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Mineralogical and petrographic studies of Jurassic and Cretaceous sediments from southern England and their relevance to radioactive waste disposal

A. E. MILODOWSKI
British Geological Survey, Nicker Hill, Keyworth, Nottinghamshire NG12 5GG

AND

R. D. WILMOT
British Geological Survey, Gray's Inn Road, London WC1X 8NG

ABSTRACT. A sequence of sedimentary rocks from the Lower Chalk to the Oxford Clay has been studied as part of a reasearch programme into the feasibility of the disposal of low- and intermediate-level radioactive wastes in argillaceous strata. The preservation of aragonite in such rocks suggests that little groundwater movement has occurred through the bulk of these clays and this would imply that most groundwater movement is likely to occur along any fractures that may be present. Such fractures may interconnect with adjacent aquifer units along which most transport of radionuclides would occur. In these sediments the presence of smectitic clay minerals would strongly influence their retention of radionuclides by sorption and ion-exchange reactions. Compaction, calcite cementation, calcite dissolution and the diagenesis of biogenic silica have influenced the porosity and permeability of the rocks. These processes have also controlled the chemistry of many pore surfaces in contact with groundwater and these in turn will affect groundwater chemistry, and the nature of interactions with transported radionuclides.

KEYWORDS: radioactive waste, sedimentary rocks, southern England, clays.

A SEQUENCE of Cretaceous to Carboniferous strata beneath the Atomic Energy Research Establishment at Harwell in Oxfordshire was penetrated by a number of closely spaced cored boreholes (Robins *et al.*, 1981). These strata are summarized in fig. 1. The boreholes provided access to Cretaceous and Jurassic argillaceous rocks currently under investigation as part of a generic study to determine their potential as host-rocks for the disposal of low- and intermediate-level radioactive wastes.

Detailed petrographical and mineralogical analysis of core-samples were performed to characterize the various lithologies in terms of their mineralogy, pore-surfaces, and pore-shape. These features are important to an understanding of the movement of groundwater and transport of solutes through these rocks. The sequence from the Oxford Clay to the Lower Chalk has so far been examined. Within this sequence diagenetic and post-depositional processes have affected the mineralogy and hydrogeological characteristics of these strata.

Experimental

Petrographic studies were carried out principally by Scanning Electron Microscopy (SEM) using a Cambridge Instruments Stereoscan 250, interfaced with a Link System 860 energy-dispersive X-ray analysis system (EDS). Stubs were prepared by mounting small rock-chippings on aluminium holders using a conductive carbon cement (Leit-C) and then coating with a thin film of carbon or gold. Polished-sections were also examined using back-scattered electron imaging, as well as conventional secondary-electron imaging, since the former technique can often provide additional textural and mineralogical information (Krinsley *et al.*, 1983).

Supporting optical thin-section and hand-specimen examinations were also undertaken. Chemical and mineralogical analysis were performed on similar core-samples and are reported in detail by Wilmot and Morgan (1982).

Petrography

Only a brief summary of the petrography of the various units studied is given here; detailed accounts are available elsewhere (Milodowski *et al.*, 1982; Milodowski and Wilmot, 1983). Relevant

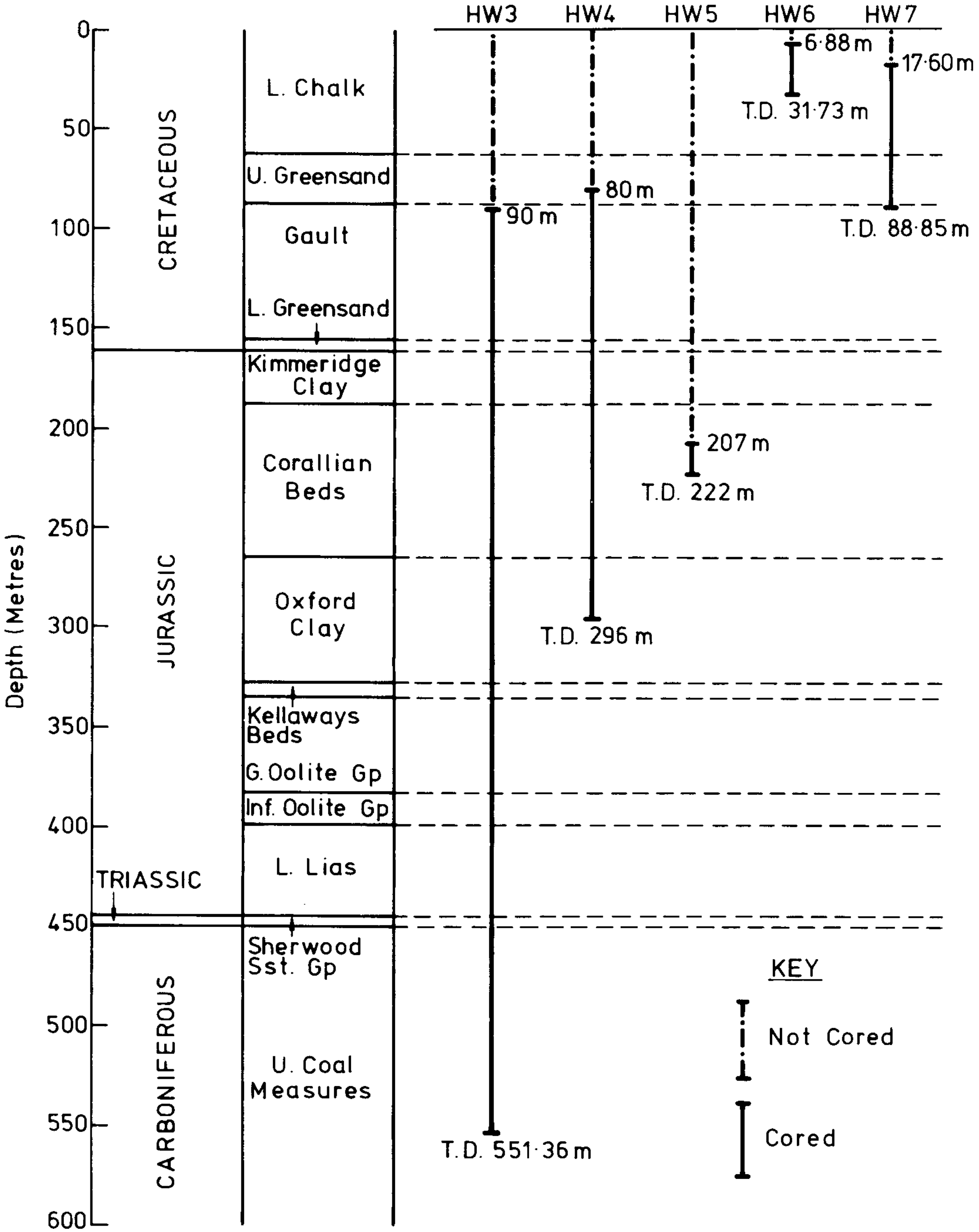


FIG. 1. Strata penetrated in the Harwell boreholes.

TABLE I. Mineralogical and petrographical features of the Mesozoic rocks

UNIT	LITHOLOGY	SAND/SILT FRACTION	DETRITAL CLAY	AUTHIGENIC	MAJOR MINERALOGY
LOWER CHALK	Microporous coccolithic limestone and marl. Sandy glauconitic marl at base.	quartz, glauconite, calcite (shell debris) Mica, sponge remains (siliceous), K-feldspar Minor zircon, tourmaline, rutile.	illite-smectite, mica, calcite (coccolithic debris)	opal-CT, calcite smectite, zeolite phosphate, pyrite.	calcite, illite-smectite, mica, opal-CT (in some chalks)
U. GREENSAND	Siliceous muddy fine sandstones and siltstones.	quartz, calcite (shell debris), mica, glauconite sponge remains (siliceous) Minor K-feldspar, illmenite.	illite-smectite mica, calcite (coccolithic). Minor kaolinite.	Opal-CT, calcite smectite, zeolite Minor phosphate, pyrite.	Quartz, opal-CT, illite smectite, calcite.
GAULT					
(a) Above 120m	siliceous muddy fine siltstone and silty mudstone.	quartz, mica, calcite (shell debris, glauconite) plant-debris, Minor feldspars, rutile, biotite.	illite-smectite mica, calcite (coccolithic) minor chlorite, kaolinite	opal-CT, smectite zeolite, pyrite minor calcite, phosphate, siderite.	illite-smectite, mica quartz, opal-CT, calcite.
(b) Below 120m	silty mudstone. Often fissile.	quartz, mica. Minor feldspars calcite (shell debris), glauconite, plant-debris, biotite.	mica, kaolinite, (illite-smectite).	pyrite, minor phosphate, siderite.	mica, kaolinite, quartz, subsidiary illite-smectite.
L. GREENSAND	Friable glauconitic quartz sands partly cemented by calcite in places. Shelly horizons present.	quartz, glauconite, calcite (shell-debris) mica, K-feldspar, plagioclase, plant-debris.	minor illite-smectite mica, kaolinite.	calcite, illite, iron oxides, minor K-feldspar, apatite, siderite, pyrite.	quartz, glauconite, calcite.
KIMMERIDGE CLAY	Fissile, silty, mudstones often calcareous with shelly horizons. Often bituminous and pyritic.	quartz, calcite (shell-debris) mica, aragonite (shell-debris) biotite, K-feldspar, plant-debris.	mica, kaolinite, calcite (coccolithic or algal), iron oxides, organic (kerogen ?).	pyrite, illite, ankerite-dolomite, calcite.	mica, kaolinite, quartz, calcite, (pyrite occasionally) organic matter.
CORALLIAN	Highly variable cyclic sequences of mudstones, siliceous siltstones, quartz sands, calcareous sandstones, bioclastic wackestones, micrites, oolitic grainstones and packstones. Generally indicating progressive shallowing. Similar to underlying Oxford Clay below 225m.	quartz, calcite (oolithic, shelly) mica, sponge-remains (siliceous). Minor K-feldspar, plagioclase, plant-debris.	illite smectite, mica, kaolinite calcite (coccolithic micritic).	calcite, opal-CT smectite, pyrite zeolite, dolomite-ankerite, illite, K-feldspar	quartz, clays, calcite, opal-CT (pyrite and dolomite occasionally). Smectite dominant clay above 240m.
OXFORD CLAY	Fissile, silty mudstones, occasionally calcareous, especially above 300 m. Bituminous and pyritic horizons occur.	quartz, mica, calcite (shell debris). Minor K-feldspar, rutile, iron oxides.	mica, kaolinite, minor illite-smectite, calcite (algal or coccolithic).	calcite, illite, pyrite, ankerite-dolomite.	mica, kaolinite, quartz, calcite (occasionally pyrite).

diagenetic or sedimentological features are discussed in more detail later. The petrography of the sequence is summarized in Table I.

The Lower Chalk is a clay-rich, microporous, coccolithic limestone or marl, often with abundant siliceous sponge remains. It is far less pure than the familiar white Middle–Upper Chalk, with up to 50% non-carbonate material (Jeans, 1968; Morgan-Jones, 1977). The Lower Chalk becomes marlier and siltier with depth, passing through a green, glauconitic, current-bedded, marly siltstone into the underlying Upper Greensand. Terrigenous components are chiefly quartz, mica, and mixed-layer illite–smectite with trace amounts of K-feldspar, zircon, tourmaline, and rutile. Molluscan and echinoid shell fragments are common.

Porosity is dominantly interparticulate within the fine lime–mud matrix. Pore-sizes are of the order of less than 1 μm. Joints or fractures were encountered within the Lower Chalk.

Authigenic precipitation of opal-CT lepispheres within the matrix pores and diagenetic voids has occurred. Opal-CT is usually associated with authigenic blocky zeolites (clinoptilolite–heulandite type) and smectite bushes and films. The coccolithic sediment may also be partially cemented by calcite overgrowths. Authigenic framboidal pyrite and ferroan mixed-layer clay (glauconite *sensu lato*) have been deposited within foram tests.

The Upper Greensand and Upper Gault are siliceous, muddy, fine sandstones, siltstones, and silty mudstones, becoming progressively finer with depth. Fine current-bedding is seen but is usually obliterated by extensive bioturbation. The rock is composed chiefly of detrital quartz, mica, mixed-layer illite-smectite with coccolithic calcite, and abundant siliceous sponge debris. Minor glauconite *s.l.* grains, detrital K-feldspar, and ilmenite and bioclastic debris are common.

Extensive diagenetic alteration of biogenic silica has taken place with the development of large (up to 300 μm) voids lined with authigenic opal-CT, zeolite, and smectite. These voids are often flattened through compaction and are less abundant in the more clay-rich Gault. Minor overgrowth of calcite on fossil fragments has occurred. Framboidal pyrite occurs in the matrix and as foram infills. Pyrite euhedra also occur along partings in the Upper Gault. Glauconite *s.l.* has precipitated in voids in foram tests and sponge-spicule canals.

Porosity is affected by the presence of diagenetic voids in the Upper Greensand. Interparticulate porosity is present in the clay matrix and pores are less than 1 μm. In the Gault, compaction has produced a planar fabric of clay particles with laminar pores between. Joints or fractures are present in the sequence.

The Lower Greensand is a greenish, coarse, friable sand, dominantly composed of corroded, rounded quartz and lesser amounts of glauconite *s.l.* Minor detrital K-feldspar, plagioclase, mica, and clay are present together with shelly bands. Remnant corroded granular calcite cement is seen. The presence of corroded quartz suggests that calcite cement was once more extensive. Authigenic K-feldspar and filamentous illite now line the dominantly intergranular porosity.

The Oxford Clay, Kimmeridge Clay, Lower Gault are all silty, fissile mudstones; the lower two commonly have bituminous horizons with amorphous kerogeneous matter. They are composed of detrital quartz, mica, and kaolinite with lesser amounts of illite–smectite, chlorite, biotite, and feldspars. Plant remains are common. Shell debris is abundant in the Kimmeridge and Oxford Clay and often forms shelly bands responsible for the high calcite contents of the clays. Calcite microspar cement associated with concentrations of primary carbonate (e.g. coccolithic debris) is present. In the Lower Gault and lower part of the Oxford Clay only minor calcite is present as scattered shell fragments. In the Kimmeridge Clay shell fragments commonly show their original nacreous fabric, similar to that of molluscan shells described by Bathurst (1975). These are responsible for the presence of aragonite and give evidence of the impervious nature of this lithology since aragonite would have been expected to have inverted to more stable calcite in sediments of this age.

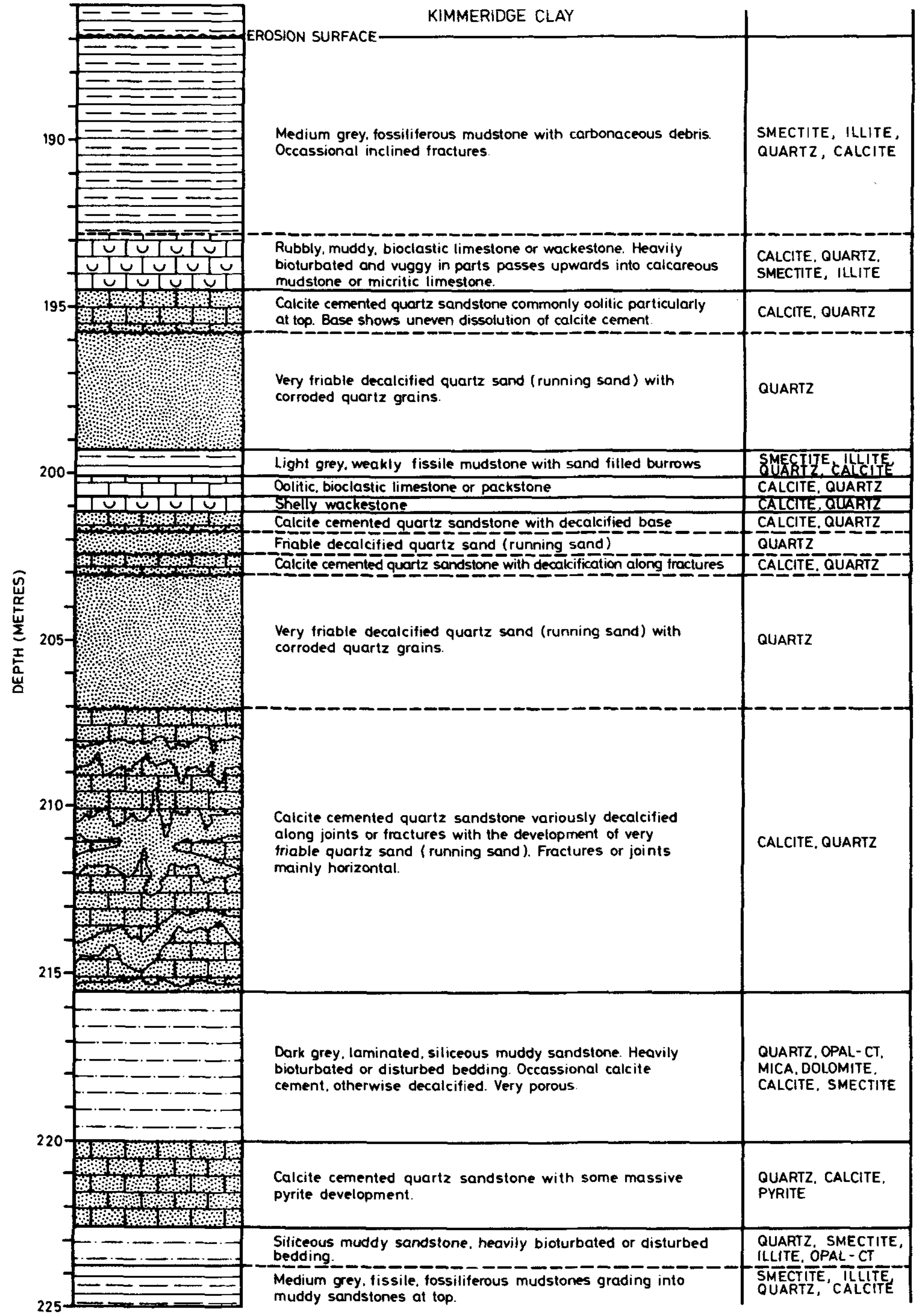
Authigenic pyrite occurs throughout as framboidal aggregates and fossil infills, and as euhedral crystals which commonly appear to have recrystallized from associated framboidal pyrite. Pyrite is often concentrated along laminae rich in organic matter and shows no sign of alteration in fresh rock. Within the Jurassic strata well-formed illite clay plates have grown parallel to the fabric, presumably by alteration of pre-existing clays. Dolomite and ankerite rhombs are common minor components in these clays.

Compaction produced a fabric of closely packed clay-mineral plates with laminar pores between them. These pores are considerably less than 1 μm in size. Fractures or joints are the only other major source of porosity in these sediments; these are inclined and commonly show slickensided surfaces.

Corallian Beds. The lower 40 m of this unit are similar to the underlying Oxford Clay; however, the upper part is a complex sequence of clays, siltstones, siliceous sandstones, calcareous sands and sandstones, bioclastic, oolitic, and micritic limestones which form the Corallian aquifer (fig. 2). Smectite dominates the clay mineral assemblage of the Corallian aquifer and the argillaceous sediments above 240 m. Below this depth the argillaceous rocks are composed chiefly of illite, mica, and quartz. Authigenic calcite cement is common in much of the sandstone but has been partially dissolved to produce running sand along joints and fractures. At the base of the aquifer, between 215 and 225 m, fine laminated silts and muds rich in sponge debris (siliceous) have been diagenetically altered to produce opal-CT, zeolites (clinoptilolite–heulandite) and smectite, leaving large diagenetic voids. Pyrite also was extensively formed in muddier horizons. Dolomite rhombs grew within the siliceous sediment matrix.

The lithologies seen in the Corallian Beds indicate a progressive shallowing during deposition, with the formation of open shelf, deltaic, lagoonal, and beach environments. Each cycle is separated by an eroded surface representing periodic marine transgression (Wilson, 1968*a*, *b*; Brookfield, 1973; Talbot, 1973, 1974; Wright, 1981).

Porosity within the Corallian Beds is variable. Argillaceous beds have pore-structures similar to the major clay units described already. In the siliceous siltstones at the base of the aquifer porosity is dominated by large spherical or sub-spherical interconnected diagenetic voids up to 200 μm diameter lined by opaline silica, zeolites, and smectite. In the 'running sands' porosity is intergranular with pores up to 300 μm diameter and pore-throats as wide as 100 μm. In the cemented beds both intergranular and fracture porosity exists with



Medium grey, fossiliferous mudstone with carbonaceous debris. Occassional inclined fractures.	SMECTITE, ILLITE, QUARTZ, CALCITE
Rubbly, muddy, bioclastic limestone or wackestone. Heavily bioturbated and vuggy in parts passes upwards into calcareous mudstone or micritic limestone.	CALCITE, QUARTZ, SMECTITE, ILLITE
Calcite cemented quartz sandstone commonly oolitic particularly at top. Base shows uneven dissolution of calcite cement.	CALCITE, QUARTZ
Very friable decalcified quartz sand (running sand) with corroded quartz grains.	QUARTZ
Light grey, weakly fissile mudstone with sand filled burrows	SMECTITE, ILLITE, QUARTZ, CALCITE
Oolitic, bioclastic limestone or packstone	CALCITE, QUARTZ
Shelly wackestone	CALCITE, QUARTZ
Calcite cemented quartz sandstone with decalcified base	CALCITE, QUARTZ
Friable decalcified quartz sand (running sand)	QUARTZ
Calcite cemented quartz sandstone with decalcification along fractures	CALCITE, QUARTZ
Very friable decalcified quartz sand (running sand) with corroded quartz grains.	QUARTZ
Calcite cemented quartz sandstone variously decalcified along joints or fractures with the development of very friable quartz sand (running sand). Fractures or joints mainly horizontal.	CALCITE, QUARTZ
Dark grey, laminated, siliceous muddy sandstone. Heavily bioturbated or disturbed bedding. Occassional calcite cement, otherwise decalcified. Very porous.	QUARTZ, OPAL-CT, MICA, DOLOMITE, CALCITE, SMECTITE
Calcite cemented quartz sandstone with some massive pyrite development.	QUARTZ, CALCITE, PYRITE
Siliceous muddy sandstone, heavily bioturbated or disturbed bedding.	QUARTZ, SMECTITE, ILLITE, OPAL-CT
Medium grey, fissile, fossiliferous mudstones grading into muddy sandstones at top.	SMECTITE, ILLITE, QUARTZ, CALCITE

FIG. 2. Summary of the lithological types within the Corallian aquifer.

fracture porosity becoming more important as the degree of cementation becomes greater.

Implications for radioactive waste-disposal studies

In the sequence examined, porosity and permeability have been affected by diagenetic events and later fracturing and dissolution. Clay mineral growth and pyrite formation may also have implications for nuclide adsorption and mineral stability. These points are discussed in detail below.

Silica diagenesis has affected Corallian, Gault, Upper Greensand, and Lower Chalk sediments originally rich in sponge debris (Wilson, 1966, 1968*a*, *b*; Talbot, 1973; Jeans, 1978). The dissolution of biogenic silica and its reprecipitation as opal-CT together with zeolite and smectite is similar to the sequence described by Jeans (1978), resulting in the formation of large diagenetic voids lined by opal-CT, zeolite, and smectite (fig. 3*a*, *b*). These pores represent easier migration routes for groundwater than the small interstitial laminar pores in the clay matrix giving rise to higher permeabilities within these rocks than in other clay-rich sediments. In the Corallian aquifer interlamination of these siliceous-rich sediments with clay-rich sediments on a fine scale has produced a rock which possesses high horizontal permeabilities (up to 1.7×10^{-2} m/day measured in the laboratory) but with much lower vertical permeabilities (1.1×10^{-4} m/day).

Zeolite and smectite are capable of ion-exchange and sorption reactions and their presence in pores may have a significant effect on the retention of solutes from groundwater. Opal-CT is relatively unstable and is known to be reactive towards

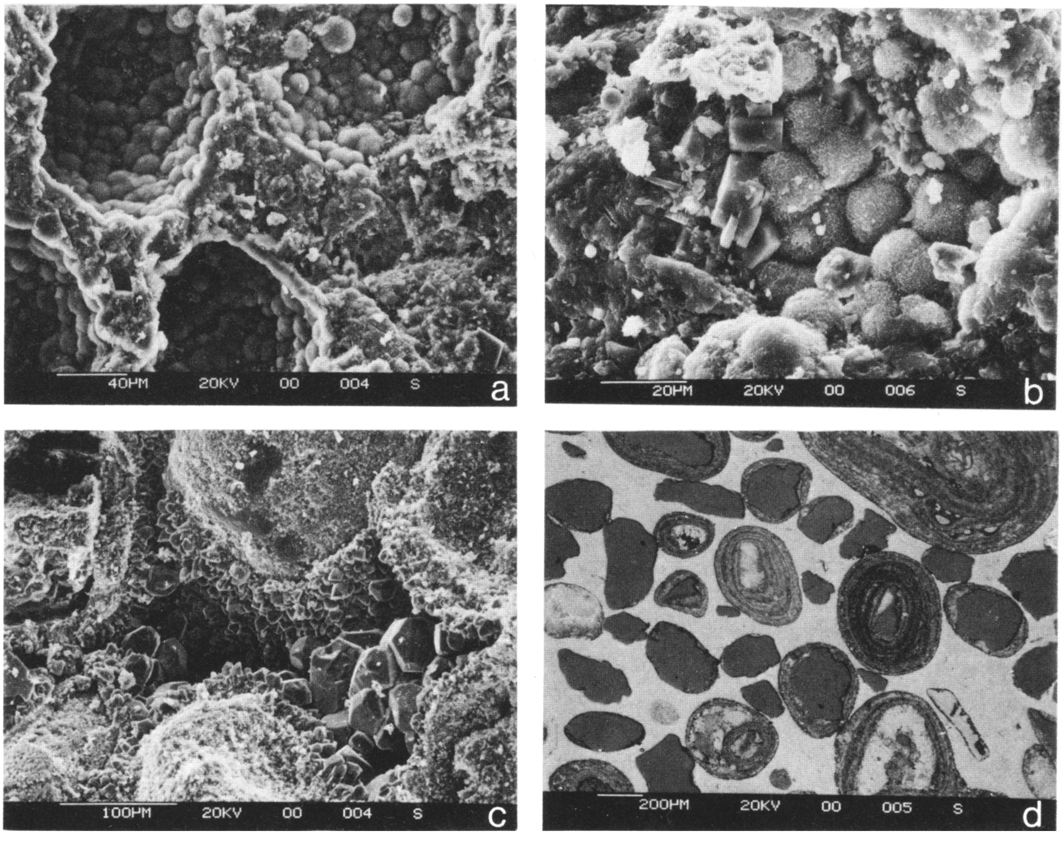


FIG. 3. (*a*) SEM micrograph of large diagenetic voids lined by opal-CT within siliceous muddy sandstone and siltstone. (*b*) SEM micrograph showing detail of diagenetic void-surfaces showing opal-CT lepispheres, blocky zeolite crystals and clay minerals. (*c*) SEM micrograph of calcite-cemented oolitic sandstone showing early 'fibrous' rim cement and later equant pore-filling calcite spar. (*d*) Back scattered electron micrograph of calcite cemented oolitic limestone showing complete pore-filling by calcite cement. All from the Corallian Beds.

cement (Baker and Poole, 1980; French, 1980; Gillot, 1980). Its presence in these rocks may lead to poor sealing properties against cementitious backfill materials in any waste repository.

Calcite cementation. The principal effect of calcite cementation has been to reduce pore-sizes and permeability and this is best developed within the aquifer units. Calcite may partially fill pores with sparry crystalline linings (fig. 3*c*) or may completely fill them (fig. 3*d*) such that the only porosity remaining exists as intergranular cracks, micropores, or large-scale fractures and joints. In such well-cemented rocks the major path for water movement must be along joints or fractures.

Calcite dissolution. Following cementation by calcite, fracturing has allowed corrosive groundwaters to penetrate into the rocks dissolving away calcite cement and grains. The result has been to form beds of highly porous and permeable 'running' sand (fig. 4*a*) along which most movement of groundwater occurs (e.g. Corallian Beds and Lower Greensand). Corroded detrital quartz grains testify to the former existence of extensive calcite cement. Dissolution has followed fractures and bedding and may have taken place as a result of the downwards penetration of meteoric water charged with carbon dioxide.

Clay Minerals. The argillaceous rocks of the Upper Gault and basal Corallian Beds, and the more permeable beds of the Upper Greensand and the Corallian aquifer contain a significant amount of smectitic clay. This mineral has a high capacity for ion-exchange and sorption reactions and will strongly influence the sorption of radionuclides from groundwater passing through these rocks. McKinley and West (1982) have demonstrated that

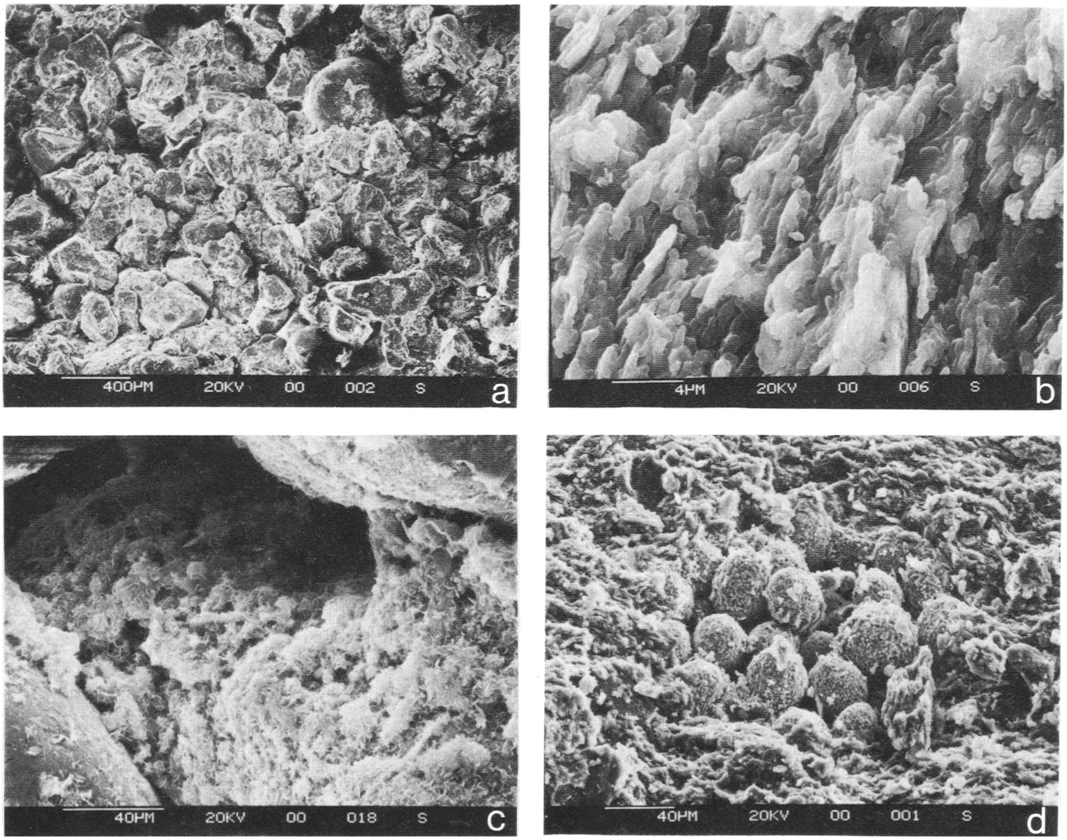


FIG. 4. SEM micrographs. (*a*) Decalcified 'running-sand' showing friable, porous, loose sand. Corallian Beds. (*b*) Neoforming illite platelets growing on detrital illite and kaolinite matrix. Kimmeridge Clay. (*c*) Authigenic flakey illite and iron oxides lining and filling pores in sand. Lower Greensand. (*d*) Framboidal pyrite clusters in clay matrix. Kimmeridge Clay.

the smectitic Gault and Corallian rocks show a greater retention for Co, Cs, and Sr than do the other argillaceous rocks within this sequence. Well formed platy illite clay is found growing on the kaolinite and illite matrix of the Kimmeridge and Oxford Clay, producing illite-rich mudrocks (fig. 4*b*). Presumably illite has replaced earlier detrital clays. This authigenic growth further reduces the porosity and permeability of these rocks.

In the Lower Greensand and Corallian 'running-sands' authigenic fibrous illite and 'amorphous' iron oxides line the intergranular pores (fig. 4*c*). These phases not only reduce pore-volume but may choke pore-throats, thus reducing the permeability. These authigenic coatings are capable of ion-exchange and sorption reactions with groundwaters in the pores. It has been shown that the Lower Greensand is capable of considerable retention of solutes derived from landfill-leachates, particularly of heavy metals due to sorption and ion-exchange mechanisms with clay and iron oxide films (Ross, 1980; Campbell *et al.*, 1983).

Authigenic pyrite. Disseminated authigenic pyrite is associated with organic matter diagenesis (Hudson, 1978). It is present in significant amounts in the Kimmeridge Clay and Oxford Clay and common in many other parts of the sequence (fig. 4*d*). This mineral is susceptible to oxidation and microbial attack by organisms such as *Thiobacillus ferrooxidans* (West *et al.*, 1982). Pyrite oxidation results in the formation of sulphates and sulphuric acid and these may cause corrosion of metals and cementitious materials in underground structures. It should be noted that the pyrite in these clays is finely disseminated throughout and in the fresh core material was unoxidized.

Aragonite. The presence of aragonite in the Kimmeridge Clay and of well preserved fossil shells points to the impervious nature of these clays. Aragonitic shells would be expected to have dissolved or recrystallized during diagenesis in more permeable rocks (Bathurst, 1975).

Conclusions

Characterization of the geochemistry and movement of groundwater through rocks is fundamental to a study of the geological containment of radioactive waste since this is the medium by which released radionuclides will be transported. In this context a knowledge of the factors affecting porosity, permeability, and geochemistry are important. Sorption of solutes from groundwaters will take place along surfaces in contact with groundwater, namely pore and fracture linings. Petrographic studies on Mesozoic rocks from the Harwell Research Site have shown that authigenic minerals such as smectite, illite, and zeolite often line some pores, and these may influence the sorption of radionuclides in these rocks.

Any migration of radionuclides in groundwater out of the argillaceous units is most likely to occur along fractures. The presence of unstable aragonite within these clays is attributed to their impervious character. The most probable route for radionuclide migration will be along the aquifer horizons and in particular the Corallian Beds and Lower Greensand (above the Oxford Clay). Within these beds most groundwater movement will occur through the highly porous and permeable 'running-sands' or along fractures in well-cemented beds. Some movement may be expected along the siliceous horizons within the Corallian Beds and Upper Greensand. In these lithologies groundwaters will encounter authigenic smectite, zeolite and opal-CT in the Upper Greensand and siliceous Corallian beds, and authigenic illite and iron oxides lining the pore-walls within the running-sands of the Lower Greensand and Corallian Beds. These minerals are capable of ion-exchange and sorption reactions and their presence may help in the retardation of radionuclide migration in groundwaters.

Finally the bulk mineralogies of some of the argillaceous rocks (i.e. Corallian Beds, Upper Gault, Upper Greensand) are smectite-rich. The presence of this mineral will therefore be of importance in considering the feasibility of radioactive waste-disposal in these formations.

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