initial study of the chemical behaviour of toxic metals in contaminated sewage sludge. It seems that the validity of the results given by the current empirical methods used to assess the availabilities of the various metals is subject to considerable doubt.

Mineralogy

Interlayering in soils and clays. Acidity in soils often originates from sorbed weak bases rather than from free hydrogen ions, and is usually associated with exchangeable or non-exchangeable aluminium; hydrogen ions are released when the acid-base equilibrium is displaced so that the weak base hydrolyses. Basic aluminium cations replace exchangeable cations in montmorillonite, but as the structure of such aluminium clays is irregular, their detection in soils by X-ray diffraction is often uncertain. As an alternative approach we have assessed the value of chemical methods for determining interlayer aluminium; treatments that have been proposed for this purpose include extractions with sodium citrate at 100°C and with cold $CaCl_2$ at pH 1·5.

In preliminary experiments we investigated the chemical and structural changes that result from these extractions; as clay fractions were required for the structural examinations, we also studied the changes that occur during the pretreatments recommended for dispersing clay fractions (decalcification with Na acetate at pH 5, oxidation of organic matter with H_2O_2 and dissolution of free iron oxides after reduction with sodium dithionite). An acid soil from Sawyers field (pH 4·4) was used; this contains little exchangeable Al and might therefore be expected to contain basic aluminium interlayers.

The elements dissolved at each stage in the procedure were determined, as well as the chemical composition of the clays; the structural changes were examined by X-ray diffraction.

The structure of the clay was apparently little changed by the treatments; observed changes in the diffraction patterns were attributed to changes in the proportions of the constituent minerals rather than to structural changes.

The main chemical effects of the treatments were as follows: (1) Na acetate (pH 5) dissolved more than half the total Mn in the soil, but little else, (2) oxidation of the organic matter with H_2O_2 decreased the exchange capacity, (3) dithionite dissolved 65% of the total Fe and most of the remaining Mn, (4) 0·5*M* $SrCl_2$ (pH 1·5), which was used in place of $CaCl_2$ so that changes in the Ca content could be measured, extracted more Al than Fe, but the amounts were small compared with those dissolved by dithionite, and (5) sodium citrate dissolved Fe in amounts similar to those extracted by dithionite, and also substantial amounts of Si, Al and K, in proportions similar to those of the original clay.

We concluded that the pretreatments for dispersing the clay cause little bulk dissolution or structural change of the clay, although all dissolved a substantial proportion of the total Mn. At pH 1·5, $SrCl_2$ is relatively ineffective for extracting interlayer Al, but citrate, by contrast, is too destructive and dissolves both free iron oxides and whole clay minerals. Neither of these methods is suitable for determining basic aluminium interlayers in soil clays. (Henn and Newman)

An interstratified expanding clay mineral from Denchworth series soil in weathered Oxford Clay. Continuation of the work reported last year (*Rothamsted Report for 1972*, Part 1, 67–68) has shown that the very fine clay fraction ($<$0·04 μm) of a Denchworth soil contains a mineral that is a randomly interstratified mixture of non-expanding illite layers and expanding smectite layers. When the mineral is Ca-saturated and expanded with ethylene glycol, its X-ray diffraction pattern is best matched by that from a mixture of 55% illite and 45% smectite layers in diffracting particles containing on average five silicate layers each. This ratio of non-expanding to expanding layers can be reconciled with the proportions of non-exchangeable potassium to exchangeable calcium ions, found by chemical analysis, if it is assumed that the outer basal surfaces of the five-layer diffracting particles contain no potassium, and that the potassium content of the illite layers is about 70% of that of mica minerals.

Replacement of the exchangeable Ca-ions in the $<$0·04 μm fraction by bulky dimethylammonium ions, and analysis of the resulting diffraction pattern, confirmed the proportion of expanding layers found with ethylene glycol solvation. However, when dimethylammonium ions were exchanged into coarser clay fractions, the resulting diffraction pattern showed the presence of two expanding minerals. One was similar to that described above and probably comes from fine clay not removed in the separation process, but the other was smectite or vermiculite in which all the layers were expanded by dimethylammonium ions. These coarser fractions (0·04–0·2 and 0·2–2·0 μm) contain kaolinite and a separate illite phase in addition to the expanding minerals.

Fractionation of the $<$2 μm clay fraction thus shows that the Denchworth soil contains at least two K-containing minerals, illite and interstratified illite–smectite, and two expanding minerals, and that their relative proportions change with particle size. Such complex clay mineral assemblages are probably the rule rather than the exception for soils and sediments, a fact that needs to be considered when studying the uptake and release of plant nutrients and other ionic reactions in soil solutions. (Rayner and Weir)

Mineralogy of soils from Western State of Nigeria. The savanna region of the Western State of Nigeria, much of which has considerable potential for agricultural improvement, is some 39 000 km² in extent. The area is the subject of a Land Resources Study by the Overseas Development Authority, and mineralogical information is required to assist the evaluation of a soil survey that forms part of the Land Resources Study.

The minerals present in the $<2\ \mu$m fractions of 22 soil series were determined, mainly by X-ray diffraction. Although all the soil clays contain some 2 : 1 phyllosilicates, kaolin is usually dominant. Exceptions are the Origo series vertisol, and soils of the Dejo, Woro, Oyo and Pabiku series which have planosolic characteristics, in which swelling minerals are dominant; in an Ekiti series lithosol a mica is the dominant clay mineral.

The K_2O contents of selected samples were shown to be related to their mica contents, as determined by X-ray diffraction, which can therefore be used to estimate the potassium contents of the clays. The abundance of swelling minerals was shown to be related to the unexpectedly large cation exchange capacities of some of the sub-soil clays; soils that contain an appreciable proportion of swelling minerals are noticeably sticky in the field when wet. The contents of dithionite-extractable iron oxide range from 0·18% in the immature gley Ofiki soil to about 10% in the mature Makun and Olorunda series soils; the amounts of silica and alumina extracted by sodium hydroxide are least in the Ofiki and Ekiti soils. (Brown and Ormerod)

Mixed layer montmorillonite–chlorite from Huy, Belgium. This material, which was briefly mentioned previously (*Rothamsted Report for 1970*, Part 1, 65) has been studied in detail. Preliminary studies on impure material indicated that the mineral was a dioctahedral mixed-layer montomorillonite–chlorite. Few examples of minerals of this kind have been examined in detail. By careful particle size separation a purified sample was obtained that was estimated to contain 1·3% of a kaolin mineral, probably nacrite, and 0·5% quartz as almost the only impurities. X-ray powder diffraction confirmed that the mineral was a regularly interstratified montmorillonite–chlorite. In the air-dry state basal spacings of 28·9 Å and 26·6 Å were obtained from the Mg- and Na-saturated material respectively. These spacings were altered to 31·1 Å, 30·0 Å and 32·1 Å after treatment with ethylene glycol, ethylene glycol monoethyl ether and glycerol respectively; there was no significant difference in spacing for these organic complexes of Na- and Mg-saturated materials. Heating to 300°C decreased the basal spacing to 23·8 Å. The spacing of the 06 reflection was 1·506 Å.

Chemical analysis of Mg-saturated clay gave the percentage composition: SiO_2 39·74, Al_2O_3 35·87, TiO_2 0·01, Fe_2O_3 0·98, FeO 2·77, MgO 3·08, CaO 0·06, Li_2O 0·51, Na_2O 0·12, K_2O 0·62, P_2O_5 0·10, H_2O 15·70, total 99·56%. The cation exchange capacity of the air-dry Mg-saturated clay was 49 meq/100 g. The structural formula calculated from the chemical analysis is:

$$(\mathrm{Mg}_{0\cdot48}\mathrm{K}_{0\cdot26}\mathrm{Na}_{0\cdot08}\mathrm{Ca}_{0\cdot02})(\mathrm{Si}_{13\cdot03}\mathrm{Al}_{2\cdot97})(\mathrm{Al}_{10\cdot88}\mathrm{Fe}\,\frac{3+}{0\cdot24}\,\mathrm{Fe}\,\frac{2+}{0\cdot76}\,\mathrm{Mg}_{1\cdot02}\mathrm{Li}_{0\cdot67})\,\mathrm{O}_{40}(\mathrm{OH})_{20}$$

The total number of octahedral cations, 13·57, exceeds the 12 required if each octahedral layer were strictly dioctahedral.

Further study of similar minerals is required to establish the compositional range of aluminous mixed layer montmorillonite–chlorites before names are given to species. The presence of appreciable lithium in this sample suggests that this element should be determined when these minerals are analysed. Until more work is done this mineral is best described as a lithium-bearing aluminous regularly interstratified montmorillonite–chlorite. (Brown, with Professor P. Bourguignon and Dr. J. Thorez, University of Liege.

Effect of heating on the specific surface area of soils. Soils are sometimes heated to eliminate the effect of organic matter when measuring their surface areas. The surface area of several soils was measured after heating in the range 125–800°C; for most soils there was a discontinuous decrease in area, but for some there was a small increase between 200 and 275°C. As the surface area of an ironstone soil increased by 35%, it seemed probable that the increase in surface area is caused by changes undergone by iron oxide when these soils are heated. When heated to 275°C the surface area of a synthetic geothite increased from 50 to 113 $m^2 g^{-1}$, and decreased to 27 $m^2 g^{-1}$ after heating to 600°C. The surface area of a soil of the Tadmarton series, formed on Northampton iron-rich sandstone, had a surface area of 84 $m^2 g^{-1}$ after heating at 175°C, which increased to 116 $m^2 g^{-1}$ after heating at 275°C and decreased to 60 $m^2 g^{-1}$ after heating at 600°C. The same soil, after treatment with sodium dithionite, which removed 37·5% iron oxide, had surface areas of 91 $m^2 g^{-1}$ and 88 $m^2 g^{-1}$ after heating at 175 and 275°C respectively.

X-ray diffraction examination of the goethite and the soil before and after heating showed that the increase in surface area after heating to 275°C is accompanied by the transformation of goethite to a very poorly crystallised form of hematite. Heating to 600°C leads to growth and increased perfection of the hematite crystals. The increase in surface area on heating to 275°C is therefore attributed to the transformation of goethite to poorly crystalline hematite with a large surface area, and the decrease on further heating to growth of the hematite crystals. (Brown and Ormerod, with Pritchard, Soil Survey)

A saponite-like mineral with small layer charge. An unusual saponite-like mineral has been concentrated from slimes obtained in mining South African kimberlites. The mineralogical study was made because of the great difficulty encountered when it was attempted to remove solids from the slimes by flocculation. It was thought that unusual colloidal properties of the clay mineral might be the cause.

The slimes contained particles up to 5·5 μm equivalent settling diameter, and the bulk material was shown by X-ray diffraction to consist of about 50% of a trioctahedral smectite with talc and chlorite. Most of the material was found to consist of particles in the $<0{\cdot}2$ μm fraction. The smectite was concentrated in the fine fraction, but this still contained talc and chlorite impurity. The $<0{\cdot}2$ μm fraction was size-fractionated by repeated dispersion and centrifugation. The finest material was dark green in colour and almost free of impurities; reflections from talc and chlorite were just detectable.

Chemical analysis of the purified material in the Na-saturated form gave the percentage composition (ignited weight basis) SiO_2 54·50, Al_2O_3 10·19, TiO_2 0·05, Fe_2O_3 5·75, FeO 3·61, CaO 0·03, MgO 22·87, MnO 0·10, K_2O 0·27, Na_2O 1·59. The cation exchange capacity of the material, after drying at 110°C, was 49 meq/100 g. The smectite-like mineral gave a basal spacing of 12·8 Å when Na-saturated material was examined in the air-dry state. Treatment with ethylene glycol gave a pattern showing 11 orders of a mean basal spacing of 16·86 Å, the values obtained from individual reflections ranging from 16·6 to 17·6 Å. Treatment with glycerol gave a similar pattern, with a mean basal spacing of 18·1 Å with a range of 17·6–18·3 Å. The Na-saturated clay lost interlayer water and collapsed to give a 9·6 Å basal spacing after heating to 80°C for 2 hours. Further heating up to 500°C did not produce any change in the diffraction pattern. The 06 spacing of 1·536 Å confirmed the trioctahedral character of the smectite.

Although both ethylene glycol- and glycerol-treated specimens gave a near-rational series of basal reflections, these were not all equally sharp, so that there may be a small amount of interstratification of layers other than smectite. These are probably talc-like because the ready collapse to 9·6 Å at 80°C and the small amount of K_2O argue against there being an appreciable number of chlorite or mica layers. The mineral seems to be saponite-like, with an unusually small negative layer charge, and possibly a small proportion of interstratified talc-like layers. (Brown and Ormerod)

Clay mineralogy of sediments of the Nile Delta. The building of the Aswan Dam stopped the seasonal accumulation of silts in the Nile Delta. Since the completion of the project the lower Nile has flowed silt-free and the Delta is being actively eroded along its Mediterranean coast. As part of a large study of recent changes in the Delta and its sediments we have examined some 50 samples taken from the bed of the mouth of the West Rosetta branch of the Nile, offshore samples from the Mediterranean Sea near Abou Qir Bay and from seven boreholes from the delta itself between Abou Qir Bay and Lake Idkou. The cores from the boreholes, which were sampled to a depth of 23 m, consist of unconsolidated muds and sands ranging from 2 to 74% of <2 μm clay, average 46%, and from 1 to 91% of 63–2000 μm sand, average 28%. The frequent large variations of the textures of the samples with depth is typical of delta sedimentation where meandering rivers continually sort and re-deposit the sediments. Several samples contained peat, and most were calcareous, the calcium carbonate contents tending to increase towards the west.

In contrast to the large variation in texture, the mineralogy of the <2 μm clay fractions showed comparatively minor variations. The clays are dominated by expanding minerals, ranging from 65 to 85%, with minor amounts, 10–20%, of kaolinite. The contents of illite, 5–15%, and of chlorite, 0–13%, are somewhat more variable than for the other two minerals.

The expanding minerals were studied by saturating them with Ca- or Mg-ions, and expanding them by solvation with ethylene glycol or glycerol. They vary in composition from smectite–illite to smectite, and many, including the Nile samples, also contain a little vermiculite. The Nile samples and those from the shores of the Mediterranean Sea cannot be distinguished from the borehole samples in terms of their clay mineralogy.

The composition of the expanding minerals was investigated in more detail by separating four samples into $<0{\cdot}04$, 0·04–0·2 and 0·2–2 μm fractions and applying the techniques developed for the Denchworth soil described above. The finest clay fractions of two of the samples contain almost pure iron-rich (18% Fe_2O_3) smectite of a very small particle size. The third sample contains a randomly interstratified 70 : 30 smectite–illite, and the only Nile sample investigated an 85 : 15 smectite–illite. Vermiculite occurs only in the coarser clay fractions of these samples.

The clay minerals in the Nile Delta show strong similarities to soil clays and do not appear to have been altered following burial or deposition in saline water. The relatively minor variations in clay mineralogy that we observed probably result from seasonal variations in the amounts of erosion that occur between different regions of the head waters of the Nile and its tributaries. (Weir, Ormerod and El Mansey)

Loess in soils of eastern Yorkshire and Lincolnshire. Silty and often very flinty brown earths >60 cm deep over Chalk (the Wold series) have been mapped on plateau surfaces in the north-west part of the Yorkshire Wolds (Matthews, B. 1974, Soils in Yorkshire IV, Sheet SE 76 Westow; *Soil Survey Record No. 23.* In the press). A rapid survey of other likely areas showed that most plateau surfaces west of the Devensian till margin have similar soils; also, although much thinner soils are typical of the steeper Chalk slopes, thick accumulations, often $>1{\cdot}5$ m, of silty colluvium occur on and near valley floors. However, on the Lincolnshire Wolds, only one plateau surface, near Binbrook, had $>0{\cdot}3$ m of the silty drift. On the Hambleton Hills in north-east Yorkshire, podzols are developed in another silty deposit, which is approximately 1·0 m deep over the Corallian Hambleton Oolite Limestones.

The detailed particle size distribution and mineralogical composition of coarse silt (20–60 μm) fractions of samples from the Lincolnshire and Yorkshire Wolds and the Hambleton Hills show that, as in the silty soils of north Norfolk (*Rothamsted Report for 1971*, Part 1, 79–80), most of the soil material is loess derived from the debris brought by the Late Devensian glacier, approximately 18 000 years ago. However, the amounts of sand and clay in some of the soils are slightly greater than in typical loess, and may reflect minor incorporation of weathered remnants of pre-Devensian glacial deposits, and the illuvial accumulation of clay in subsurface horizons.

On the Yorkshire Wolds the silty drift extends beneath the feather edge of the Devensian boulder clay as a very chalky, flinty, silty head deposit, which is variable in thickness, reaching >6 m at Sewerby above the Ipswichian marine interglacial deposits. This head was formed just before the ice reached its maximum extent, when the loess was deposited on, and mixed with broken and disturbed Chalk material on the periglacial land surface. Although it is now preserved only beneath the protective cover of relatively impervious boulder clay, it once extended over the whole Chalk outcrop, and the silty, flinty soils beyond the edge of the boulder clay are a decalcified and somewhat weathered residue of it. Similar silty head deposits probably once covered other limestones within the area of loess deposition, for example the Hambleton Oolite.

In Northern England generally, loess-containing soils are almost exclusively restricted to the main limestone outcrops. As loess must have been deposited indiscriminately on all substrata, this distribution is best explained as the result of selective removal of loess from surfaces other than limestones, probably by extensive run-off and soil erosion during wetter periods since deposition. The loess on limestones was possibly stabilised during such periods by cementation with secondary carbonate derived by dissolution of the abundant fragmentary limestone material in the head deposits. (Catt and Weir)

Soil geochemistry

Motorway contamination of reservoir water. We were asked by The Trent River Authority to examine samples of water feeding into the Blackbrook reservoir (Leics.) for possible metal contamination from motorway run-off, which carries appreciable amounts of particulate material into the Blackbrook during heavy rain following dry weather. Samples, which included material in suspension, were taken from the stream at different times at points upstream and downstream of the inflow from the motorway drain. Analysis of the separated solids of the water showed that whereas the amounts of heavy metals in true solution were small (Pb 0·004, Zn 0·1 ppm), the solids contained large quantities of these metals. Those sampled from the motorway drain contained upwards of 3000 ppm Pb and up to 880 ppm Zn, compared with <500 ppm Pb and 250 ppm Zn in mud collected in the water upstream of the drain. It therefore appears that metal-contaminated solids are carried into the reservoir, where presumably they deposit on the bottom. Further work is envisaged to see what danger there is of the metals subsequently dissolving and passing into the water supply. (Bloomfield, Le Riche and Pruden)

Soil pollution by flood waters from the River Trent. Several soils subject to frequent flooding by the Trent and suspected of consequent metal contamination were compared with soils of the same series (Wharfe, Trent and Fladbury) that are rarely flooded. There were substantial enhancements, especially in surface horizons, in the total amounts of Cr, Cu, Ni, Pb and Zn in the flooding soils (Table 1). Similar enhancements also occurred in the amounts of the metals extractable by N/2 acetic acid, which indicates an increase in the amounts available for uptake by plants. (Le Riche, with A. J. Thomasson and M. J. Reeve, Soil Survey)

TABLE 1

Total amounts of metals as ppm in oven-dry soil

		Non-flooding soils							Flooding soils						
Series		Co	Cr	Cu	Mn	Ni	Pb	Zn	Co	Cr	Cu	Mn	Ni	Pb	Zn
Wharfe	10–15 cm	9	32	30	1300	23	59	180	25	68	500	2200	53	220	1500
Wharfe	35–40 cm	10	39	23	1100	24	43	140	19	29	110	1700	38	190	470
Trent	10–15 cm	14	52	45	1900	37	94	250	14	78	310	1100	58	180	430
Trent	35–40 cm	18	51	30	3100	39	77	130	12	50	46	1600	39	93	260
Fladbury	10–15 cm	19	59	45	1100	45	209	180	17	120	200	990	70	160	450
Fladbury	35–40 cm	15	82	38	820	51	120	87	12	73	30	1400	27	68	140

Major and minor elements in the profiles of the Denchworth soil series. Major and minor elements in 30 profiles of soils from two areas that have been mapped as Denchworth series were determined by X-ray fluorescence analysis. The soil profile similarities calculated from these measurements ranged from 93·2% for two soils both developed on Kimmeridge Clay near Compton Beauchamp in Berkshire, to 68·5% when one of these soils was compared with one developed on Oxford Clay near Grendon Underwood in Buckinghamshire. The group of soils on Oxford Clay can be distinguished from the other soils by several different mathematical treatments of the table of similarities, but the other soils could not be divided into two clearly separated groups. Principal coordinate analysis shows that a first vector divides the soils into an Oxford Clay group accompanied by four of the Kimmeridge Clay soils; the second and third vectors can be combined to divide the Kimmeridge from the Gault Clay soils, but the two groups remain in contact, which suggests a continuous gradation of properties between the two groups. As well as this study of the relationship of the soils, the same table of measurements was used to find relationships between the elements present in the soils as a whole, and in the separate horizons. Principal coordinate analysis of the correlations between the elements (calculated by equating the range of correlation coefficients from +1 to −1 to a similarity range of 1 to 0) picked out four main groups: elements associated with (1) the organic matter (as assessed by loss on ignition) (2) the clay fraction ($<2\ \mu$) (3) the silt fraction ($50\ \mu$–$2\ \mu$) (4) calcium carbonate (Table 2). The association obtained depends on the tendency of the properties to occur for the same sample—a close chemical association may be responsible and is being sought by separation of the soil into fractions, but a

TABLE 2

The associations of major and minor elements down 30 Denchworth profiles

Horizon	Silt	Clay	Loss on ignition	$CaCO_3$
All four	Si–Zr	Al, K, Mg, Ni, Cu, Rb	Br, Pb, P	Ca, Sr
A horizon (1)	Si–Zr	Al, K, Mg, Ni, Cu, Rb	Br, P, S, (Ca)	
(2)	Si–Zr	Al, K, Mg, Ni, Cu, Rb	S (Br)	
B horizon	Si–Zr	Al, K, Mg, Ni, Cu, Rb, Zn	Clay	Ca (pH)
C horizon		Al, K, Mg, Ni, Cu, Rb, P		Ca, Sr

common displacement by a third element may sometimes also be the cause of association. The associations change with horizon, as different soil characteristics become predominant. P and S are grouped with the organic matter in the two upper horizons, where organic matter exerts a strong influence on the soil composition, but the effect fades lower in the soil, where organic matter is a less important component, and in the lowest horizon P is associated with the clay minerals. (Carolyn Williams and Rayner)

The distribution of major elements between size fractions of a clay soil. The study of major and minor element distributions in soils can suggest relationships with various soil fractions and components. To test some of these relationships, a core of a calcareous Denchworth soil (0–100 cm) was separated into horizons and each horizon separated into coarse and fine sand, silt, and coarse and fine clay. The samples were dispersed ultrasonically, as dispersion with Calgon would cause contamination by phosphorus and sodium. The fine clay was flocculated with the organic flocculating agent polyacrylamide, and the clay fractions of the calcareous horizons were decalcified with solid CO_2, under pressure; water- and carbonic acid-soluble fractions of the elements would be lost by this process.

Very little sand was recovered from the clay soil we studied, but major element analysis of the silts, coarse clays and fine clays of this profile showed trends down the profile, and with particle size. The silt fractions consisted of 55–86% SiO_2; the SiO_2 content fell as CaO increased in the calcareous horizons, and TiO_2 also accumulated in both the silt and coarse clay. Phosphorus also accumulated in the coarse clay in the A horizon although it was fairly evenly distributed throughout the three fractions. Fe, K, Al, Mn and Mg were all concentrated in the fine clay. Variations in the amounts of the elements down the profile showed a strong relationship in the movement of Si, Al and K in the fine clay fraction, reflected to a lesser extent in the coarse clay. A marked decrease of these elements in the fine clay at the top of the B horizon coincided with a strong accumulation of Fe. Although it showed the same distribution as Si in the silt fraction, Ti showed a similar pattern to P and Ca in the fine clay. The increase in the P content in the C horizon is reflected in the composition of the whole soil. Work is continuing on the determination of the minor element distribution in the same profile. (Carolyn Williams)

The distribution of trace elements in the soils of Northern Pembrokeshire. Thirteen elements have been determined by X-ray fluorescence spectroscopy in soils of North Pembroke that were sampled on a grid basis by the Soil Survey of England Wales. Distribution maps show that the elements can be divided into groups. Rb, and to a certain extent Fe, Cu and Ni can be related to the geology of the area, very much larger amounts being found in areas of Silurian shales than in areas of igneous rocks. Pb and Br are related to the loss on ignition of the soils, whereas Ga, Sr, Y are all concentrated in the eastern mountainous part of the area. Small contents are consistently related to poor drainage, and in the subsoil Mn, Cu, Ni and Zn show similar distribution patterns. Contamination by Pb from major roads in the area is not apparent in the Pb distribution map. (Carolyn Williams, with C. C. Rudeforth and R. I. Bradley, Soil Survey)

The regional distribution of sulphur in the soils and herbage of North Pembrokeshire. The soils of Pembroke have been sampled on a regular 1 km grid to a depth of 10 cm and 50 cm (Rudeforth and Bradley, *Rothamsted Report for 1970*, Part 1, 287) by the Soil Survey of England and Wales. We have determined S in soil samples from the northern half of the area, sampling every other soil from the 1 km grid. The S contents of the soils vary from 200–10 000 ppm in the top soil, and 115–3570 ppm in the subsoil. Ninety per cent of the topsoils we examined had less than 1200 ppm S and 15% loss of weight on ignition, but large sulphur contents of soils were associated with larger losses on ignition and, hence with larger organic carbon contents.

Two hundred samples of herbage collected from the same grid sites as the soils were analysed. Herbage was collected from the whole area during July, and, to study the seasonal variation in sulphur uptake, herbage was sampled monthly from two smaller areas. The sulphur content of herbage varied from 1800–6420 ppm, which is well above the quoted values in which deficiencies may occur. The sulphur content of plants was not related to the amount of sulphur in the soil, the largest sulphur contents in the soils tending to be associated with small amounts of sulphur in the plants.

Herbage samples harvested at monthly intervals throughout the summer contained slightly smaller amounts of S in May and June, but the S contents of grass and clover did not vary markedly. (Carolyn Williams)

The accumulation of Pb in soils and herbage at Rothamsted. The contamination of the environment by lead from petrol has caused much concern, especially in the USA where very large amounts of Pb occur in soils and herbage near major roads. We determined the Pb contents of Rothamsted topsoils taken from the same sites at various times since 1881 to see if the amount of Pb in Rothamsted soil has increased as traffic becomes heavier, and with the building of the M1 motorway. Pb was also determined in soils sampled in 1955 and 1971 from three ADAS Experimental Husbandry Farms, Bridgets in Hampshire, Rosemaund in Herefordshire, and Gleadthorpe in Nottinghamshire. The results showed that the Pb content of Rothamsted soils has increased by 17–46% from the background level of the 1880–1920 period (this is equivalent to an increase of between 3 and 20 kg Pb ha, calculated from the bulk densities of the 0–22·9 cm depth sample). Between 1955 and 1971 the Pb contents of the soils sampled from the Experimental Husbandry Farms did not increase detectably.

The Pb contents of herbage sampled from Park Grass in the years 1920–23, 1940–43, 1956–59 and 1972 were determined; Ti was determined as a measure of contamination by soil dust. Increases in the Pb contents of herbage samples taken during the course of particular years were associated with increased Ti contents, which indicates that the increased Pb contents were essentially caused by dust contamination. The increase is very noticeable between the first and second harvests, but it cannot be related to motor traffic because it was equally apparent in the 1920–23 and 1972 samples. (Carolyn Williams)

Soil chemistry

Radiocarbon dating of soil organic matter. Table 3 gives the radiocarbon content of soil from four plots on Rothamsted Farm, sampled in September 1972, and two tropical

TABLE 3

Radiocarbon age of soil organic matter

Site	Sampling depth, cm	Sampling date	Organic C, %	Total N, %	$\delta^{14}C$*	$\delta^{13}C$†	Equivalent age, years
Broadbalk plot 3	0–23	1972	0·90	0·098	−85	−25·6	715±85
Broadbalk plot 8	0–23	1972	1·08	0·112	−91	−25·7	756±85
Park grass plot 3	0–23	1972	3·09	0·247	−13	−28·2	—
Park grass plot 7	0–23	1972	3·29	0·290	−56	−25·0	460±85
Zipa (Ghana)	0–23	1955	0·72	0·055	−41	−18·3	335±85
Asikuma (Ghana)	0–15	1955	2·60	0·254	−69	−28·9	575±85

$$* \ \delta^{14}C = \frac{\text{Activity of sample} - 0{\cdot}95 \text{ activity of standard}}{0{\cdot}95 \text{ activity of standard}} \times 1000$$

$$\dagger \ \delta^{13}C = \frac{{}^{13}C/{}^{12}C \text{ of sample} - {}^{13}C/{}^{12}C \text{ of standard}}{{}^{13}C/{}^{12}C \text{ of standard}} \times 1000$$

soils, both supplied by Dr. D. K. Acquaye of the University of Ghana. The Broadbalk soils give earlier dates than the corresponding samples taken three years earlier from the same plots (*Rothamsted Report for 1969*, Part 1, 84); the 1969 samples from plots 3 and 8 on Broadbalk had equivalent ages of 520 and 550 years, respectively, showing that the concentration of bomb-derived radiocarbon in the soil organic matter has now passed its maximum and is declining, just as it is in the atmosphere, although in the atmosphere the maximum was reached much earlier, in 1963. The Ghanaian soils were both collected before the heyday of nuclear testing, and show the great age of soil organic matter, even under conditions favourable to its decomposition. The Asikuma soil is a forest oxisol developed under high rainfall and the Zipa soil is from Savanna grassland. Of all the soils so far examined, the Zipa has the largest $\delta^{13}C$ ratio; presumably this is because savannah grasses use the C-4 photosynthetic cycle rather than the more usual C-3 (Calvin) cycle. (Jenkinson)

Effects of cultivation on tropical soils. Two Nigerian soils, supplied by Dr. B. T. Kang of the International Institute for Tropical Agriculture, were used in an exploratory investigation. One was from an area under secondary forest and the other from an adjacent area that had been cleared of forest and put under cultivation two years earlier. As expected, the cultivated soil contained less organic carbon than the forest soil (1·23 and 1·46% respectively). Respiration was also slightly less in the cultivated soil, the oxygen consumption (10 days at 25°C) being 22 and 32 mg/100 g soil respectively. The amounts of nitrogen mineralised in 10 days by the two soils were about the same, probably because of more vigorous immobilisation in the forest soil. However, the flush of decomposition caused by chloroform fumigation revealed much greater differences between the soils—there were 37 mg biomass carbon/100 g of cultivated soil and 72 mg/100 g of forest soil. This was confirmed by direct microscopic measurements of soil biovolume (*Rothamsted Report for 1972*, Part 1, 75); the cultivated soil contained 2·4 mm^3/g soil and the forest soil 3·9 mm^3. This suggests that changes in the soil biomass are likely to be a much more sensitive indication of a decline in fertility than are slower-moving changes in, say, total soil organic matter. (Jenkinson and Powlson)

The measurement of soil biomass by the fumigation technique. Some controversy has developed over our earlier suggestions (Jenkinson, 1966, *Journal of Soil Science* **17**, 280–302) that the amount of biomass in a soil can be calculated from the size of the flush of decomposition that follows fumigation, by using the relationship

$$\text{Biomass carbon} = k^{-1}(\text{CO}_2\text{–C evolved by fumigated soil} - \text{CO}_2\text{–C evolved by untreated soil})$$

If part or all of the flush is caused by the effect of chloroform vapour on some non-living part of the soil organic matter and not, as we propose, by the decomposition of organisms killed during fumigation, then the fumigation of a soil already sterilised by irradiation should increase the size of the flush. This was tested on a soil from Broadbalk that had been sterilised by gamma radiation (2·5*M* rad). Fumigation did increase the flush slightly. The oxygen uptake of the irradiated soil was 87 mg/100 g soil in 10 days at 25°C and that of the fumigated irradiated soil 101 mg. The corresponding figures for the production of CO_2–carbon were 18 and 24 mg, and for nitrogen mineralisation, 5·8 and 5·9 mg. However, even these increases cannot be attributed to an effect of chloroform on the non-living parts of the soil organic matter. At least some, and possibly all, of this increase was shown to arise because organisms killed by irradiation are less rapidly decomposed than those killed by chloroform.

The fumigation of an acid soil gives a very much smaller flush than the fumigation of an otherwise comparable neutral soil, implying, if our theory is correct, that acid soils contain relatively small biomasses. This was confirmed in an experiment using glucose uniformly labelled with radiocarbon. When the glucose was incubated in a near-neutral soil from Broadbalk, 28·8% of the glucose–carbon remained in the soil after 52 days. Of this, 58% was in the biomass (calculated using a value of 0·3 for k). The corresponding retention of labelled glucose–carbon in an acid soil from Geescroft (pH 3·9) was 31·6%, of which 25% was in the biomass. This suggests that although the overall loss of glucose–carbon was similar in both soils, the amount of biomass produced per unit of substrate is much smaller under acid conditions. (Jenkinson and Powlson)

A scaled-up version of the fumigation method for measuring soil biomass has been developed for routine use. This was tested on a set of soils from Rothamsted farm. Soil from the unmanured plot (03) of Broadbalk (under continuous wheat) contained 900 kg biomass carbon/ha in the top 23 cm. The corresponding figures for a plot receiving inorganic fertilisers (08) was 1000 kg, and 1900 kg for the farmyard manure plot. Broadbalk Wilderness, a small area of mixed deciduous woodland, contained 3200 kg, and unmanured permanent grassland (Park Grass, plot 3) 3400 kg. In all instances between 3 and 5% of the organic carbon in the soil was in the biomass. (Jenkinson, Powlson and Tie)

The effects of aerobic and anaerobic incubation on the extractabilities of heavy metals from digested sewage sludge. The maximum permissible dressing of sewage sludge that can be safely applied to agricultural land is currently assessed on the basis of the amounts of Zn and Ni extracted by 0·5*M* acetic acid, and of Cu extracted by 0·05*M* EDTA (Permissible levels of toxic metals in sewage used on agricultural land. *ADAS Advisory Paper*, No. 10, 1971). It is implicit in this procedure that the extractabilities of the metals do not change significantly once the sludge is applied to the land, and to test this assumption we have studied the effects of incubating sludge aerobically and anaerobically on the amounts of Cu, Ni, Zn, Cd, Pb and Cr extracted by water as well as by the ADAS reagents.

Aerobic incubation. Aerobic incubation increased the solubility of the metals in all three reagents. After four months' incubation EDTA removed three times as much Cu, twice as much Ni after one month, and eight times as much Pb after six months. The increased water-solubilities were much less dramatic, but nevertheless they were highly significant.

Adding plant matter to the sludge made little difference to the effect of aerobic incubation. Although the increases in extractability were smaller when the sludge was incubated with topsoil, the effect of aerobic incubation was still very great.

Anaerobic incubation. After three months' anaerobic incubation, the amounts of Cu extracted by all three reagents decreased almost to zero. The amount of Ni extracted by water decreased to about one-third the zero time value, but with respect to acetic acid and EDTA the solubility of Ni increased sharply during the first month, and then decreased almost as rapidly during the following two months. The amount of water-soluble Zn did not alter significantly, but considerably more Cd, Pb and Cr were extracted. Anaerobic incubation caused large decreases in the amounts of Cd extracted by acetic acid and EDTA, but Cr and Pb become considerably more soluble.

These results suggest that the total toxic metal content of contaminated sludge would be a safer guide in this context. (Bloomfield and Pruden)

https://doi.org/10.23637/ERADOC-1-130

Staff and visiting workers

The department suffered a tragic loss with the deaths of Dr. R. Greene-Kelly and Mr. R. C. Gallavan. Dr. Greene-Kelly joined us in 1949; his meticulous experimental work and his extensive theoretical knowledge made him an outstanding figure in the field of soil physics. Mr. Gallavan worked with Dr. Greene-Kelly since he came to Rothamsted in 1955, and his craftsmanship as an instrument maker contributed largely to their success in the difficult task of defining soil structure parameters and devising methods of measuring these in a more fundamental way than was hitherto possible. Their deaths within a few days of each other after nearly 20 years of friendly cooperation was doubly poignant.

We regret that Dr. H. G. C. King had to resign for health reasons, after ten years in the department.

Visiting workers included Mr. Tie Yue Liong, from Reading University, Mr. I. M. El Mansey, of The Academy of Science and Technology, Cairo, who worked on the mineralogy of Nile sediments under a UNESCO Fellowship, Mr. L. C. Surcan dos Santos, Universidade Federal Rio de Janeiro, who came to learn X-ray diffraction methods for studying clay minerals, and Mr. E. Badokwaya-Faith, from Uganda, who worked for three months on soil erosion.

https://doi.org/10.23637/ERADOC-1-130