

DISCRIMINATION OF KAOLINITE VARIETIES IN PORTERS CREEK AND WILCOX SEDIMENTS OF NORTH-CENTRAL MISSISSIPPI

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Abstract—Use of a discriminant analysis has verified and grouped three suspected varieties of kaolinite found in kaolin-rich clay strata of late Paleocene to early Eocene age across north-central Mississippi. Initial identification of each type of kaolinite was based on clay-texture characteristics observed on scanning electron micrographs and the differences in pattern configurations of X-ray diffractograms. The discriminant function used for data treatment clearly segregated and grouped each variety. The discriminant variables were found to be the Hinckley index and, to a lesser extent, the Si^{++} content relative to the Al^{3+} content.

The oldest variety is the Blue Mountain clay, composed of preserved hexagonal plates usually clustered into booklets with a vermiform texture. The Ashland variety, stratigraphically younger than the Blue Mountain clay, appears to have been derived from the erosion of the Blue Mountain clay. The Ashland cannot be recognized by any type of diagnostic texture, as it is made up of individual plates that have been corroded and abraded to the point where a hexagonal outline can no longer be recognized. The Sardis variety is the stratigraphically youngest of the three varieties and is at least a second, or possibly a third generation detrital product. The Sardis clay can be recognized by a distinct “ribbon” or “swirl” texture commonly found in ball clays.

Data from this study are not sufficient for complete petrogenetic interpretation. However, speculation on possible differences in depositional environments and modes of deposition can be based on the data at hand. The Blue Mountain variety is considered from previous studies to be primary. The Ashland variety is probably a first generation alluvial clay. The Sardis variety appears to be a multiple generation, detrital product that accumulated as part of overbank swamp deposits.

Key Words—Kaolinite, Discriminant analysis, Scanning electron microscopy, Hinckley index, Ball clay, Clay deposition.

INTRODUCTION

Kaolinite is a common mineral ingredient in most of the clay-rich sediments of Paleocene and Eocene age across north Mississippi. Kaolin-rich clays, including the Panola County ball clays (Patterson and Murray, 1975) mined in north Mississippi, are used in the manufacture of china, brick and whiteware.

This report is based on data obtained through an earlier investigation concerning the probable occurrence and economic potential of high alumina clays in north-central Mississippi. This earlier study involved the mapping and mineral analysis of kaolinite-rich and bauxitic strata in: (1) the upper portions of the Porters Creek Formation, (2) strata equivalent to the Naheola Formation, and (3) the lower part of the undifferentiated Wilcox Formation. The purpose of the present study was to determine possible variations in the chemistry and structure of the kaolinite contained in kaolinite-rich strata exposed over north-central Mississippi.

During the early 1940's Wilcox and upper Porters Creek strata of north-central Mississippi were considered as potential economic sources of high alumina clay and bauxite and became the focus of petrologic studies conducted by the United States Geological Survey. The results of these and other studies (Tourtelot,

1964; Conant, 1941, 1965; Reed, 1948; Priddy, 1943; Lusk, 1956) alluded to the fact that kaolin-rich beds of late Paleocene and early Eocene age were located within and just west of a north-south trending outcrop of bauxite. The bauxite outcrop, approximately 0.8 km (0.5 mile) to 5 km (3 miles) wide is positioned along the uppermost limit of the Porters Creek outcrop (Figure 1).

East of the bauxite Paleocene and Cretaceous strata crop out. These units contain beds of sandy limestone, micrites, clays, muds, and siliciclastics. The clays and the clay portion of the muds and siliciclastics are composed chiefly of smectite, illite, a mixture of opal-CT and montmorillonite, and the zeolites clinoptilolite and phillipsite (Raybon, 1982). Beds of Wilcox sediments composed of fluvial and deltaic sands, muds, and kaolinite-rich clay and clay-sands occur west of the bauxite outcrop.

SAMPLING AND ANALYSIS

Analytical procedure

Samples of clay, sandy clay, and mud were collected from 31 localities over five counties in north Mississippi (Figure 1). Road-cut and stream-cut exposures were sampled over an area extending from the western

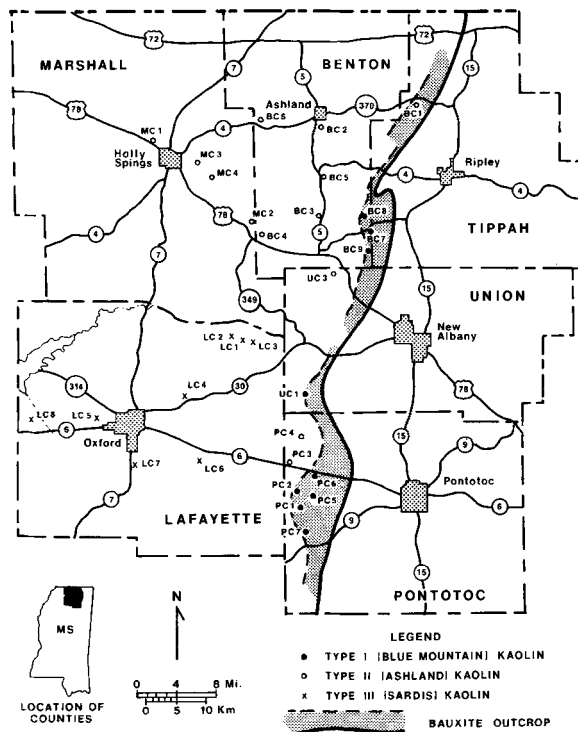


Figure 1. Study area and sample site locations.

margin of the bauxite outcrop westward across the Eocene Wilcox outcrop.

Sampled material was air dried, crushed and dry sieved. The $<62\text{-}\mu\text{m}$ fraction was examined by X-ray diffraction (XRD) to determine the bulk mineralogy. Next, the $<4\text{-}\mu\text{m}$ fraction of each sample was separated by sedimentation and centrifugation (Whittig, 1965) then examined by X-ray diffraction using scans from 3° to $40^\circ 2\theta$. A split from the $<4\text{-}\mu\text{m}$ fraction of each sample was also examined by scanning electron microscopy (SEM) for textural and fabric characteristics. A second $<4\text{-}\mu\text{m}$ split of each sample was commercially analyzed for the concentrations of Al^{3+} , Si^{4+} , Ti^{4+} , Fe^{3+} , Na^+ , Ca^{2+} , K^+ , Mg^{2+} , P^{5+} , Mn^{2+} and Cr^{3+} oxides, and moisture and organic contents. The percentage values of all oxides less than 0.1 were combined and listed numerically as secondary oxides.

X-ray diffraction and SEM analysis

The XRD patterns indicated variation in the apparent kaolinite crystallinity which appeared to range from being "well crystallized" to b-axis disordered (Figure 2). Therefore, Hinckley index values (Hinckley, 1963) were determined for the kaolinitic portion of each sample. These values, however, were not determined for the purpose of establishing any sort of scale of kaolinite crystallinity. Rather, these numerical values were determined for use as a predictor variable in the following discriminant analysis.

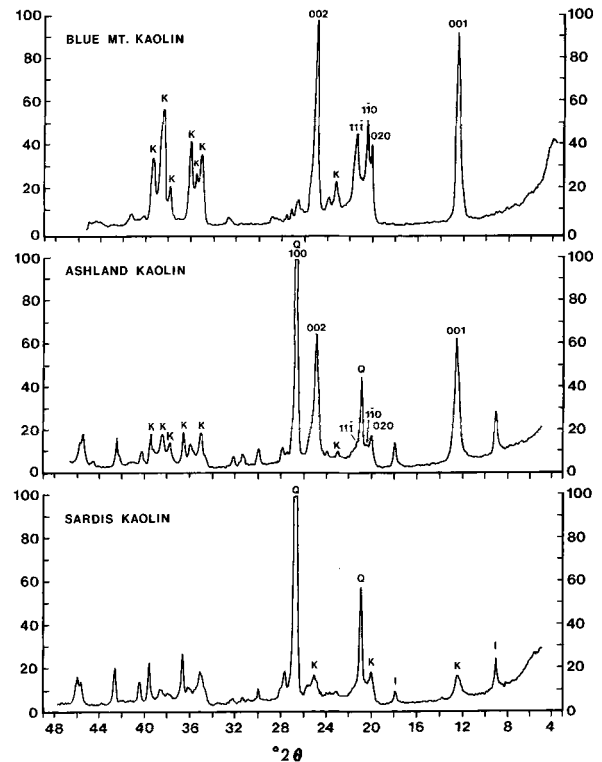


Figure 2. X-ray diffraction patterns of powdered Blue Mountain, Ashland, and Sardis kaolinites. Numerical values are d spacings for kaolinite. K = Kaolinite, Q = quartz, I = illite or fine-grained muscovite.

According to studies by Keller (1977), specific varieties of kaolinite can be recognized on the basis of clay texture characteristics as observed in scanning electron micrographs. Furthermore, Keller (1976a, 1976b, 1976c) has demonstrated that it is possible using SEM to recognize and even define environments of origin, as well as identify source material.

SEM examination of the clay fraction of north-central Mississippi samples suggested the possibility, based on textural characteristics, that more than one variety of kaolinite may exist. This idea prompted an initial establishment of three varieties or types of kaolinite within the Paleocene and lower Eocene strata of north-central Mississippi. The first type or variety is the Blue Mountain kaolin. It can be easily recognized in scanning electron micrographs by a vermiform texture comprised of long books of well-formed, unaltered hexagonal plates of kaolinite generally 0.1 to $0.2\ \mu\text{m}$ thick (Figure 3). Individual kaolinite particles can also be recognized as well-formed, unaltered hexagonal plates. The second type of variety is the Ashland kaolinite that has a texture consisting of random accumulations of individual plates. Most of the individual clay particles are thin, severely fragmented, and highly etched plates. The plates have irregular and ragged-appearing edges to the extent that the hexagonal outline is no

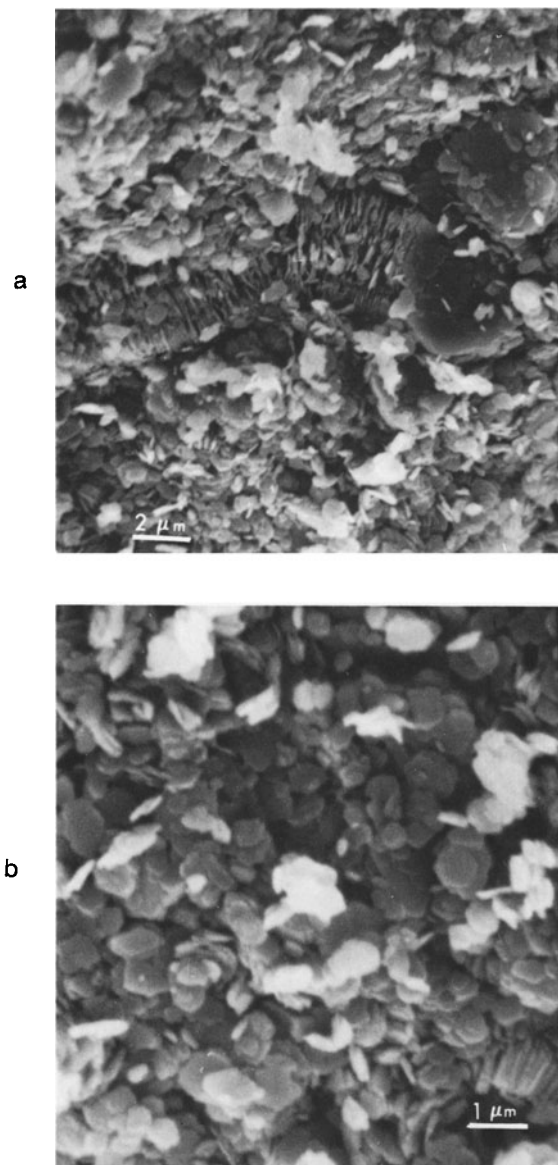


Figure 3. Scanning electron micrographs of Blue Mountain kaolin. a) vermiform texture, b) hexagonal outline of individual kaolinite grains.

longer recognizable (Figure 4). The third type or variety, the Sardis kaolinite, has a texture in which it is difficult even to recognize individual plates. Such plates are greatly deteriorated and essentially nondescript. The outstanding, characteristic texture is a tight binding or agglutination of these plates into laminar ribbon-like structures (Figure 5) similar to the characteristic "swirl" texture of ball clays (Keller, 1976b).

Variability in the Hinckley index and in the silica and alumina composition further indicate a possible grouping. Figure 6 is a composite of a series of graphs that illustrate in an empirical way the relationships

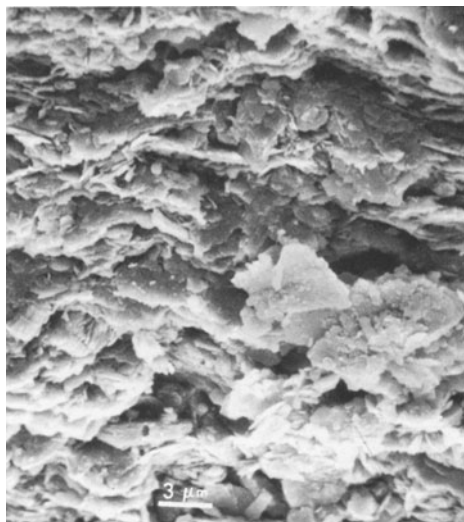


Figure 4. Scanning electron micrographs of Ashland kaolinite showing a lack of texture and the irregular outlines of individual etched grains.

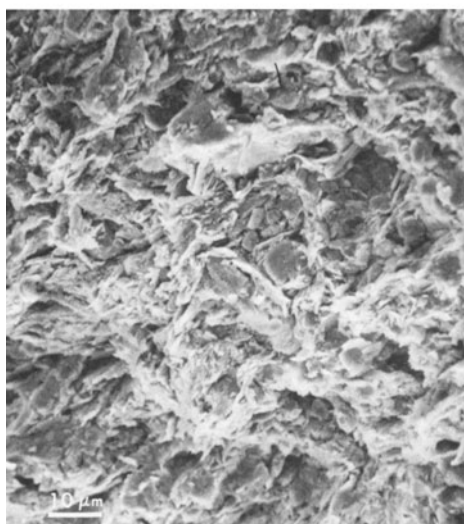
between the derived variables. The abscissas represent samples only and have no numerical scaling. The ordinates, however, consist of scaled percentage values plotted in ascending order from left to right. It is apparent from the figure that the weight percentages of alumina, silica, the secondary oxide categories, plus the Hinckley index are the most probable discriminating variables if the observed separate grouping of three types of kaolinite actually exists.

Figure 6 shows that values of the Hinckley index are

a



b



c

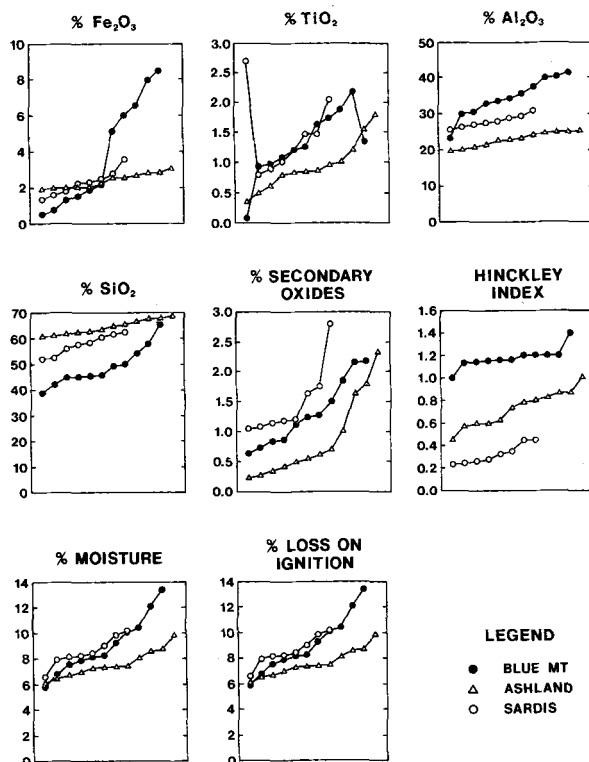
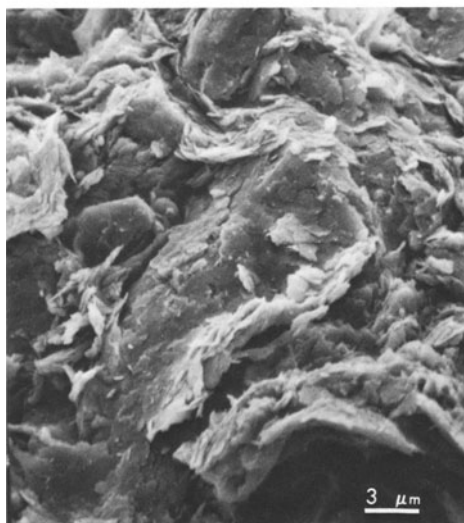


Figure 6. Comparison of specific physical and chemical parameters between the Blue Mountain, Ashland and Sardis kaolinite varieties.

greater than 1.0 for the Blue Mountain kaolin, and range from 0.4 to 0.9 for the Ashland kaolin and 0.2 to 0.4 for the Sardis kaolin. Also shown is a separation in Al_2O_3 values with the Blue Mountain clay having the highest values and the Ashland variety the lowest. Separation is also evident for the SiO_2 content, which is highest for the Ashland clay and lowest for the Blue Mountain clay. It is also interesting to note that the Sardis kaolin has the lowest Hinckley values, but higher Al_2O_3 and lower SiO_2 values than those for the Ashland kaolin. This suggests that the Ashland and Sardis varieties may have been derived from different sources.

Data treatment

Data for eight variables per sample location were used to calculate the multiple discriminant function. This procedure allowed a determination of whether or not clay type could be discriminated by values of Hinckley index, Fe_2O_3 , TiO_2 , Al_2O_3 , SiO_2 , secondary oxides, moisture content, or loss-on-ignition.

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Figure 5. Scanning electron micrographs of Sardis kaolinite. a) laminar texture, b) laminar-to-‘swirl’ texture, c) ‘swirl’ or ‘ribbon’ texture.

Table 1. Discriminant analyses.

	Function 1	Function 2
Eigenvalue	14.693	2.052
Relative percent	87.75	12.250
Canonical correl.	00.968	00.820
Wilks lambda	00.021	00.328
Chi-Square	94.789	27.335
Degrees of freedom	16.000	7.000
Significance level	00.000	00.000

The use of a discriminant function to analyze a series of variables assumes that these variables belong to a set of samples that have been classified into two or more groups. The purpose of a discriminant analysis is to demonstrate a grouping of sample sets that is based essentially on the relationship between criterion and predictor variables (Kachigan, 1986). The criterion variable is generally dichotomous and will have two or more qualitative values. The predictor variables, on the other hand, are quantitative in nature. Basically, discriminant analysis is a technique which maximizes the difference between *a priori* groups. It does so through the calculation of a linear combination of independent variables (canonical variables) that can be used as predictors for group membership.

The basis for a discriminant analysis is the discriminant function. This function uses a weighted combination of values for predictor variables to classify an object into one of two or more criterion variable groups. The criterion variable in this study is the clay type based on XRD patterns and the textural characteristics observed using SEM. The statistical software used for the discriminant function analysis is STATGRAPHICS distributed by STSC, Inc., Rockville, Maryland, and SPSS-X (Statistical Package for the Social Sciences), distributed by SSPS, Inc., Chicago, Illinois.

The discriminant function uses one or more linear combinations of discriminating variables in the form:

$$D_i = \beta_{11}Z_1 + \beta_{12}Z_2 + \beta_{13}Z_3 + \dots + \beta_{1p}Z_p$$

where D_i is the discriminant score on the function, i , the β 's are the weighted coefficients, and the Z 's are the standardized values of the p variables used in the analysis. The functions are formed in such a way as to achieve maximum separation of the groups. The maximum number of functions that can be derived is equal to the number of discriminating variables or one less than the number of groups, whichever is the smaller.

In this study only two functions are possible. The eigenvalues and associated canonical correlations (Table 1) indicate the relative ability of each function to separate the groups. The first function has considerable discriminating power (88%). The small value for Wilks lambda further suggests that a considerable amount of discriminating power exists in the variables being used (Cooley and Lohnes, 1971).

Table 2. Standardized discriminant function coefficients.

Variables	Function 1	Function 2
CI	1.12371	0.41254
SiO ₂	8.74182	-0.11267
Al ₂ O ₃	6.59934	-1.39437
Fe ₂ O ₃	3.09711	-0.07731
TiO ₂	1.32798	-0.02226
SEC-OX	0.39509	-0.21804
IG-LOSS	2.11878	0.70214
MOISTURE	0.57980	-0.31235

CI = Hinckley index; SEC-OX = secondary oxides; IG-LOSS = loss on ignition.

The discriminant equations for each function are, for function 1;

$$D_1 = 9.096 I + 1.659 \text{ Si}^{4+} + 1.863 \text{ Al}^{3+} \\ + 1.665 \text{ Fe}^{3+} + 2.474 \text{ Ti}^{4+} + 0.665 \text{ SEC-OX} \\ + 1.305 \text{ IGL} + 1.273 \text{ MOIS}$$

and, for function 2;

$$D_2 = 3.339 I - 0.021 \text{ Si}^{4+} - 0.394 \text{ Al}^{3+} \\ - 0.042 \text{ Fe}^{3+} - 0.041 \text{ Ti}^{4+} - 0.365 \text{ SEC-OX} \\ + 0.422 \text{ IGL} - 0.686 \text{ MOIS}$$

where; I = Hinckley index, SEC-OX = secondary oxides, IGL = loss-on-ignition, and MOIS = moisture content.

The value of lambda, however, is increased in function 2. This indicates a decrease in discriminating power to 12% as some of the discriminating power is removed and placed in the first function. The Chi-Square value, on the other hand, indicates that a small, yet statistically significant amount of discriminating information still exists in function 2.

The traditional methods for evaluating a discriminant analysis are using either the group means and associated F-values for each predictor or the magnitudes of the standardized discriminant weights. Both methods can be highly misleading if the predictor variables happen to be intercorrelated themselves (Dillon and Goldstein, 1984, p. 372). Using the discriminant loadings, which give the actual correlation of each predictor variable with a discriminant function, is probably the most effective way of evaluation.

In this study, the standardized discriminant function coefficients (Table 2) indicate that silica and alumina are the strongest contributors to function 1. Alumina and loss-on-ignition appear to be the main contributors to function 2. From the raw data it appeared that the Hinckley index should have been the strong contributor in function 1. Perhaps the real strength of this particular variable is subdued because it is correlated with one or more other variables. On the other hand, the discriminant weights of silica and/or alumina, because they are collinear predictors, may be inflated artificially at the expense of the Hinckley index. If we

Table 3. Discriminant loadings.

Variables	Function 1	Function 2
CI	0.71720	-0.25958
Fe ₂ O ₃	0.09994	0.08355
Al ₂ O ₃	0.27007	0.72855
SiO ₄	-0.23938	-0.65760
IG-LOSS	0.05856	0.24263
SEC-OX	-0.01006	0.22015
MOISTURE	-0.06050	0.19160
TiO ₂	0.06631	0.18711

CI = Hinckley index; SEC-OX = secondary oxides; IG-LOSS = loss on ignition.

examine the discriminant loadings, which actually are the correlations between the discriminating variables and the canonical discriminant functions (Table 3), we can see that the Hinckley index is the most highly correlated variable in function 1. Silica and alumina are more highly correlated in function 2. Furthermore, iron has its highest correlation in function 1 and titanium, loss-on-ignition, moisture content, and secondary oxides are better correlated in function 2. Each standardized discriminant function in Table 3 represents the relative contribution of the associated variable to that function.

Function 1 distinguishes groups 1, 2 and 3. Function 2 distinguishes between groups 2 and 3, and 2 and 1 (Figure 7). All samples designated as Blue Mountain (type I) kaolin fall into the predicted group 1. There is a slight overlap between groups 2 and 3 as one sample could be classified as either an Ashland (type II) or a Sardis (type III) kaolinite (Table 4). A plot of the discriminant scores (Figure 7) shows a distinct grouping for each of the three kaolinite varieties. The relative locations of each group are summarized by the group centroids.

DISCUSSION

Strata of Blue Mountain kaolinite are restricted to narrow and discontinuous bands along the uppermost portion of the Porters Creek clay and early Naheola outcrops (Figure 1). These beds range from 1–1.5 m (3 to 5 ft) in thickness and are composed mostly of white, unctuous clay plus minor amounts of clay-size quartz and muscovite. Blue Mountain clay produces XRD traces that show a complete family of well-defined kaolinite reflections, including high intensity and sharp

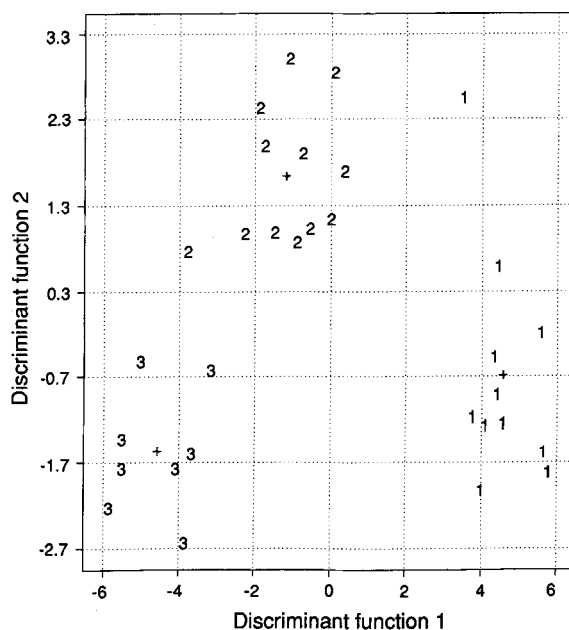


Figure 7. Plot of the discriminant function for Blue Mountain (1), Ashland (2), and Sardis (3) kaolinites. All three kaolinite varieties are well defined by function 1 as well as function 2. The centroid for each group is located at (+).

001 and 002 reflections (Figure 2). Furthermore, the reflections at 110 and 111 have peak-height ratios which yield Hinckley indices of 1.0 and greater.

Traditional residuum, detrital deposition, and ionic or colloidal precipitation have been among the various genetic models considered for the origin of the Blue Mountain kaolinite. The localized leaching of a smectite or illite producing residual deposits of bauxite, which later were locally resilicified to produce a kaolinite, was initially proposed as a petrogenetic model by Mellon (1939) and later by Priddy (1943). One problem with this model lies in the fact that the required parent smectite would have to be derived from the upper Porters Creek clay. Unfortunately, this material in north Mississippi is predominantly opal-CT, not smectite-rich (Raybon, 1982). Usually, a kaolinite resilicification product will commonly retain the pisolitic structure of the original bauxite. The Blue Mountain kaolinite is indeed locally pisolitic, but petrographic examination of the pisolite structures suggests that the pisolite morphology is not the type likely to have been

Table 4. Classification results.

Actual group	Predicted group (Count & Percentage)						
	1	2	3	Total	1	2	3
1	11	00	00	11	100.00	00.00	00.00
2	0	11	1	12	0.00	91.67	8.33
3	0	0	8	8	0.00	0.00	100.00

formed during the leaching of a smectite with subsequent formation of residual kaolinite (Thompson, 1981). The morphology of these structures suggests instead either a direct precipitation of suspensoid (Allen, 1952) or the diagenetic development of kaolinitic structure from precipitated gibbsite (Curtis and Spears, 1971).

Other genetic models that have been considered include: (1) differential flocculation of kaolin-rich detritus along the seaward regions of deltas where the fresh waters bearing the clay particles in suspension mix with saline marine waters (Griffin and Parrott, 1964; Smoot, 1960; Williams and Bergenback, 1968; Snowden and Forsthoft, 1976), (2) transport of detrital kaolin into coastal swamps and marshes (MacNeil, 1951; Reed, 1952; Tourtelot, 1964; Conant, 1965), and (3) selective, colloidal precipitation of kaolinite (Williams and Bergenback, 1968; Clark, 1979), or ionic precipitation of gibbsite and/or kaolinite in marine-fringed, paludal or lacustrine environments (Burchard, 1924, 1925; Curtis and Spears, 1971; Velton, 1972).

A more detailed and comprehensive study of north Mississippi bauxite and kaolinite by Thompson (1981) concluded that these materials are ionic precipitates. Accordingly, the events that led to the deposition of the bauxite and kaolinite started with the accumulation of Porters Creek and Naheola prodelta muds and clays over a broad, shallow-but-quiet marine shelf. This initial event was followed by the construction of juvenile deltas with broad interlobate regions consisting of tidal, supratidal and freshwater marshes and swamps. Sluggish, low-gradient streams draining into these small basins contained high concentrations of Al^{3+} , Fe^{2+} and Si^{4+} , and colloidal detritus derived from the erosion of weathered uplands that included the outcrops of the Porters Creek and Clayton Formations, and several Cretaceous units. As the ion-saturated stream waters entered the nearshore basins the change in pH and electrolyte concentrations caused the precipitation of gibbsite. Subsequent formation of kaolinite took place during periods of high silica influx.

Beds of Ashland clay range in thickness from 1 to 4.5 m (3–15 ft), are light grey in color and commonly quite silty. Ashland clay is composed mainly of kaolinite but does contain small amounts of smectite, muscovite and clay-size quartz. This variety of kaolinite yields a Hinckley index between 1.0 and 0.5 and prominent 001 and 002 XRD peaks (Figure 2). Almost all of the XRD reflections for kaolinite are present but they are not nearly as well defined as those given by the Blue Mountain clay.

The Ashland clays are Naheola and early Wilcox in age, which was a period of accelerated delta construction following the initial stages of delta formation that began during latest Porters Creek time. Deltas formed at this stage eventually coalesced to form broad, low-gradient delta plains. Stream systems that flowed over

these low-gradient delta plains became the depositional sites of fine-grained and clay-size detritus (Duplantier, 1975) containing beds of Ashland clay.

The Sardis clay occurs in thin beds ranging in thickness from less than 0.3 m to no more than 1.5 m (1 to 5 ft) in thickness. These beds are light grey in color and interlaminated with a dark grey-to-black, organic-rich, predominantly kaolinitic clay. These beds are also interbedded with ferruginous muds and fine, argillaceous sands containing organic debris deposited in swamp or marsh environments. The Sardis clays are capped by soil and typically overlie ferruginous sands and ironstones. The low Hinckley index, usually <0.5 , and the broad, low 001 and 002 XRD reflections suggest that Sardis clay might be halloysite. Scanning electron micrographs, however, do not reveal the elongate morphology typical of halloysite (Keller, 1977), but show instead a textural morphology previously described as “ribbons” or “swirls” of tightly-packed, fragmented kaolin particles.

The dominant clay species of the Sardis clay is a b-axis disordered kaolinite. Secondary mineralogy includes silt-to-clay size quartz, smectite, muscovite, and heavy minerals. The Sardis clay is found higher in the Wilcox section than the Ashland clay. It appears to be a ball clay similar to that mined to the west in Panola County.

CONCLUSIONS

The object of this study was to verify statistically an apparent grouping of three varieties of kaolinite that can be recognized in the kaolin-rich strata of late Paleocene to early Eocene age in north-central Mississippi. Initial identification of each of the three varieties was accomplished through interpretation of XRD patterns and SEM photomicrographs. It was verified statistically that the apparent differentiating parameter is the degree of crystallinity (Hinckley index) and composition, especially the Al^{3+} content relative to Si^{4+} . Why this difference exists may be related to a number of variables, especially the petrogenetic parameters that could have existed in the final depositional environment of each variety.

Data obtained in this study were not substantial enough to permit a complete petrogenetic interpretation as to why or how the three kaolinite varieties formed in the upper Paleocene and lower Eocene strata of north-central Mississippi. Keller and Stevens (1983) have suggested that certain aspects of clay texture observed on SEM micrographs may reveal not only a mode of deposition, but possibly an identification of the depositional and post-depositional environments. This idea is feasible because the textural pattern of a detrital clay can be the result of clay particle movement during detrital or even ionic/colloidal sedimentation, and also during post-depositional dewatering and compaction (Keller, 1976b). Therefore, some suppositions

can be made from the available data. Possible differences in depositional environments and modes of deposition based on clay texture characteristics combined with stratigraphic position, composition and geometry of the outcrop patterns may account for the different varieties of kaolinite.

The Blue Mountain variety is late Paleocene in age and the oldest of the three varieties. This material is presently considered to be a primary clay. It is considered to be the product of either ion or colloidal deposition in a marine fringed environment.

The Ashland variety, on the other hand, is also late Paleocene in age but stratigraphically younger than the Blue Mountain variety (probably Naheola or very early Wilcox). This variety is perhaps a first generation detrital product of eroded Blue Mountain clay deposited as an alluvial clay within the channels of delta plain fluvial systems.

The Sardis variety is the youngest of the three types being early Eocene (middle Wilcox) in age. Perhaps this variety is a multiple generation, organic-rich detrital product with final deposition in overbank swamps and marshes associated with a delta plain fluvial system.

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