

## MINERALOGICAL TRANSFORMATIONS DURING WEATHERING OF LIGNITE OVERBURDEN IN EAST TEXAS

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**Abstract**—Mineralogical weathering sequences in sediments overlying lignite beds were investigated in a core (27 m deep) from Calvert, Robertson County, northeast Texas. Weathering trends were evaluated based on the properties and relative distributions of both the expandable and non-expandable minerals. The sulfide minerals in these sediments are the most susceptible to weathering and were only observed in the unaltered (reduced) zone below 7 m. Oxidation of the sulfides has resulted in the formation of jarosite and gypsum in the upper 7 m of the core (oxidized zone). The oxidized zone is further characterized by reddish brown colors (high chroma), a greater quantity of dithionite-extractable iron, and absence of chlorite. Although the major clay minerals in these sediments (smectite, kaolinite, mica) are largely detrital, weathering has resulted in an increase in the content of kaolinite and a decrease in the content of mica towards the surface. The mica appears to have altered to a high-charge smectite characterized by basal spacings of 32 Å after intercalation with octadecylammonium cations. The high-charge smectite is most abundant in the soil horizons at the top of the core and gradually decreases with depth. A low-charge smectite is the most abundant species in the unweathered parent sediments and increases with depth.

**Key Words**—Lignite, Oxidation, Reclamation, Smectite, Soil, Weathering.

### INTRODUCTION

Lignite occurs extensively in the Tertiary (Eocene) Wilcox Formation which outcrops in a northeast-southwest trend across Texas (Figure 1), Arkansas, and parts of Louisiana. The sediments overlying the lignite consist of mainly muds, silts and sands. According to Kaiser (1974), sedimentation was cyclic resulting in alternating sands and mud-silt-lignite units. A typical sequence is ~15 m thick and begins with lignite, then passes into clay, mud, interlaminated mud, and fine sands, and finally into units of flat lying fine sand. As a result of strip mining operations for the lignite, vast quantities of these sediments will be placed on surface of the reclaimed areas. The reclamation strategy in Texas is to level the nonsegregated, randomly mixed sediments down to contours and drainage patterns benchmarked during premining surveys (White, 1978). There are no plans to segregate and replace soil horizons because the native soils are generally infertile, with sandy surface horizons and dense clay subsoils. It is essential that the properties of these sub-soil sediments be investigated in order to predict the long-term processes that may occur when they are exposed to soil-forming processes.

The mineralogical composition of these sediments has been influenced by several factors including variations in the original source of the argillaceous particles, the reducing environment which existed during the lignite-

generating period, the diagenetic and lithification processes which occurred after burial, and finally the present-day weathering processes. Weathering has affected these relatively unconsolidated sediments to depths of 10 to 20 m, depending on the texture of the materials (Dixon *et al.*, 1982). The large quantities of smectite in the soils developed from these sediments may be partly due to alteration of the mica and chlorite during weathering, although it is difficult to distinguish between the smectite that formed as a result of pedogenic weathering and that which may be inherited from the unweathered parent sediments.

The purpose of this study was to investigate the chemical and mineralogical properties of the sediments overlying the lignite and to predict the long-range mineralogical changes which may be expected to occur on exposure of these materials to soil-forming processes. Six cores from locations along the major lignite-bearing belt in east Texas (Figure 1) were investigated. The following discussion is based mainly on data from core Ro 1 obtained from the main lignite-bearing Calvert Bluff Formation at Calvert in Robertson County. Samples from this core show all the transitions from unoxidized to oxidized, reddish brown materials and finally to soil horizons at the top of the core.

### MATERIALS AND METHODS

#### *Physical and chemical analyses*

All cores were kept in sealed plastic bags to minimize oxidation during storage. Composite samples representative of the various portions of the core were taken

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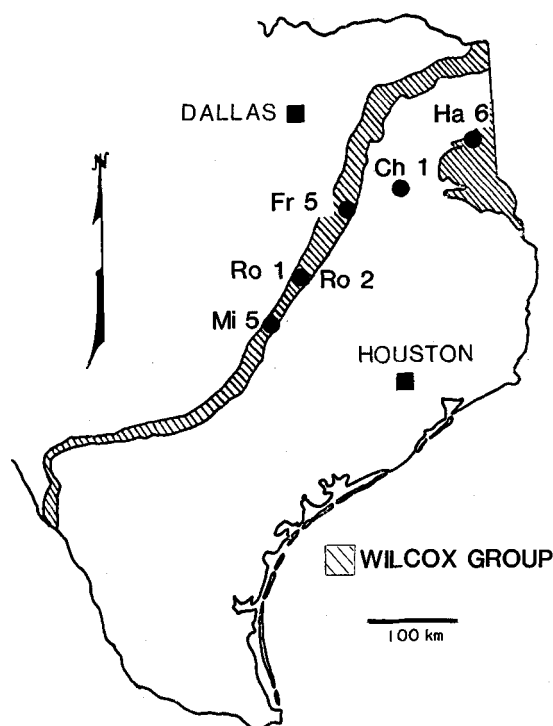


Figure 1. Map of east Texas showing locations of the cores investigated.

from freshly exposed surfaces to avoid contamination. The samples were freeze dried and gently ground to pass through a 2-mm sieve. The pH was determined in 1:1 soil to water suspensions using a digital Chemtrix type-60 pH meter. Pyritic S was determined by extracting the samples with dilute nitric acid followed by determination of the iron released as described by the ASTM method (ASTM, 1974). For particle-size separation, samples were treated with dithionite-citrate-bicarbonate (Mehra and Jackson, 1960) to remove free iron oxides. The Fe released was determined by atomic absorption spectrophotometry. Excess salts were removed by dialysis before size fractions were obtained by the method of Jackson (1969). The clay ( $<2 \mu\text{m}$ ) and silt ( $5\text{--}2 \mu\text{m}$ ) fractions were separated by centrifugation using an International #2 centrifuge. The  $<0.2\text{-}\mu\text{m}$  fractions were separated into medium ( $0.2\text{--}0.08 \mu\text{m}$ ) and fine ( $<0.08 \mu\text{m}$ ) clay fractions using a Sharples centrifuge.

#### Mineralogical characterization

**X-ray powder diffraction (XRD).** A qualitative mineralogical characterization of the coarse ( $2\text{--}0.2 \mu\text{m}$ ), medium ( $0.2\text{--}0.08 \mu\text{m}$ ), and fine ( $<0.08 \mu\text{m}$ ) clay fractions was made by XRD. Samples were saturated with K and Mg by washing with either KCl or  $\text{MgCl}_2$  solutions. Oriented samples were prepared by drying clay suspensions on glass slides. XRD diagrams of air-dry samples

(K- or Mg-glycerated samples) and heated K-saturated samples were obtained using a Philips diffractometer with a graphite monochromator and Cu radiation at 35 kV and 14 ma.

**Semiquantitative determination.** A semiquantitative estimation of the layer silicate minerals in the clay ( $<2.0 \mu\text{m}$ ) fractions was carried out as follows: The mica content was calculated from total K ( $10\% \text{K}_2\text{O} = 100\% \text{mica}$ ). For the determination of total K, samples were digested in HF overnight at room temperature using a procedure similar to that described by Bernas (1968) except that plastic volumetric flasks were employed instead of the heated-digestion pressure vessels. The calcium and potassium cation-exchange capacities (CaEC and KEC, respectively) were determined according to Jackson (1969, p. 256). The difference between CaEC and KEC was used to calculate the K-fixation capacity ( $\beta$ ) of the clay using the following expression  $\beta = [(CaEC - KEC)/CaEC] \times 100$ . The smectite and vermiculite contents were calculated using the procedure of Alexiades and Jackson (1966) assuming a cation-exchange capacity (CEC) of 154 meq/100 g for vermiculite and 105 meq/100 g for smectite. Estimation of kaolinite and chlorite were based on relative peak intensities obtained from clay films prepared as described by Karlsson *et al.* (1978). This method gives highly reproducible XRD intensities. Peak areas of smectite ( $17\text{--}18 \text{ \AA}$ ), mica ( $10 \text{ \AA}$ ), chlorite ( $7.2 \text{ \AA}$ ), and kaolinite ( $7.2 \text{ \AA}$ ) were multiplied by factors of 1, 4, 2, and 2, respectively, following the procedure of Biscaye (1965). In samples containing both chlorite and kaolinite, the kaolinite and chlorite contributions to the  $7\text{-}\text{\AA}$  peaks were obtained from slow ( $\frac{1}{4}^\circ/2\theta/\text{min}$ ) scans over the 002 and 004 peaks ( $3.56\text{--}3.54 \text{ \AA}$ ) of kaolinite and chlorite, respectively.

**Infrared spectroscopy.** Infrared (IR) absorption spectra were obtained with a Perkin-Elmer model 283 spectrophotometer using the KBr disc method (0.75 mg of sample in 300 mg of KBr).

**Intercalation with *n*-alkylammonium ions.** For characterization of the expandable 2:1 mineral species, coarse clay ( $2\text{--}0.2 \mu\text{m}$ ) samples were intercalated with octadecylammonium cations using a procedure similar to that of Lagaly and Weiss (1969). Portions of Na-saturated clay samples (50–100 mg) were placed in 40-ml plastic centrifuge tubes. The samples were then suspended in a minimum of 95% alcohol and mixed well with a vortex mixer. A solution of hot ( $75^\circ\text{C}$ ) octadecylammonium hydrochloride (0.1 N) was added ( $\sim 5 \text{ ml}$ ), and the suspensions were immediately placed in a water bath set at  $75^\circ\text{C}$  for 24 hr. The samples were then washed three times with a  $75^\circ\text{C}$  solution of 50:50 water-alcohol. A 30-min wash at  $50^\circ\text{C}$  with an ethanol-water mixture was found to be adequate by Walker (1967) to remove excess organic molecules from the sample. The sam-

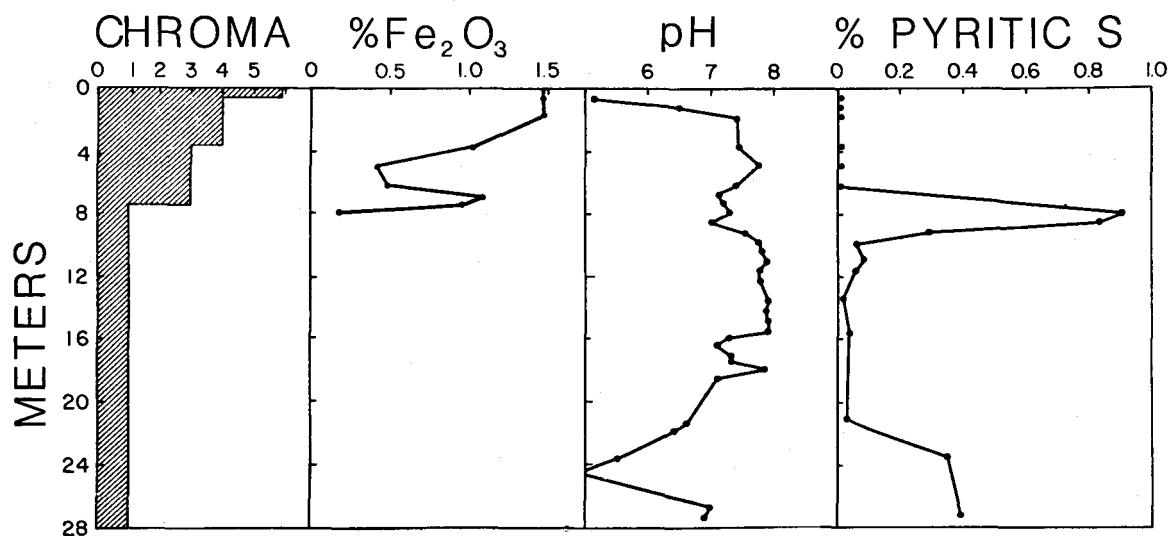


Figure 2. Comparison of the oxidized (0–7 m) and reduced zones in core Ro 1 based on color, free iron oxides, pH, and pyritic S.

ples were then deposited on glass slides and dried overnight at room temperature before being X-rayed. Two reference montmorillonite samples, Mt02 (Camargo bentonite from Oklahoma supplied by Dr. Lester Reed) and Mt05 (Ca-montmorillonite, STX-1, from Gonzales County, Texas, obtained from The Clay Minerals Repository) were also intercalated with octadecylammonium ions for comparison.

## RESULTS AND DISCUSSION

### *Physical and chemical properties*

Fine materials (mainly clay loam, Table 1) were observed in the upper 10-m depth of the core. Much of the Calvert Bluff sediments in east Texas consist of similar materials which are referred to as overbank sediments. These sediments were deposited in marshes and swamps between channels (Kaiser, 1974). The data in Table 1 indicate a high clay content in the B21t horizon of the soil developed from these sediments. Mottling is common in the partly truncated, heavy clay B21t and B22t horizons indicating low permeability. This soil belongs to the Axtell series (L. P. Wilding, Department of Soil & Crop Sciences, Texas A&M University, College Station, Texas 77843, personal communication) which is classified as fine, montmorillonitic, thermic Udertic Paleustalfs. Soils with high clay contents in the subsoil horizons are common throughout the lignite belt of east Texas (Henry and Kastning, 1978).

The materials in the upper 7 m of the core (oxidized zone) were characterized by reddish brown colors with a Munsell chroma of 3 or greater and more free iron oxides (Figure 2) than underlying materials. Oxidation of pyrite in this zone has resulted in the release of sul-

fate ions and the precipitation of gypsum and/or jarosite. Gypsum and jarosite were observed along bedding planes in the oxidized zone and are common on recently exposed mine spoils. The occurrence of jarosite and gypsum along bedding planes suggests that these minerals were not formed while the core was in storage but were formed *in situ* as a result of natural weathering processes. More intense weathering at the top of the core is indicated by a gradual decrease in pH towards the surface. Variations in pH were also observed below the oxidized zone, but are attributed to the presence of sulfuric acid produced by the oxidation of pyrite during storage of the core. There appears to be a correlation between zones of low pH at depths of 8, 16, and 24 m and higher contents of pyritic S (Figure 2).

### *Clay mineral alterations*

*Formation of kaolinite.* The greater abundance of kaolinite, particularly in the B21t horizon at the top of the core (Table 2), may be explained in several ways. It is possible that differential eluviation resulted in a concentration of the relatively larger particles of kaolinite in this horizon. However, it appears that the more intense weathering at the top of the core favored the formation of kaolinite. Increasing quantities of fine clay-size kaolinite from the C to the B21t horizon at the surface are indicated by the XRD (Figure 3) and IR (Figure 4) data. The IR absorption bands at 3695 and 3620  $\text{cm}^{-1}$  are due to hydroxyl stretching of the  $\text{Al}_2\text{OH}$  bonds in kaolinite (Farmer, 1974). However, absorption of the  $\text{Al}_2\text{OH}$  bonds in the 2:1 structures also contributes to the intensity of the 3620  $\text{cm}^{-1}$  bands. The intensity of the 3695  $\text{cm}^{-1}$  bands which are entirely due to kaolinite increases from the C to the B21t horizon. Although most

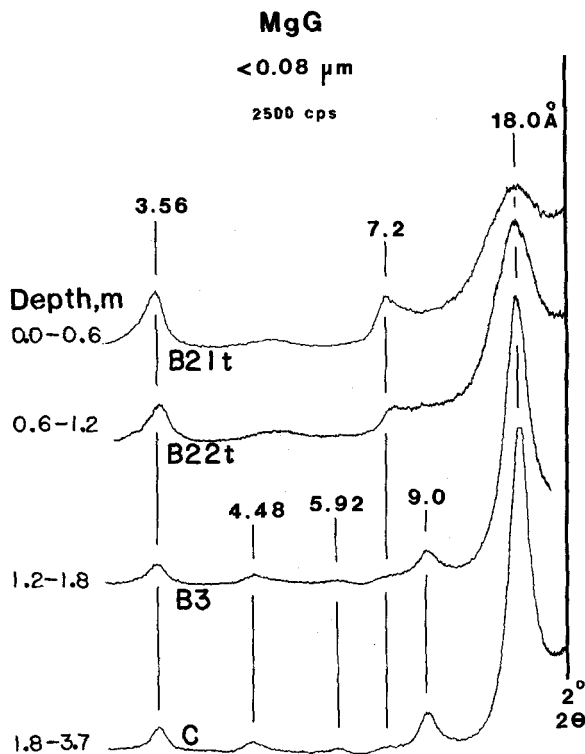


Figure 3. X-ray powder diffraction patterns of fine ( $<0.08 \mu\text{m}$ ) clay fractions from core Ro 1 showing increasing peak intensities of kaolinite (7.2 Å) from the C to the B21t horizon (CuK $\alpha$  radiation).

of the kaolinite in these materials is thought to be detrital (Fisher and McGowen, 1967), increasing amounts of kaolinite towards the surface (both in the coarse and fine size fractions) suggest that at least some of the kaolinite has formed as a result of weathering.

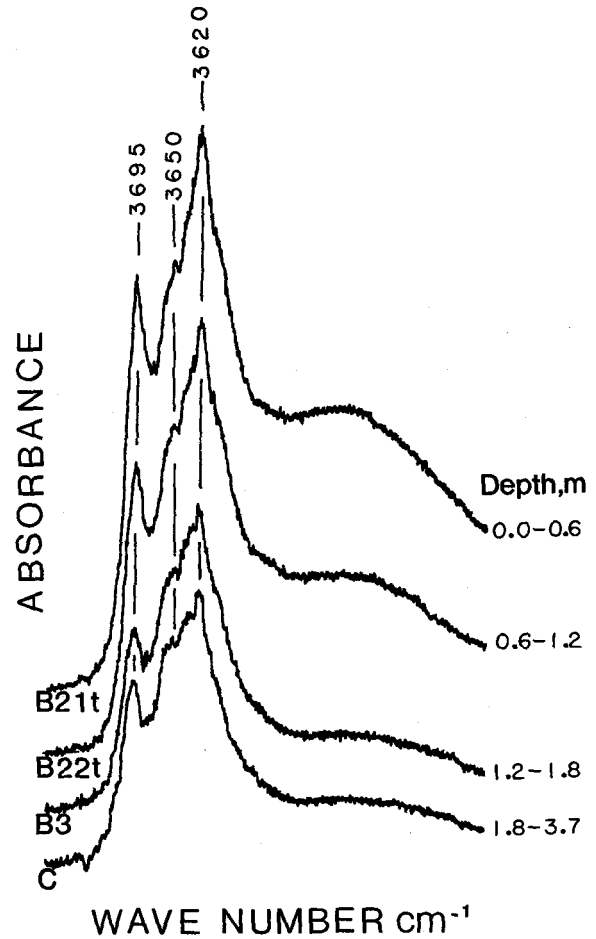


Figure 4. Infrared spectra of the ( $<0.08 \mu\text{m}$ ) fractions from core Ro 1 showing increasing contents of kaolinite from the C to the B21t horizon.

Table 1. Physical properties of representative overburden samples from Robertson County, Texas, Core Ro 1.

Sample	Horizon	Depth (m)	Particle size distribution (%)					Textural <sup>1</sup> class	Moist <sup>2</sup> color
			Sand >50 $\mu\text{m}$	Silt 50-5 $\mu\text{m}$	5-2 $\mu\text{m}$	Clay 2-0.2 $\mu\text{m}$	<0.2 $\mu\text{m}$		
1	B21t	0.0-0.61	28.3	16.0	1.0	5.3	44.7	c	5YR4/6
2	B22t	0.61-1.22	39.7	27.6	1.3	4.7	23.0	cl	10YR5/4
3	B3	1.22-1.83	30.8	27.9	3.4	10.9	23.4	cl	10YR6/4
4	C	1.83-3.66	33.0	40.8	2.9	10.4	11.1	cl	10YR5/4
11		8.54-9.15	25.1	41.7	3.9	14.4	11.7	cl	10YR4/1
12		9.15-9.76	16.1	44.0	6.0	18.0	13.1	sicl	10YR4/1
14		10.37-10.98	26.3	36.1	4.9	17.5	13.1	cl	10YR4/1
15		10.98-11.59	26.9	36.5	6.0	16.4	12.2	cl	10YR4/1
17		13.05-13.66	50.6	27.0	3.3	11.4	5.5	scl	10YR4/1
21		15.12-15.73	86.5	7.0	1.2	3.5	2.3	ls	10YR6/1
27		20.52-21.13	86.9	5.4	1.3	3.6	1.9	ls	10YR6/1
28		21.13-21.74	58.9	16.0	3.6	11.1	8.5	scl	10YR4/1
29		22.87-23.48	60.2	16.7	2.4	11.3	8.0	scl	10YR4/1
32		26.62-27.26	85.3	6.5	1.2	3.1	2.5	ls	10YR4/1

<sup>1</sup> Abbreviations: c = clay, cl = clay loam, sicl = silty clay loam, ls = loamy sand, scl = sandy clay loam.

<sup>2</sup> Symbols used are from Soil Survey Staff (1951).

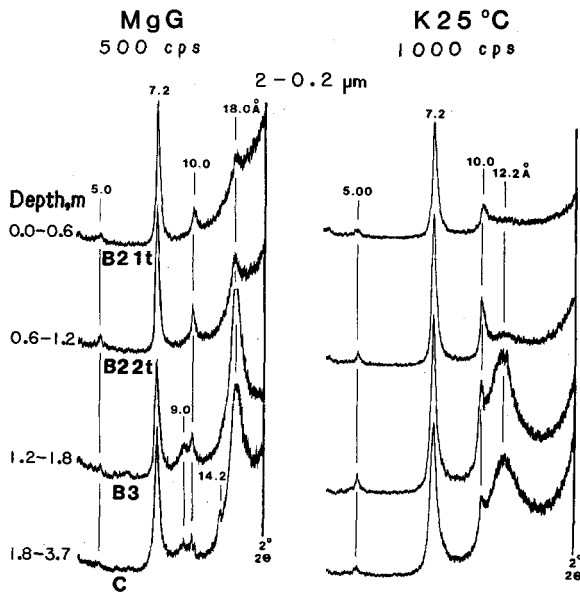


Figure 5. X-ray powder diffraction patterns of coarse (2–0.2 μm) clay fractions from core Ro 1 showing the expansion and collapse of high-charge smectite after various treatments (see text) (CuKα radiation).

**Alteration of mica to high-charge smectite.** The smectite in the coarse clay fractions (2–0.2 μm) from B21t and B22t horizons shows characteristics which are intermediate between those of vermiculite (collapsed to 10 Å on saturation with K and air drying) and smectite (Mg-saturated samples expanded to 17.7 Å on glycerol solvation). This phenomenon was also observed by Egashira *et al.* (1982) who described this type of smectite as high-charge smectite. The proportion of the ex-

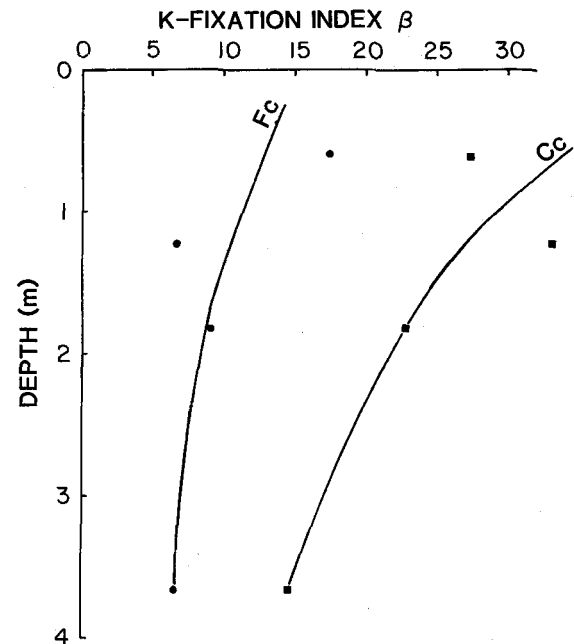


Figure 6. Changes with depth in the K-fixation capacity ( $\beta$ ) of coarse clay (2–0.2 μm) (Cc) and fine clay (<0.2 μm) (Fc) fractions from core Ro 1. Values of  $\beta$  for the Mt02 and Mt05 reference smectites are 27.4 and 15.7, respectively.

pandable 2:1 minerals which collapsed to 10 Å after saturation with K and air-drying (high-charge smectite) decreased with depth (Figure 5), indicating that the high-charge smectite is more abundant in the most highly altered samples at the top of the core. Most of the smectite in the least altered sample (horizon C) collapsed to only 12 Å after saturation with K and air-drying. The

Table 2. Semiquantitative mineralogical composition of clay samples (<2.0 μm) from core Ro 1 estimated from cation-exchange capacity, K<sub>2</sub>O, and X-ray powder diffraction data.

Sample	Horizon	CaEC (meq/100 g)	KEC (meq/100 g)	K <sub>2</sub> O (%)	Smec. (%)	Verm. (%)	Kaol. (%)	Mica (%)	Chl. <sup>1</sup>	Quartz
1	B21t	48.4	34.8	1.21	28	9	31	12		++
2	B22t	48.1	27.1	1.36	31	7	15	14		++
3	B3	57.9	45.4	1.53	38	8	19	15		++
4	C	52.7	42.3	1.47	36	7	21	15		++
5		63.9	52.4	ND	45	8	ND	ND		++
10		60.2	49.5	2.31	42	7	12	23		++
11		54.0	43.5	2.04	36	7	12	20	+	++
12		48.1	42.5	2.25	32	10	14	22	+	++
14		47.8	42.3	2.02	33	11	ND	20	+	++
15		51.8	38.1	1.96	32	9	15	20		++
17		51.9	37.5	2.09	31	9	16	21		++
21		43.8	30.4	1.39	24	9	29	14		+
27		51.4	39.5	1.56	23	13	32	16		+
28		44.6	34.2	2.10	28	7	16	21		++
29		50.9	43.0	1.73	36	5	20	17		++
32		51.4	41.9	1.70	35	6	27	17	+	+

<sup>1</sup> Relative mineral content estimated from X-ray powder diffraction data. ++ > +. ND = not determined.



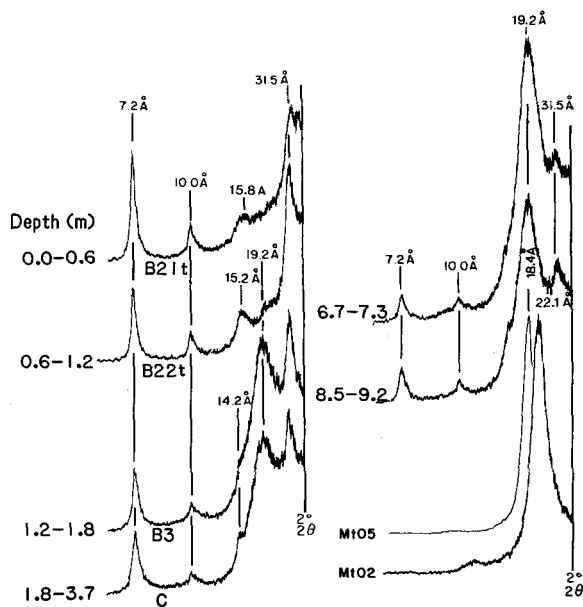


Figure 7. X-ray powder diffraction patterns of coarse (2–0.2  $\mu\text{m}$ ) clay fractions from core Ro 1 showing variations with depth in d-spacings of the octadecyl- $\text{NH}_3$ -clay complexes. All patterns were run at 1000 cps and a time constant of 2 sec (CuK $\alpha$  radiation).

increase in the amount of high-charge smectite and the corresponding decrease in the amount of mica from horizon C to B21t (Table 2) suggest that the formation of the high-charge smectite may be related to weathering of the mica.

The K-fixation capacity ( $\beta$ ) is a direct measure of the loss of CEC on saturation of the clay with K and drying overnight at 110°C and is related to the amount of high-charge smectite present. Decreasing values of  $\beta$  indicate that the high-charge smectite is most abundant in the B21t horizon and decreases with depth (Figure 6). The greater abundance of the high-charge smectite in the coarse clay fractions (2–0.2  $\mu\text{m}$ ) also indicates that it is a product of mica weathering because mica occurs mainly in the coarse clay fraction.

Expandable 2:1 layer silicate minerals react with large organic n-alkylammonium cations to give basal spacings which are strongly dependent upon the layer charge of the mineral and the chain length of the cation (Weiss, 1963; Lagaly and Weiss, 1969, 1976; Lagaly, 1981). The octadecylammonium complexes of the high- and low-charge species of smectite in the overburden materials are characterized by peaks at 32 and 19 Å, respectively (Figure 7). The basal spacings of two reference smectites Mt02 and Mt05 (with CEC values of 140 and 85 meq/100 g, respectively) are also included in Figure 7. The reference smectite with a higher charge (Mt02) gave a higher basal spacing (22 Å) compared to that of the low-charge smectite (18.4 Å). The d-spacing of the high-

charge smectite in the overburden materials is greater than that of reference smectite Mt02 by approximately 10 Å, suggesting that the high-charge smectite in the overburden materials may be regularly interstratified with a 10-Å, mica-like component. Such interstratification would explain the fairly large d-spacing (32 Å) observed for this mineral. According to Lagaly (1979), n-alkylammonium cations only react with the smectitic component of such a mixed-layered mineral when the sample is treated for only a short time (24 hr). If this interpretation is valid, the d-spacing of the smectitic or expandable component is simply the difference between 32 and 10 Å (22 Å), a value approximately equal to that of reference smectite Mt02.

The low-charge smectite in the less weathered samples at greater depth gave d-spacings that are fairly close to those of the reference low-charge smectite (Mt05). From the relative changes with depth in the intensities of the 32 and 19 Å peaks, the amount of high-charge smectite increases, whereas the amount of low-charge smectite decreases towards the surface. The trends in Figure 7 suggest that the formation of the high-charge smectite is related to weathering processes. An alternative explanation is that the finer grained, low-charge smectite has been leached, leaving the coarser, high-charged material in greater abundance in the upper layers. However, this latter explanation does not explain fully these trends over the full depth investigated.

Low-charge smectite (montmorillonite) and high-charge smectite (beidellite) were observed by Nash (1979) in soils developed from similar sediments in Mississippi. The high-charge smectite was the dominant species in the surface horizons, whereas both low- and high-charge smectite were present in the deeper horizons. The high-charge smectite in the surface horizons showed basal spacings of 18 Å after Mg-saturation and glycerol solvation, and spacings of 14 Å after K-saturation and glycerol solvation. The basal spacings of the low-charge smectite remained at 18 Å after K-saturation and glycerol solvation. Nash (1979) concluded that the high-charge smectite is an alteration product of mica or glauconite and that the low-charge smectite was inherited from the parent materials.

The undisturbed native soils developed from the lignite overburden sediments in east Texas contain large quantities of smectite, particularly in the fine clay (<0.2  $\mu\text{m}$ ) fraction. Removal of these soils by strip mining operations could improve the agricultural potential of this region provided that materials with a medium texture are preferentially deposited on surface of the reclaimed land and plant nutrient needs are met by adequate fertilization. Only materials from the oxidized zones, however, should be placed on reclaimed land to reduce the acidity potential, because all of the acid-forming (sulfide) minerals in these materials have been oxidized.

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## REFERENCES

- Alexiades, C. A. and Jackson, M. L. (1966) Quantitative clay mineralogical analysis of soils and sediments: in *Clays and Clay Minerals, Proc. 14th Natl. Conf., Berkeley, California, 1965*, S. W. Bailey, ed., Pergamon Press, New York, 35–52.
- ASTM (1974) Standard method of test for forms of sulfur in coal (method D2492-68, reapproved in 1974): Annual Book of Standards part 26: *Amer. Soc. Test. Mater.* Philadelphia, Pennsylvania.
- Bernas, B. (1968) A new method for decomposition and comprehensive analysis of silicates by atomic absorption spectrometry: *Anal. Chem.* **40**, 1682–1686.
- Biscaye, P. E. (1965) Mineralogy and sedimentation of recent deep sea clay in the Atlantic Ocean and adjacent seas and oceans: *Geol. Soc. Amer. Bull.* **76**, 803–832.
- Dixon, J. B., Hossner, L. R., Senkay, A. L., and Egashira, K. (1982) Mineralogical properties of lignite overburden as they relate to mine spoil reclamation: *Proc. Symposium on Acid Sulfate Soils, Fort Collins, Colorado, 1979*, Amer. Soc. Agronomy, Madison, Wisconsin (in press).
- Egashira, K., Dixon, J. B., and Hossner, L. R. (1982) High-charge smectite from lignite overburden of east Texas: *Proc. Inter. Clay Conf., Bologna, Pavia, 1981* (in press).
- Farmer, V. C. (1974) The layer silicates: in *The Infrared Spectra of Minerals*, V. C. Farmer, ed., Miner. Soc., London, 331–363.
- Fisher, W. L. and McGowen, J. H. (1967) Depositional systems in the Wilcox Group of Texas and their relationship to the occurrence of oil and gas: *Gulf Coast Assoc. Geol. Soc. Trans.* **17**, 105–125.
- Henry, C. D. and Kastning, E. H. (1978) Environmental geology of the east Texas lignite belt: in *Proc. Gulf Coast Lignite Conference: Geology, Utilization and Environmental Aspects*, W. R. Kaiser, ed., Texas Bur. Econ. Geol. **R.I. 90**, 189–198.
- Jackson, M. L. (1969) *Soil Chemical Analysis-Advanced Course*: 2nd ed., Publ. by author, Dept. Soil Science, Univ. Wisconsin, Madison, Wisconsin, 895 pp.
- Kaiser, W. R. (1974) Texas lignite: near-surface and deep-basin resources. *Texas Bur. Econ. Geol. R.I. 79*, 69 pp.
- Karlsson, W., Vollset, J., Bjollykke, K., and Jorgensen, P. (1978) Changes in mineralogical composition of Tertiary sediments from North Sea wells: in *Proc. Int. Clay Conf., Oxford, 1978*, M. M. Mortland and V. C. Farmer, eds., Elsevier, Amsterdam, 281–289.
- Lagaly, G. (1979) The layer charge of regular interstratified 2:1 clay minerals: *Clays & Clay Minerals* **27**, 1–10.
- Lagaly, G. (1981) Characterization of clays by organic compounds: *Clay Miner.* **16**, 1–21.
- Lagaly, G. and Weiss, A. (1969) Determination of the layer charge in mica-type silicates: in *Proc. Int. Clay Conf., Tokyo, 1969, Vol. 1*, L. Heller, ed., Israel Univ. Press, Jerusalem, 61–80.
- Lagaly, G., and Weiss, A. (1976) The layer charge of smectitic layer silicates: in *Proc. Int. Clay Conf., Mexico City, 1975*, S. W. Bailey, ed., Applied Publishing, Wilmette, Illinois, 157–172.
- Mehra, O. P. and Jackson, M. L. (1960) Iron oxide removal from soils and clays by a dithionite-citrate system buffered with sodium bicarbonate: in *Clays and Clay Minerals, Proc. 7th Natl. Conf., Washington, D.C., 1958*, Ada Swineford, ed., Pergamon Press, New York, 317–327.
- Nash, V. E. (1979) Mineralogy of soils developed on Pliocene-Pleistocene Terraces of the Tombigbee River in Mississippi: *Soil Sci. Soc. Amer. J.* **43**, 616–623.
- Soil Survey Staff (1951) Soil survey manual: *U.S. Dept. Agriculture Handbook* **18**, 503 pp.
- Walker, G. F. (1967) Interactions of n-alkylammonium ions with mica-type layer lattices: *Clay Miner.* **7**, 129–143.
- Weiss, A. (1963) Mica-type layer silicates with alkylammonium ions: in *Clays and Clay Minerals, Proc. 10th Natl. Conf., Austin, Texas, 1961*, Ada Swineford and P. C. Franks, eds., Pergamon Press, New York, 191–224.
- White, R. L. (1978) Land reclamation in Texas—an opportunity: in *Proc. Gulf Coast Lignite Conf.: Geology, Utilization and Environmental Aspects*, W. R. Kaiser, ed., Texas Bur. Econ. Geol. **R.I. 90**, 199–208.

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**Резюме**—Исследовалась последовательность минералогического выветривания в осадках, залегающих лигнитовые пласты в стержне (глубиной 27 м) из Кальверт, Область Робертсон, северо-восточный Техас. Тенденции выветривания определялись на основе свойств и относительного распределения расширяющихся и не расширяющихся минералов. В этих осадках серные минералы являются наиболее чувствительными к выветриванию и могут наблюдаться в неизменной (уменьшенной) зоне только ниже 7 м. Образование ярозита и гипса в верхних 7 м стержня (окисленная зона) является результатом окисления сульфидов. Окисленная зона характеризуется также красно-коричневыми цветами (большое количество желтого цвета), увеличенным количеством железа, извлекаемого дитионитом и отсутствием хлорита. Хотя в этих осадках главные глинистые минералы (смектит, каолинит, слюда) в основном являются детритовыми, результатом выветривания есть увеличение содержания каолинита и уменьшение содержания слюды в направлении к поверхности. Повидимому, слюда изменилась в высоко-заряженный смектит, характеризующийся основным расстоянием 32 Å после прослойки с катионами октаэдрилового аммония. Высоко-заряженный смектит главным образом присутствует в почвенных пластах в верхней части стержня и его количество постепенно уменьшается с глубиной. Низко-заряженный смектит является наиболее обильным в невыветренных материнских осадках и его количество увеличивается с глубиной. [E.C.]

**Resümee**—Mineralogische Verwitterungsabfolgen in Sedimenten, die Lignit-Schichten überdecken, wurden in einem Bohrkern (27 m tief) von Calvert, Robertson County, Nordost-Texas untersucht. Verwitterungstrends wurden anhand der Eigenschaften und der relativen Verteilung von expandierbaren und nicht expandierbaren Mineralen ausgewertet. Die Sulfidminerale in diesen Sedimenten sind am meisten für die Verwitterung anfällig und wurden nur in der unveränderten (Reduktions-)Zone unter 7 m beobachtet. Die Oxidation der Sulfide führte zur Bildung von Jarosit und Gips in den oberen 7 m des Bohrkerns (Oxidationszone). Die Oxidationszone ist weiters durch rotbraune Farben (hoher Chromgehalt), einen höheren Gehalt an durch Dithionitextrahierbares Eisen und das Fehlen von Chlorit gekennzeichnet. Obwohl die häufigsten Tonminerale in diesen Sedimenten (Smektit, Kaolinit, Glimmer) weitgehend detritisch sind, führte die Verwitterung in Richtung zur Oberfläche zu einer Zunahme des Kaolinitgehaltes und einer Abnahme des Glimmergehaltes. Der Glimmer scheint in einen "high-charge" Smektit umgewandelt zu sein, der nach dem Austausch mit Oktadecylammoniumkationen durch einen Basalabstand von 32 Å charakterisiert ist. Dieser Smektit ist in den Bodenhorizonten an der Spitze des Bohrkerns am häufigsten und nimmt allmählich mit der Tiefe ab. Ein "low-charge" Smektit ist am häufigsten in den unverwitterten Ausgangssedimenten und nimmt mit der Tiefe zu. [U.W.]

**Résumé**—Des séquences d'altération minéralogique dans des sédiments emplacements au dessus de lits de lignite ont été investigués dans une carotte (27 m de profondeur) de Calvert, Robertson County, Texas du nord-est. Une évaluation des tendances d'altération a été faite basée sur les propriétés et les distributions relatives des minéraux expansibles et non-expansibles. Les minéraux sulfides dans ces sédiments sont plus susceptibles à l'altération et n'ont été observés que dans la zone non-altérée (réduite) sous 7 m. L'oxidation des sulfides a résulté en la formation de jarosite et de gypse dans les 7 m supérieurs de la carotte (zone oxidée). La zone oxidée est d'avantage caractérisée par des couleurs rouge-brunâtres (chroma élevé), une quantité supérieure de fer qui peut être extrait de la dithionite, et l'absence de chlorite. Malgré que les minéraux argileux majeurs dans ces sédiments (smectite, kaolinite, mica) sont largement détritiques, l'altération a résulté en une augmentation du contenu en kaolinite et une réduction du contenu en mica vers la surface. Le mica semble s'être altéré en une smectite à charge élevée caractérisée par des espacements de base de 32 Å après intercalation avec des cations octadecylammonium. La smectite à charge élevée est la plus abondante dans les horizons de sol au haut de la carotte et décroît de manière graduée proportionnellement à la profondeur. Une smectite à basse charge est l'espèce la plus abondante dans le parent non-altéré et augmente proportionnellement à la profondeur. [D.J.]