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PETROLOGY OF THE DESMOINESIAN EXCELLO BLACK SHALE OF THE MIDCONTINENT REGION OF THE UNITED STATES

OMER ISIK ECE[1]

Department of Geosciences, University of Tulsa
Tulsa, Oklahoma 74104

Abstract—The Excello Shale is one of the best exposed and most laterally continuous of the Pennsylvanian cyclothemic black shales in the midcontinent region of the United States. Its petrology and paleoenvironmental significance were studied to understand the nature of cyclic black shales in general and how they relate to the habitat of hydrocarbon source beds. The Excello is thinly laminated, fissile where weathered, and rich in organic matter and phosphate nodules; it is 90–120 cm thick. Its thin laminations, fine particle size, and high total organic carbon (TOC) content suggest that it was deposited in a quiet water environment of an epeiric sea having anaerobic bottom water. The Excello Shale consists of two lithofacies: non-bioturbated black shale and bioturbated yellow-brown shale. Petrographic studies show that the black shale contains wavy to straight laminations and that the yellow-brown shale contains discontinuous and random laminae and mottled stratification. The close association of organic matter and phosphate-controlled nodule morphology (spherical, elongated, bladed, and platy) appear to be related to progressive decreases in nutrient supply of the sea water from the ancient ocean (Panthalassa).

Clay-mineral assemblages consist mainly of detrital illite, kaolinite, chlorite, and illite/smectite (I/S). Grain size and amount of these clays increase and TOC decreases shoreward towards the probable source areas of the clay minerals in the northern Ouachita region and northern Iowa. Quartz, carbonate-fluorapatite, carbonate fossil fragments, and minor feldspar and pyrite are the principal minerals in the Excello Shale. Minor amounts of fine-silt-size carbonate minerals are present in some samples. Limestone concretions, 90–150 cm in diameter and 30 cm thick, were found at only one locality in Missouri where the Excello is a maximum of about 3 m thick. Fissility increases with weathering along the bedding plane, and laminae are separated by a limonite film. Four petrographically distinct microfabric variations of the Excello Shale appear to be related to the TOC content and bioturbation. Textural and structural properties of the shale are more developed with increasing organic matter content.

Key Words—Black shale, Illite/smectite, Kaolinite, Nodules, Organic carbon, Petrology, Phosphate.

INTRODUCTION

The origin of shales rich in organic matter is difficult to ascertain from thin-section studies because the opacity of the organic matter masks many important paragenetic relationships. Understanding the origin of such black, carbonaceous shale, however, is economically important because these rocks are the primary source of oil and gas. Pennsylvanian cyclothemic black shales, of which about 60 are known from the midcontinent, are rich in phosphate nodules (Heckel, 1977, 1986; Kidder, 1985). The Excello Shale Member of the Senora Formation exposed around the Ozark Uplift provides an excellent opportunity to study one of the most representative of these shales (Figure 1). A generalized stratigraphic section of the study interval is shown in Figure 2. The petrology and depositional environments of the Excello were originally reported by Cassidy (1968). The goals of the present study were (1) to describe the regional facies, microfabric, and petrography of the Excello Shale, (2) to determine the origin of its phosphate nodules, and (3) to determine the distribution and origin of the clay minerals present in the shale.

METHODS OF INVESTIGATION

More than 100 samples were collected from outcrops of the Excello Shale in the area shown in Figure 1. Fresh samples were also taken from recently excavated strip coal mines. Thin sections were cut perpendicular to bedding. Clay minerals were separated from carbonate rocks by treating the rocks with 0.5 M acetic acid, according to the method of Ostrom (1961).

Microporous porcelain filter candles were maintained under vacuum in order to wash flocculating agents from the slurry. Clay-size fractions (>2 μm) were prepared by Stokes' law settling in deionized water. Four oriented clay mounts were prepared for X-ray powder diffraction (XRD) study by smearing the clay paste onto petrographic slides (Gibbs, 1965). Each sample was then: (a) air dried, (b) treated with ethylene glycol, (c) heated to 450°C for 1 hr, and (d) heated to 575°C for 0.5 hr. Semiquantitative XRD analyses were made following the methods of Schultz (1964). Total

[1] Present address: Mineralogy and Petrography Department, Faculty of Mines, Istanbul Technical University, Tesvikiye, Istanbul, Turkey.

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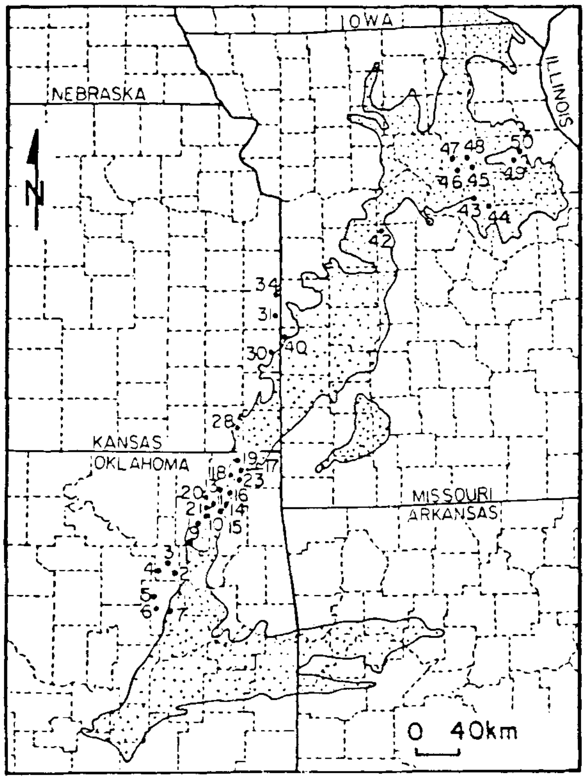

Figure 1. Map showing Cabaniss Group outcrop belt (stippled), locations of measured sections, and locations of cores taken in the interior lowland region of the United States. Modified from Branson (1954).

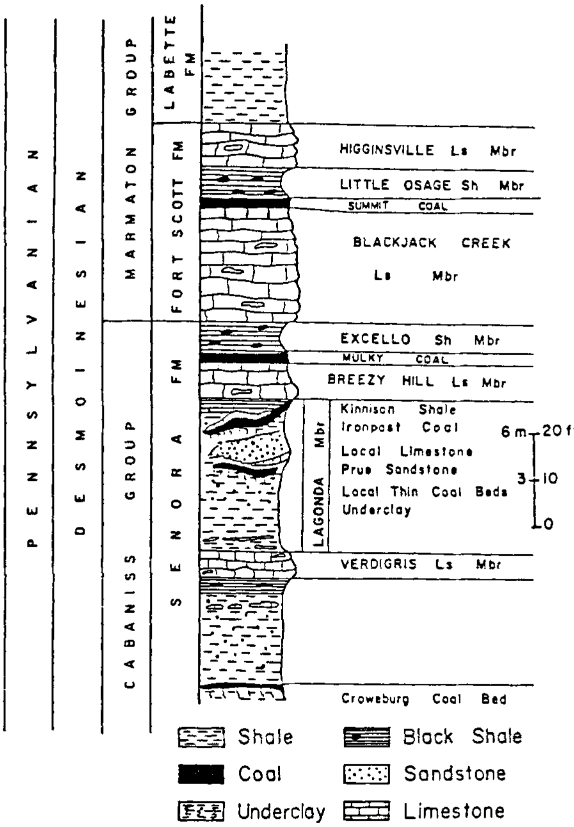

Figure 2. Generalized stratigraphic column of the Excello sequence in northeastern Oklahoma and southeastern Kansas. Modified from Branson (1954), Bennison *et al.* (1972), and Branson *et al.* (1965). (Fm = formation, Ls = limestone, Mbr = member, Sh = shale.)

organic carbon (TOC) analyses, as weight percentages of the whole sample, were made using a LECO combustion furnace.

RESULTS

Textures and sedimentary structure

The Excello Shale is thinly laminated and rich in phosphate nodules (Cassidy, 1968). Two lithofacies were recognized during the present study: (1) black shale and (2) yellow-brown shale which underlies and overlies the black shale. No bioturbation was noted in the black shale, aside from one outcrop in northern Missouri where dysaerobic conditions have been postulated due to local topographic highland (location 47, Figure 1). The TOC content of the black shales averaged 10% and ranged from 6 to 17%. The yellow-brown shales were found to contain less organic matter (<2% TOC) and showed bioturbation. Field studies revealed that fissility developed only in weathered outcrops where no bioturbation was noted, and that the laminae were separated by a yellow film of limonite. Bedding plane fissility increased as a function of weathering. In contrast, no fissility was observed in cores or in unweathered black shale.

Phosphate nodules are common in the black shales of the study area. Some show concentric layers; others are structureless. Most of the nodules are medium to dark reddish-brown or light gray to dark gray. The nodules vary in shape and size and can be classified into four groups (Figure 3), following the shape classification of Folk (1974, p. 9): (1) In the southern part of the study area, the nodules are hard and spherical or ellipsoidal; they exhibit concentric layering and range in size from 2 to 5 cm. They are rich in organic matter and contain as much as 7% TOC. (2) In the central part of the study area, the nodules are elongate; they range from 1 to 4 cm in length and contain 4–6% TOC. (3) In the central Missouri area, the nodules are bladed and partly silicified; they range from 3 to 6 mm in thickness and from 3 to 5 cm in length and contain about 1–2% TOC. (4) In the northern part of the study area, phosphates do not occur as nodules; instead, they occur as plate-like grains ranging from 1 to 5 mm in thickness and from 3 to 20 cm in length. The plates are low in organic matter content, containing as little as 0.6% TOC. These morphological variations are depicted in the paleoceanographic model shown in Figure 3.

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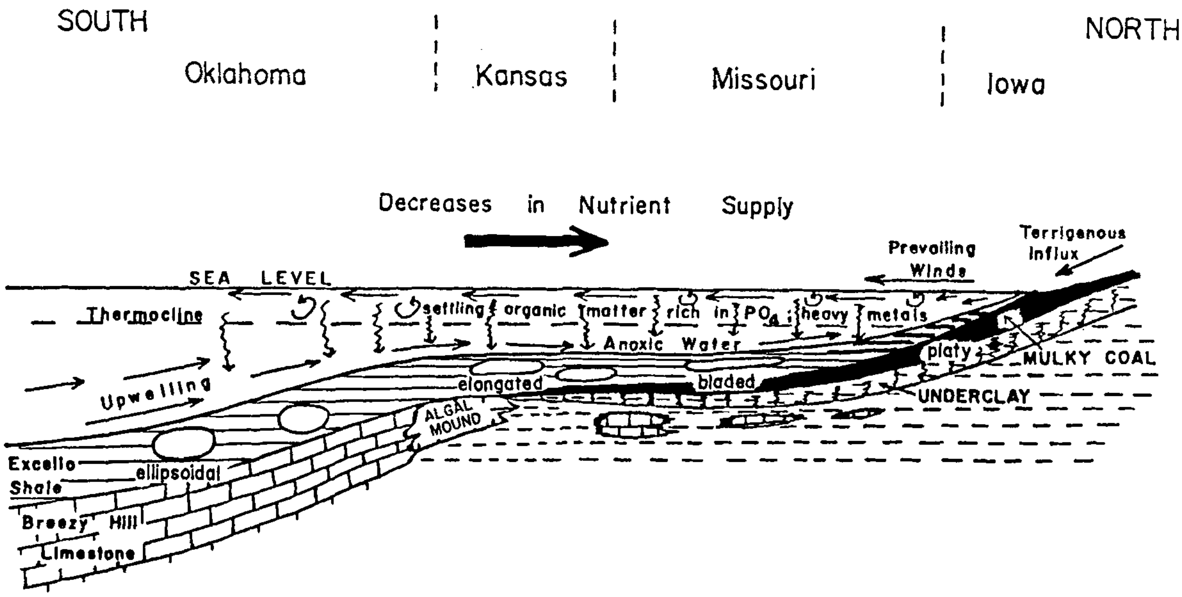

Figure 3. Illustration showing northward changes from spherical to platy in the morphology of the phosphate nodules in Excello black shale along the outcrop trend. The terms spherical or ellipsoidal, elongated, bladed, and platy were adopted from Folk's (1974, p. 9) shape classification for particle morphology. This paleoceanographic and diagenetic model is modified from Heckel (1977), Brongersma-Sanders (1971), and Kidder (1985).

In Vernon County, Missouri (location 40, Figure 1), the Excello black shale contains limestone concretions as large as 90–150 cm in diameter and as much as 30 cm thick. These limestone concretions were not noted elsewhere in the Excello Shale. In addition to calcite, they contain silt-size quartz, chert, pyrite, and organic matter.

Shale petrography

Sand-size particles are rare in the Excello Shale. Dark, blackish-reddish-brown organic-rich shale is composed chiefly of illitic clay and accessory clay- and silt-size quartz, minor amounts of silt-size carbonate-fluorapatite, and muscovite flakes and trace amounts of albite, silt-size carbonate minerals, and authigenic pyrite. According to Lewan's (1978) classification of very fine grained sedimentary rocks, the Excello Shale is an argillaceous claystone.

In thin section the shale consists of discontinuous, wavy and straight parallel laminae, as shown in Figure 4. Parallel, discontinuous and elongate micro-lenses of very fine grained quartz are diagnostic and suggest late-stage silicification. In contrast, mottled and discontinuous, random particle orientations characterize the yellow-brown shale.

In the field, gray clay laminae as much as 1 cm thick and 20 cm long were found in the black shale in the samples from northern Missouri (location 47, Figure 1); however, under the microscope, some gray clay laminae were noted as thin as 0.1 mm and as much as 0.5 cm in length. Locally, thin laminae of black shale are interlaminated with gray clay. By XRD, no mineralogical difference was noted between gray clay laminae and the black shale. Contacts between the gray clay and black shale were relatively gradational; thus, the occurrence of gray clay could have been due to dysaerobic conditions (1–2% TOC) or fluctuations in the rate of production of organic material.

Four petrographically distinct textural and compositional varieties of the Excello Shale were noted in thin section: (1) wavy to straight parallel and thinly laminated black shale containing abundant organic matter (Figure 4); (2) dark-brown shale rich in organic

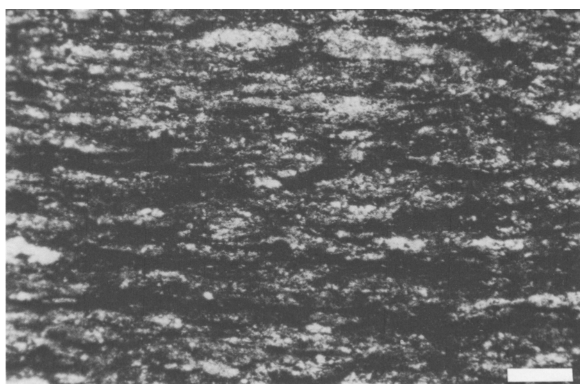

Figure 4. Photomicrograph of thin section showing thinly laminated Excello black shale from outcrop 18D, Craig County, Oklahoma. Note planar lamination and elongation of material parallel to bedding. Light-colored areas are mostly gray clay particles. Dark areas contain more organic matter. Sample contains about 12% total organic carbon. Plane polarized light. Bar scale = 250 μm.

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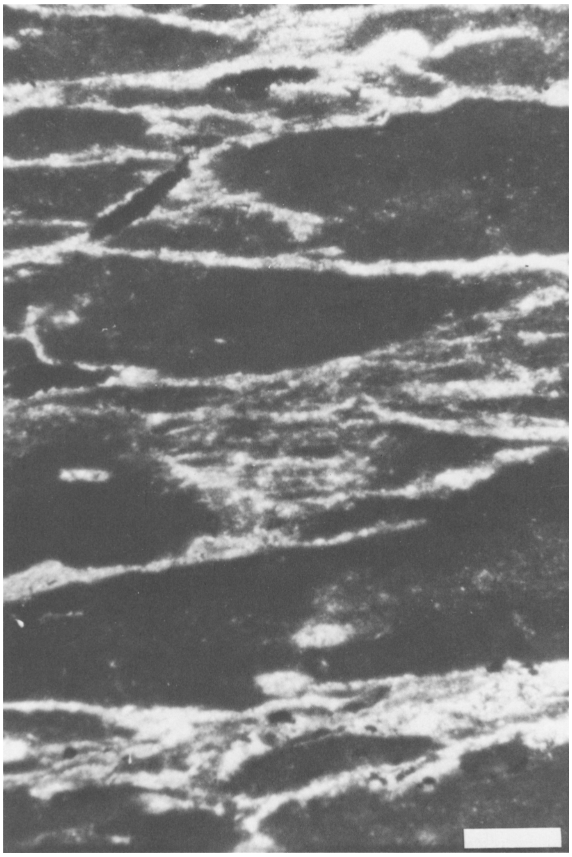

Figure 5. Photomicrograph of thin section showing thinly interlaminated yellow-brown shale and dark-brown shale. Sample was collected from the upper yellow-brown shale (2% total organic carbon) of the Excello at location 36G, Rogers County, Oklahoma. Ragged, lenticular nature of zonations may be due to bioturbations, as clearly shown in Figure 7. Plane polarized light. Bar scale = 250 μm.

matter and interlaminated with light-brown shale poor in organic matter (Figure 5); (3) shale poor in organic matter and exhibiting little structure (Figure 6); and (4) bioturbated shale (Figure 7). These textural variations are related to the TOC content and bioturbation rather than to grain size and mineralogical composition. Greater TOC contents appear to correlate with the greater development of laminations.

Some of the black shales examined contained an anisotropic matrix and displayed various patterns of parallel orientation (Figure 4). They also contained small (<0.01 mm), birefringent aggregates of clay and displayed subparallel extinction and a randomly flecked appearance. The birefringent aggregates of clay are commonly oriented as streaks parallel to the bedding (Figure 5). Fractures are subparallel to each other or are part of an irregular pattern. Some of the features may have been caused by burrowing organisms. Lenticular and irregularly broken shale laminae were found only in the yellow-brown shale (Figure 5). Optically

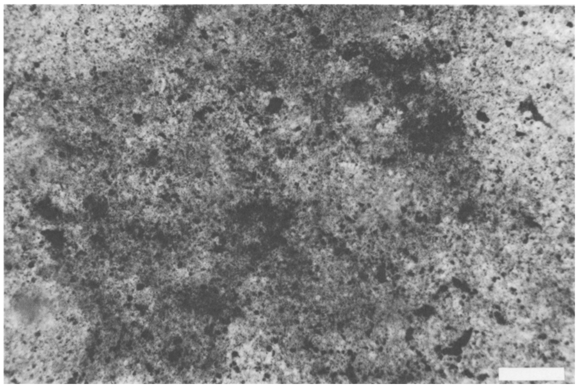

Figure 6. Photomicrograph of Excello black shale from location 47, Randolph County, Missouri, showing disseminated organic matter in homogeneous clay matrix. Darker areas may be organic structures. This photomicrograph shows an example of poorly developed bedding. Light areas indicate areas relatively poor in organic matter (about 1% total organic carbon) and rich in clay materials. Plane polarized light. Bar scale = 250 μm.

orientated aggregates of clay were noted along subparallel fractures that showed joint-plane cutans (i.e., surface coatings) or joint-plane, organic-poor argillans (Figure 5). Such cutans are characteristics of surfaces that are related to texture, internal structure, and fabric (Brewer, 1976). The organic-poor argillans consist of clay minerals stained by dark-colored organic matter (Figure 5). These fracture zones were probably filled with clay minerals during late-stage lithification.

One sample of black shale displayed multidirectional extinction patterns within the clay matrix, due to randomly oriented and unevenly distributed mineral fragments (Figure 6). The bioturbation characterized by randomly distributed masses of organic matter in Fig-

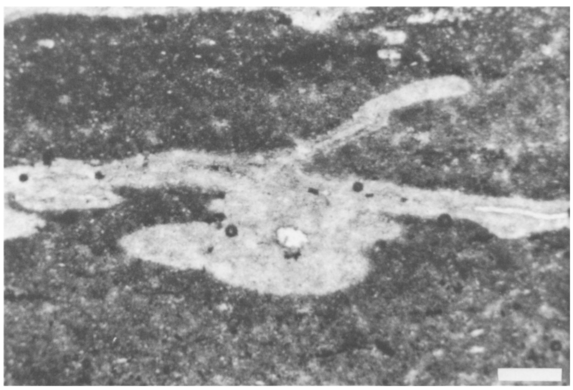

Figure 7. Photomicrograph of thin section of unweathered rock showing tracks and trails of a burrowing organism. Sample was collected from the upper yellow-brown shale (location 36G, Figure 1), Roger County, Oklahoma. The total organic carbon content is 0.4%. Plane polarized light. Bar scale = 250 μm.

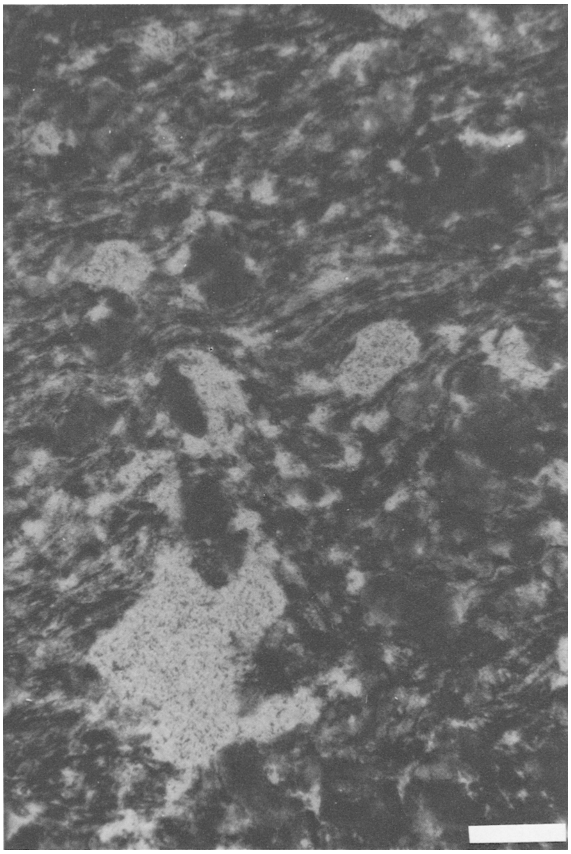

Figure 8. Photomicrograph of thin section showing colorless and light- to dark-brown, diagenetic collophane aggregates in black shale. The photomicrograph shows high organic matter content (about 2% total organic carbon) surrounding the collophane aggregates. Location 47, Randolph County, Missouri. Plane polarized light. Bar scale = 250 μm.

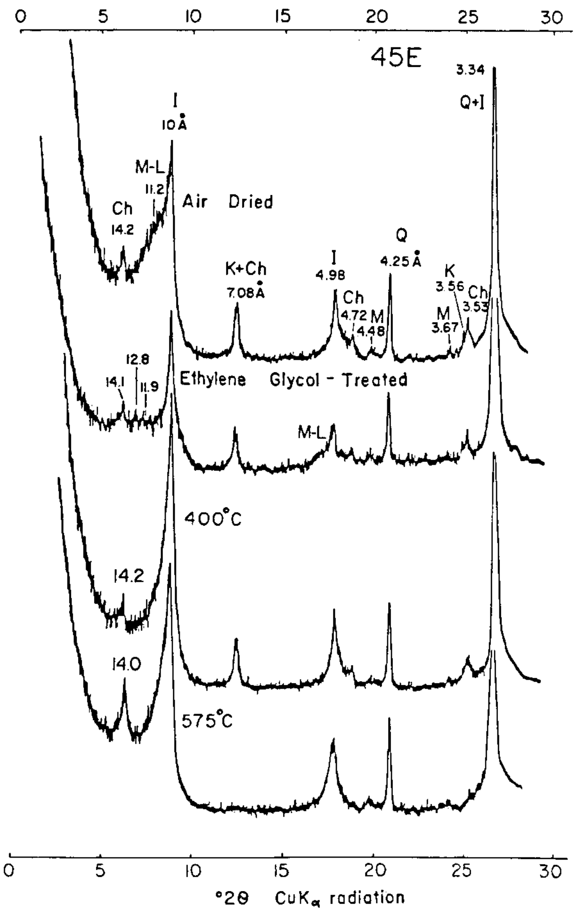

Figure 9. X-ray powder diffraction (XRD) patterns from the black shale samples, Nemo Coal Company, Randolph County, Missouri (stratigraphic section 45 in Figure 1). Abundant illite/smectite (M-L) is indicated by the expansion of the broad XRD reflection near 11.2 Å to about 12.8 Å, with glycolation. After heating at 400°C, the smectite component collapsed to 10 Å or less. Illite (I) is the major mineral and small amounts of IS-ordered illite/smectite (M-L), kaolinite (K), chlorite (Ch), and quartz (Q) are present. M indicates muscovite-1*M* reflection at 4.488 Å (19.78°2θ). The presence of muscovite-1*M* is due to imperfect size separation. Muscovite is probably detrital in origin.

ure 6 is typical of yellow-brown shale that has a weakly striated matrix.

Tracks and trails of burrowing organisms are common in the yellow-brown shale, indicating a habitable bottom. Together with low TOC content, the track and trails also indicate a dysaerobic environment (Figure 7). Burrowed units exhibit no fissility. The black shales shown in Figure 7 are characterized by a flecked orientation pattern and a random distribution of spores. The matrix contains rounded aggregates or pellets of apatite (0.05 to 0.2 cm) that have a patchy distribution in those samples relatively poor in organic matter.

As shown in Figures 4, 6, and 8, spores have been flattened by compaction. The absence of original spore shapes suggests that filling materials (sediment or cement) were not present inside the spores during early diagenesis.

Pyrite occurs as framboidal masses and cubes ranging in size from 0.01 to 0.06 mm. Pyrite aggregates typically show a cubic internal habit. Organic matter commonly coexists with pyrite either as a complete or partial rim around its perimeter. No evaporite or heavy minerals, except pyrite, were found in thin section.

Phosphates

Carbonate-fluorapatite is a major constituent of the black shale throughout the region studied (Figure 8). Rounded apatite aggregates range in size from 0.05 to 0.2 mm. In thin section both colorless apatite aggregates (ovoids) and light- to medium-brown aggregates were noted. The former is present in only small amounts (Figure 8). Layers of apatite aggregates are oriented parallel to bedding in all samples collected from northern Missouri.

Petrographic studies showed that clasts of apatite

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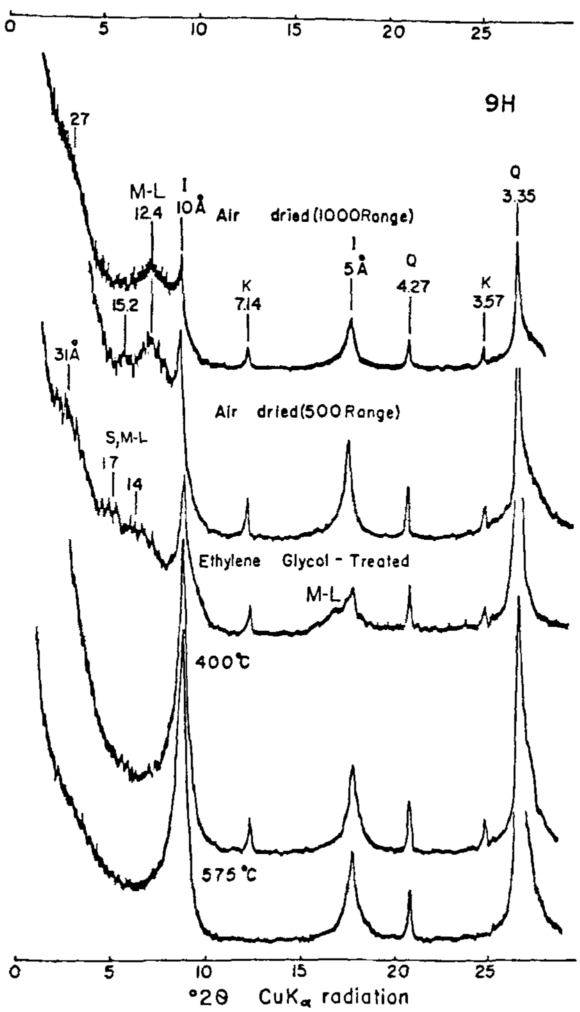

Figure 10. Typical X-ray powder diffraction (XRD) patterns of the clay-size fractions from the yellow-brown shale (road cut on Highway 20; stratigraphic section 9 in Figure 1). Broad peaks can be seen near 12.4, 15.2, and 27 Å. The 12.4-Å peak and "superlattice" maximum near 27 Å from the air-dried sample suggest the presence of illite/smectite (I/S). Partial expansion of the I/S peak at 12.4 to 14 Å on glycolation and the low-angle shift of the "superlattice" maxima to 31 Å indicate an expandable smectite component in the mixed-layer clay. The inflection point at 17 Å in the glycolated sample suggests the presence of discrete smectite or randomly interstratified I/S. Symbols and abbreviations as in Figure 9.

aggregates are present in a parallel orientation. The nodules consist of major amounts of carbonate-fluorapatite (francolite) and minor amounts of quartz, feldspar, calcite, pyrite, kaolinite, and illite. Concentric layers in some phosphate nodules suggest accretionary growth from solution. Conodonts and radiolaria are common in the phosphate nodules.

CLAY MINERALOGY

Illite is the dominant clay mineral in all of the <2-μm fractions examined. Kaolinite and chlorite are minor constituents in nearly all of the samples studied (Figures 9 and 10). XRD patterns of most samples showed relatively weak 002 and 004 reflections and relatively strong 001 and 003 reflections that are characteristic of Mg-rich chlorite (Brown, 1961).

The upper and lower yellow-brown shales near the Oklahoma-Kansas border contain slightly less kaolinite and chlorite than the black shale. Chlorite is slightly more abundant in the yellow-brown shale than the black shale in east-central Oklahoma, but the opposite relationship was noted in samples collected in northern Missouri.

The underclay below the underlying Mulky Coal (Figure 2) contains appreciably more I/S, apparently due to potassium leaching from illite in the Mulky swamp, and significantly less chlorite and kaolinite than the Excello Shale. XRD analysis and petrographic studies show that the silt-size fraction of the black shale in the central and northern parts of the outcrop belt contain very little muscovite-1*M* of possible detrital origin. Muscovite-1*M* was identified according to the data of Yoder and Eugster (1955, table 3).

The amount of each clay mineral in the shale changes from the northern shelf of the Arkoma basin (shoreward) to the Kansas-Missouri (basinward) platform (Figure 11). Increased shoreward abundances of kaolinite and chlorite, accompanied by increases in grain size and decrease in TOC content, suggest partial control of detrital clay minerals by terrestrial sedimentation.

ORIGIN OF NODULES AND CONCRETIONS

The change in shape and size of the phosphate nodules appears to be related to lateral fluctuations of organic productivity, changes in sea-water chemistry, variation in sedimentation rates, and changes in nutrient-rich terrestrial influx (Figure 3). The intimate association of organic matter and the phosphate nodules suggests that organic matter was the likely source of the phosphate in the nodules. Upwelling in the ancient Panthalassa to the southwest provided nutrient-rich water that supported an abundant phytoplanktonic population. Consequently, a decrease in TOC content and nutrient supply northward influenced nodule morphology; i.e., spherical shapes are common in the south, whereas elongate and finally platy shapes are common in the north. The calcite content also increases progressively toward the north. The fossil zooplankton and fecal pellets reported in the Excello Shale by Porter and Robbins (1981) link anoxic bottom-water and biologically associated minerals with planktonic ecosystems in the epeiric sea. The widespread distribution of phosphate nodules throughout the Excello Shale suggests sluggish lateral water circulation in the epeiric sea.

Three important sources of phosphate have been

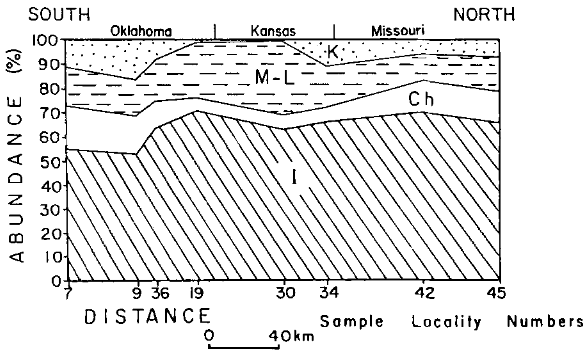

Figure 11. Generalized lateral distribution of clay minerals in the Excello black shale from the northern shelf of the Arkoma basin in northern Missouri. This illustration is an average of the semiquantitative analysis for samples collected from the top, middle, and base of the Excello black shale at each locality. Symbols and abbreviations as in Figure 9.

suggested for the anoxic environments: (1) marine phytoplankton and other organic matter in the water column (Dunbar and Berger, 1981); (2) bacterial biomass- and bacterial-algal mats (Reimers, 1982; Williams and Reimers, 1983); and (3) fish bones (Suess, 1981). The release of phosphate into interstitial water of ocean sediments is thought to occur during the sulfate reduction (Claypool and Kaplan, 1974; Berner, 1982).

A diagenetic-replacement model for the formation of phosphate nodules is proposed in Figure 12, as modified from Kidder (1985). Based on petrographic studies five stages of phosphatization can be recognized: (1) Fine-grained carbonate muds that were deposited as lenses or nodules during the anoxic episode crystallized progressively to micrite, microsparite, and, finally, sparite. (2) The large sparite crystals continued to grow; however, in addition to carbonate mud, some grew around borings, fecal pellets, conodonts, radiolaria, foraminifera, and biogenic particles from the hard parts of fossils. These fossil fragments became rimmed with apatite cement and their chemical compositions changed. (3) The limestone concretions decomposed because of Eh-pH changes in the micropore environments. Initial substrate gradually changed by calcite dissolution, phosphate diffusion, and Na^+, K^+, Cl^-, F^-, SO_4^{2-}, CO_3^{2-}, Ca^{2+}, and Mg^{2+} exchanges to maintain charge balance between the interstitial water and the nodule. (4) Some void spaces filled with pyrite. (5) Partial replacement of phosphate by siliceous materials became important during late diagenesis; however, late-stage calcite cementation took place in some nodules.

The presence of 1–4-μm-thick rims of microcrystalline apatite on radiolaria in some nodules suggests direct precipitation of apatite from interstitial water, as reported by Burnett (1977). Burnett also reported elongated apatite crystals (1–3 μm) that apparently grew in interstices of biological debris and on the surface of

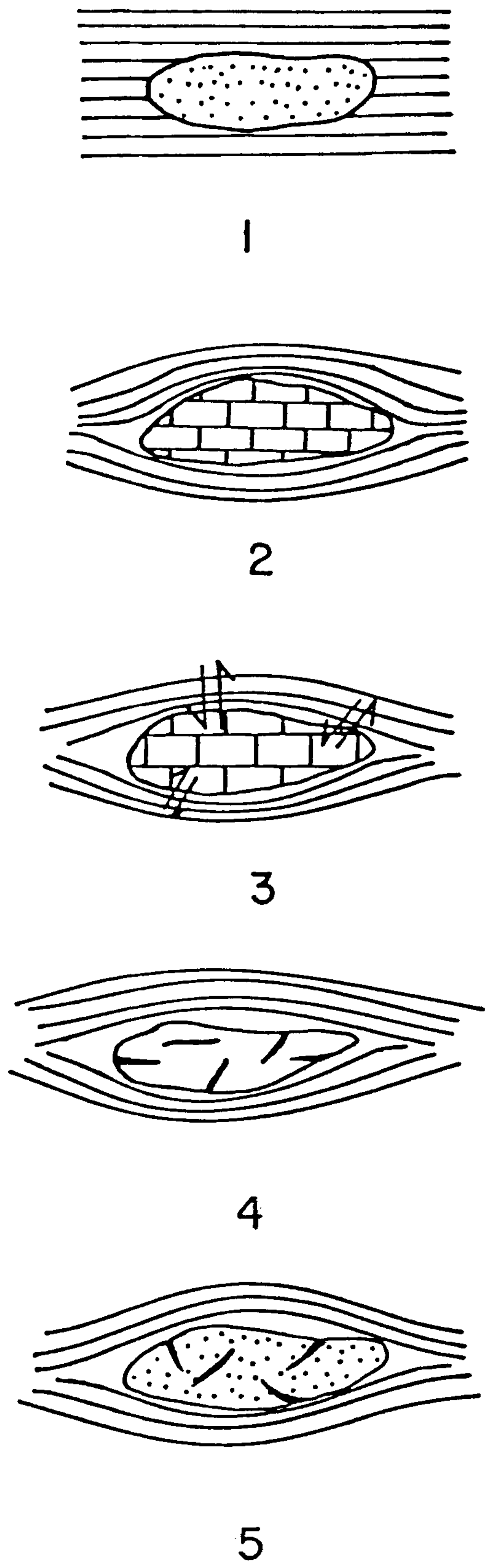

Figure 12. Schematic representation of the evolution of phosphatization. See text.

diatom frustules. Minute spherulitic crystals of apatite replacing Holocene foraminifera, however, have been found in sediments off the coast of Peru (Manheim *et al.,* 1975). Average sea water contains 0.001–0.05 mg/kg phosphate (Drever, 1984); however, pore water of the anoxic sediments from the southeastern Atlantic Shelf contains 775–2520 μg/liter phosphate (Baturin, 1972); that from Baja California contains as much as 220 μg/liter phosphate (Gieskes *et al.,* 1982); and that from the Peru continental margin contains 950–5700 μg/liter phosphate (Suess, 1981). These concentrations of phosphate, as much as several thousand times that of average sea water, probably favored direct precipitation of apatite in micropores.

Cronoble and Mankin (1965) reported that the limestone concretions in the Upper Pennsylvanian (Missourian) Stark black shale of the Hogshooter Formation of Oklahoma were epigenetic or diagenetic in origin. They suggested that the presence of thin shale laminae compacted around the limestone concretions indicated that the concretions were formed before the shale was completely compacted. Cronoble and Mankin also stated that the concretions did not form where the Stark black shale was very thin, thereby supporting the current observation that limestone concretions occur only within the Excello black shale where it attains a maximum thickness of about 3 m (location 40, Figure 1). Rims of pyrite have also been found on the limestone concretions. These pyrite rims are about 0.5 cm thick and extend 1 cm beneath the surface, which suggests a diffusion process during early diagenesis (see also, Berner, 1969). Raiswell (1971) suggested that the concretions were diagenetic and that they grew by the precipitation of calcite in the interstitial space of the shale.

Stable isotope compositions of limestone concretions from the Miocene Monterey Formation of California indicate that the concretions grew during burial increasing in abundance with temperature of formation and during the late stage of organic diagenesis (Hennessy and Knauth, 1985). Claypool and Kaplan (1974) proposed a model that getting progressively heavier $\delta^{34}S$ (+20 to +40‰) and lighter $\delta^{13}C$ (0 to −20‰) values of pore waters of deep-sea sediments are the result of progressive limestone concretion growth in the zones of microbial sulfate reduction, microbial methane production, and thermocatalytic decarboxylation with increasing depth and temperature. Similar research on the Excello limestone concretions has not been carried out.

Muscovite-1*M* may have been derived from rocks exposed to the east and north. The southern source of detrital clay minerals, especially chlorite, was apparently weathered, low-grade metamorphic rocks of the Ouachita fold belt (Flawn, 1961). The northern source, however, probably was chloritic rocks in the metamorphic terrains of the Great Lakes or northern Appalachian regions (Miyashiro, 1978, p. 221).

SUMMARY AND CONCLUSIONS

The Excello Shale is a thinly laminated, pyritic, organic matter-rich shale which was deposited in a landlocked, sluggish marine environment having anoxic bottom water. The Excello is wavy to straight-parallel laminated and contains intergrowths of pyrite and carbonate-fluorapatite. Thin-section studies revealed that laminae of clay minerals are common in many of the organic-rich shale units. The high TOC content suggests high organic productivity or good preservation and the absence of bottom-dwelling organisms in the Excello sea. Bioturbation was noted only in northern Missouri, probably the result of a local topographic high. Under such conditions, gray clay particles were deposited as parallel, discontinuous laminae within the black shale. The Excello Shale is nearly carbonate-free, but some silt-size carbonate minerals were noted in thin sections of samples collected from the thickest exposure of the formation, which also contained large limestone concretions (i.e., in west-central Missouri).

Phosphate nodules were apparently formed during early diagenesis in an environment similar to that currently existing off the coast of Peru. The morphology of the nodules was controlled by nutrient-rich waters of coastal upwelling and planktonic marine ecosystems.

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