# Fe SUBSTITUTION FOR AI IN GLAUCONITE WITH INCREASING DIAGENESIS IN THE FIRST WILCOX SANDSTONE (LOWER EOCENE), LIVINGSTON PARISH, LOUISIANA

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Abstract—Glauconites in early ankerite concretions, ferroan calcite-cemented sandstones, and uncemented sandstones in the first Wilcox sandstone of the Lockhart Crossing field, Livingston Parish, Louisiana, show a progressive substitution of Fe for octahedral Al with increasing diagenesis. An octahedral Fe content of 0.50 atoms was calculated from glauconite located in early ankeritic concretions. Octahedral Fe averaged 0.60 and 0.90 atoms in later ferroan calcite-cemented sandstone and uncemented sandstone, respectively. Corresponding octahedral Al averages were 1.16, 1.03, and 0.67, respectively. A systematic increase in average interlayer K from 0.49 to 0.54 to 0.61 was also observed, with apparent increases in diagenesis. All element determinations were made with an electron microprobe and recast on an anion equivalent basis to structural formulae based on the  $O_{10}(OH)_2$  unit. The clay preserved in the early ankerite concretions was found to be an illite/smectite containing about 20% expandable layers, and the mineral in the glauconite pellets from uncemented areas of the sandstone, an ordered glauconite. "Minus cement" porosities of the sandstone indicate that glauconitization may have taken place at burial depths greater than 0.6 to 1.8 km, but the mechanism for the incorporation of Fe<sup>3+</sup> in the glauconite at that depth is not apparent.

Key Words-Ankerite cement, Diagenesis, Glauconite, Illite/smectite, Iron, Potassium.

## **INTRODUCTION**

Boles and Franks' (1979) benchmark papers on clay diagenesis suggested that a decrease in the Fe<sup>3+</sup>/Al ratio of illite/smectite (I/S) mixed-layer clays in the Wilcox Formation of south Texas may be related to decreased expandability, higher K uptake, and increased ordering of I/S material as a function of increased diagenesis. "Glauconite" exhibits similar changes in expandability and ordering with increased K uptake (Thompson and Hower, 1975), but, although the  $Fe^{3+}$  content is high in glauconite, no systematic diagenetic pattern has emerged. The purpose of this study was to investigate the mineralogy and the chemical composition of glauconite from the first Wilcox sandstone in the Lockhart Crossing field, Livingston County, Louisiana, in an attempt to increase our understanding of glauconite diagenesis. If a consistent diagenetic change in the Fe<sup>3+</sup>/ Al ratio of glauconite could be detected, it would be useful in interpreting geologic history, because of the widespread occurrence of glauconites in ancient sedimentary rocks.

# GEOLOGIC SETTING

The Wilcox Group (upper Paleocene? to lower Eocene) crops out from south Texas across the northern part of the Gulf Coastal Plain into western Georgia and ranges in thickness from a maximum of 2440 m to <610 m (Galloway, 1968). In the Lockhart Crossing field (Figure 1), from which oil is produced from sandstones of the uppermost part of the Wilcox Group, the formation is about 1160 m thick. The main petroleum reservoir, the first Wilcox sandstone, lies at a depth of about 3030 m and a temperature of about 100°C. It consists of two very fine grained to medium-grained sandstone units sandwiched between impermeable mudstones (Self *et al.*, 1986).

The predominant facies in the first Wilcox sandstone is a well sorted, very fine grained to fine-grained, glauconitic lithic arkose or feldspathic litharenite, which was deposited as a littoral bar sand. The other unit is a non-bioturbated, very fine grained to medium-grained feldspathic litharenite channel facies deposit. Both contain glauconite pellets similar to those illustrated in Figures 2A and 2B, although the pellets are most abundant in the lower part of the bar facies. Glauconite pellets in the first Wilcox sandstone commonly occur with wisps of bioturbated clay, as in Figure 2A, and are commonly medium-green to dark-green and range in size from 100 to 1000  $\mu$ m in diameter (Figures 2B) and 2C). Most of the pellets have a smooth, rounded or lobate morphology and are internally composed of randomly arranged clay aggregates containing little or no silt-sized grains. The pellets in the cemented zones generally lack shrinkage cracks and have not been physically deformed. Pellets in the uncemented zone may show minor compaction effects. Dark organic matter (O, Figure 2C) commonly forms a rim around the pellets.

The glauconite pellets occur in three different lithologic surroundings in the sandstone. The most distinct occurrence is in ankerite concretions, in which car-

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Figure 1. Location of Lockhart Crossing field, Louisiana. Modified from Self et al. (1986).

bonate cement completely fills all of the intergranular pore space. "Minus cement" porosities (i.e., porosities of cement-free materials) in the concretions are commonly near 50%, suggesting that the concretions formed soon after the sand was deposited, before significant compaction. Other glauconite pellets near ankerite concretions are encased by a second-generation cement composed of ferroan calcite, which also forms a rim around the ankerite concretions. This second-generation cement locally makes up more than 25 vol. % of the rock. Porosities in the two cemented zones are essentially zero. Other glauconite pellets occur in uncemented or cement-free areas of the sandstone, which have porosities that average about 20%. The decreases in the "minus cement" porosity and the tightness of the cemented zones suggest that the glauconite in these areas may have been isolated from its associated pore fluids at different times. Thus, the glauconite from the ankerite zone may be the least diagenetically altered material, and the glauconite from the uncemented zone may be the most diagenetically altered material because of its association with the open pore system. In this presentation, the glauconite pellets in these zones are assumed to represent three distinct diagenetic facies; all subsequent interpretations of the mineralogical and chemical data are based on this premise.

# **METHODS**

Table 1 lists the samples from the first Wilcox sandstone studied in this investigation. They came from different wells and represent the three diagenetic types described above. All the pellets came from the same general depositional environment and probably had similar initial compositions. The glauconite pellets were analyzed by the following procedures.

## X-ray powder diffraction (XRD)

Samples of the ferroan calcite-cemented sandstone and the uncemented sandstone containing abundant glauconite pellets were disaggregated ultrasonically. The pellets were magnetically concentrated and gently ground to a fine powder in an agate mortar and pestle. Randomly oriented glauconite powders were stepscanned at  $0.04^{\circ}2\theta$ /s from 2° to  $37^{\circ}2\theta$  and at  $0.02^{\circ}2\theta$ /s from 59° to 62.5°20 in a Philips APD 3520 X-ray diffractometer with monochromatic CuK $\alpha$  radiation and a  $\theta$ -compensating slit. The higher angle random powder patterns were used to measure the d(060) values of the glauconite to confirm their dioctahedral character. Oriented smears of air-dried, glycolated, and heated (300° and 550°C) samples were step-scanned at 0.04°2 $\theta$ /s from 2° to 37°2 $\theta$  to estimate the percentages of glauconite and interstratified smectite in the pellets.

# Electron microprobe

Electron microprobe analyses of glauconite pellets were performed on five polished thin sections of sandstone using a JEOL JXA 733 electron microprobe. The thin sections were first studied in transmitted light to locate and map areas containing glauconite. Forty-seven selected spots were analyzed using a 10-mm diameter beam and a 15-keV accelerating voltage. Kakanui hornblende and Smithsonian microcline were used as standards, and the quantitative oxide values for Si, Al, Fe, Mg, Ca, Na, and K were derived using a slightly modified Bence-Albee procedure (1968). The anhydrous totals were normalized to 100 wt. %, and structural formulae for the glauconite pellets were calculated assuming that all the Fe was ferric and using the total anion equivalency method based on the  $O_{10}(OH)_2$ (Bodine, 1987).

Table 1. Samples from the first Wilcox sandstone used in this study.

Sample	Well	Depth (m) (ft)	Lithologic type
MC10	Maggie Collins #1	3123.8 (10,246)	Uncemented sandstone
MS16	M. I. Stewart #1	3116.0 (10,220.5)	Uncemented sandstone
OB22	O. M. Barnett #1	3116.7 (10,222.8)	Ferroan calcite-cemented sandstone
TH14	A. Thom #1	3101.5 (10,173)	Concretion
TH23	A. Thom #1	3107.9 (10,194)	Concretion



Figure 2. Typical glauconite occurrence in sandstone of littoral bar facies. (A) Core slab photograph. (B) Photomicrograph in plain polarized light. G = glauconite. (C) Plain light photomicrograph of a glauconite pellet containing randomly oriented clays in interior. O = organic matter rim.





Figure 3. (A) Random X-ray powder diffraction (XRD) patterns of glauconite pellets from a ferroan calcite-cemented and an uncemented sandstone. (B) XRD patterns of oriented smear of glauconite pellet from uncemented sandstone at base of littoral facies.

#### RESULTS

## X-ray powder diffraction

Figure 3a shows a typical XRD pattern of randomly oriented glauconite pellets from a ferroan calcite-cemented and an uncemented sandstone. Several of the basal and prism reflections for glauconite (or illite) are present. The broad peaks suggest that the glauconite is poorly crystallized or is composed of very small particles. Traces of kaolinite, quartz, potassium feldspar



GLAUCONITE 060 REFLECTIONS

Figure 4. 060 reflections of two glauconite pellet separates. Values > 1.51 distinguish glauconite from celadonite.

and ankerite were also detected. Figure 3b illustrates the effects of the various treatments on the oriented smear of a glauconite separate. Glycolation produced little change in the XRD pattern, but the 300° and  $550^{\circ}$ C heat treatments increased the intensity of the peak near  $8.8^{\circ}2\theta$  suggesting the presence of some expandable (smectite) layers. The d(060) values of 1.5223and 1.5189 Å (Figure 4) for two typical glauconite separates suggest that the octahedral layer contains >0.70 Fe<sup>3+</sup> atoms per O<sub>10</sub>(OH)<sub>2</sub> (Buckley *et al.*, 1978). The d(060) values of both separates (>1.51 Å) distinguish them from celadonite (Bailey, 1980). The XRD results do not clearly establish whether glauconite or illite is the dominant non-expandable phase in this mixedlayer clay.

## Electron microprobe

The average, range, and standard deviation of the oxide percentages of glauconite from the three diagenetic types are reported in Table 2. Simple t-test statistics indicate that the Al<sub>2</sub>O<sub>3</sub> means are different at the 0.5% level of significance. Iron oxide means for the ankerite and ferroan calcite-cemented samples are different at the 5% level of significance, and the other populations are different at the 0.5% level of significance. The average structural formulae for glauconite from the three types are also presented. Although the chemical composition of the pellets varied from pellet to pellet, all from the uncemented sandstone can be classified as glauconite according to the definition recommended by AIPEA (Bailey, 1980), that is, "... an Fe-rich dioctahedral mica with tetrahedral Al3+ (or  $Fe^{3+}$ ) usually greater than 0.2 atoms per formula unit and octahedral R<sup>3+</sup> correspondingly greater than 1.2 atoms. Usually Fe<sup>3+</sup> is much greater than Al<sup>3+</sup>, and Mg<sup>2+</sup> is greater than Fe<sup>2+</sup> in the generalized structural formula." The other glauconite pellets contain a less Fe<sup>3+</sup>-rich 10-Å component that may be a disordered glauconite, according to the terminology suggested by



Figure 5. Relationship between number of interlayer K and octahedral Al atoms per  $O_{10}(OH)_2$  formula unit for glauconite from the three diagenetic types.

Hower (1961). The Wilcox pellets are composed of a mixed-layer clay mineral having 10-Å and smectite layers. The smectite layers make up <20% of the phase and interlayering appears to be ordered, R=1 (Reynolds, 1980). The non-expanding layers are illitic if oct. Fe < oct. Al, or glauconitic if oct. Fe > oct. Al. Theamount of K in the interlayer position of glauconite in the first Wilcox sandstone increases systematically with increasing diagenesis. The average number of K atoms increases from 0.49 per formula unit in the early ankeritic concretions (6.08 wt. %  $K_2O$ ) to 0.54 in the ferroan calcite-cemented sandstone (6.74 wt. % K<sub>2</sub>O) to 0.61 atoms in the uncemented sandstone (7.41 wt. % K<sub>2</sub>O) (Figure 5). Bentor and Kastner (1965), Cimbalnikova (1971), and Thompson and Hower (1975) showed that an increase in the number of K atoms per formula unit is related to a decrease in the proportion of expandable layers (smectite) in the interstratified structure. The data form overlapping populations, but in general the glauconite in the ankeritic concretions is disordered, containing an average of 81% glauconite layers (Figure 6). All glauconite in the ferroan calcitecemented sandstone is disordered and contains an average of 86% glauconite layers. Nearly all the glauconite from the uncemented sandstone is ordered, containing an average of 91% glauconite layers. With increasing diagenesis, the proportion of smectite layers in the glauconite/smectite decreases, as predicted by the smectite-to-illite reaction (Figure 6) described by Thompson and Hower (1975).

The amount of Al in the glauconite in the first Wilcox sandstone decreases as  $Fe^{3+}$  increases (Figure 7) and as expandability decreases. The glauconite also shows a progressive substitution of  $Fe^{3+}$  for octahedral Al with increasing diagenesis. An average octahedral Fe content of 0.50 atoms per formula unit (0.49 atoms of  $Fe^{3+}$ and 0.01 atoms of  $Fe^{2+}$ ) was calculated for glauconite from the ankeritic concretions. The proportions of  $Fe^{2+}$ and  $Fe^{3+}$  atoms were derived by charge-balancing the



Figure 6. Relationship between number of K atoms  $(K_n)$  per  $O_{10}(OH)_2$  and percentage of illite layers in glauconite and in illite/smectite (I/S). Mineralogical forms of glauconite are based on terminology from Hower (1961). Points and bars represent average and range of values from the three types. Base modified from Thompson and Hower (1975).

Table 2	. Average,	standard	deviation	, and	range	of oxide	e
weight j	percentages	of glauco	nite from	the t	hree d	liagenetio	С
types.							

	Concretion	Ferroan calcite-cemented sandstone	Uncemented sandstone			
Number of						
analyses	7	10	30			
Average composition (standard deviation) and range						
SiO <sub>2</sub>	55.76 (1.69)	57.71 (1.44)	56.31 (1.3)			
	54.11-58.81	56.45-61.09	52.58-59.37			
$Al_2O_3$	22.56 (1.89)	18.92 (2.3)	13.87 (1.71)			
	20.46-25.74	15.61-22.84	11.45-16.96			
$Fe_2O_3$	9.59 (1.27)	11.42 (2.27)	16.79 (1.8)			
	8.35-11.82	7.04-15.41	12.98-20.08			
MgO	4.96 (0.89)	4.55 (0.34)	4.98 (0.35)			
	4.06-6.78	4.06-5.08	4.45-6.08			
CaO	0.62 (0.29)	0.35 (0.25)	0.44 (0.25)			
	0.25-1.12	0.12-0.75	0.15-1.03			
$Na_2O$	0.42 (0.36)	0.32 (0.13)	0.19 (0.07)			
	0.16-1.19	0.15-0.62	0.08-0.37			
K <sub>2</sub> O	6.08 (0.72)	6.74 (0.4)	7.41 (0.43)			
	5.06-6.75	6.21-7.38	6.43-8.2			
	Average str	ructural formulae	:			
Tetrahedral						
Si	3.49	3.63	3.63			
Al	0.51	0.37	0.37			
Octahedral						
Al	1.16	1.03	0.67			
Fe <sup>3+</sup>	0.49	0.60	0.88			
Fe <sup>2+</sup>	0.01	0.00	0.02			
Mg	0.46	0.43	0.48			
Sum	2.12	2.06	2.05			
Interlayer						
ĸ	0.49	0.54	0.61			
Na	0.05	0.04	0.02			
Ca	0.04	0.02	0.03			

<sup>1</sup> All analyses have been normalized to 100% on an anhydrous basis. Average structural formulae are also listed.



Figure 7. Relationship between octahedral Fe and Al for glauconite pellets from the three diagenetic types of first Wilcox sandstone.

glauconite formula. The octahedral Fe content is 0.60 atoms (all Fe<sup>3+</sup>) in glauconite in ferroan calcite-cemented sandstone and 0.90 atoms (0.88 atoms of Fe<sup>3+</sup> and 0.02 atoms of  $Fe^{2+}$ ) in the uncemented sandstone. With progressive diagenesis the average Al content in the octahedral layer decreases from 1.16 atoms per formula unit in the concretions to 0.67 atoms in the uncemented sandstone. Although loss of expandability during illitization has been proposed as a source for iron in late diagenetic carbonates (Boles and Franks, 1979), the glauconite analyzed in the present study seems to have been a sink for Fe, by incorporating it into its structure. Assuming that the increase in Fe took place with minimal increase in density or volume, the glauconite was a source of Al during diagenesis because of this substitution.

The major chemical variation in the octahedral sheet of glauconite is the ratio of Al to Fe<sup>3+</sup>. Previous studies have shown that Al decreases as Fe3+ increases and the percentage of expandable layers decreases (Thompson and Hower, 1975; Weaver and Pollard, 1973). Bentor and Kastner (1965) urged caution in calculating the structural Al:Fe<sup>3+</sup> ratio, because some of the Fe<sup>3+</sup> may actually be present as free iron oxides. Thompson and Hower (1975) calculated structural formulae for samples before and after progressive acid dissolution and showed that Fe-, Al-, and Mg-hydroxyl groups can exist in the glauconite as "interlayer complexes or extraneous to the structure." The slight tendency towards a trioctahedral character suggested by the sums of the octahedral components being greater than 2.0 (Table 2) may be related to this problem, but no locally high concentrations of Fe were detected with the microprobe.

The Mg content of Wilcox glauconite samples ranges from 0.38 to 0.58 atoms per  $O_{10}(OH)_2$ , and its abundance appears to increase with decreasing Al and decreasing expandability (i.e., increasing diagenesis) (Figure 8). Cimbalnikova (1971) observed the opposite



Figure 8. Relationship between octahedral Mg and Al for glauconite pellets from the three diagenetic facies.

trend and reported a slight increase in Mg with increasing percentages of expandable layers and increasing Al. Ordered Wilcox glauconite (in the uncemented sandstone) contains between 0.42 and 0.58 Mg atoms per formula unit, more than that normally expected for ordered glauconite (0.30 to 0.40 atoms) (Odom, 1984).

Tetrahedral Al varies from 0.25 to 0.6 atoms, well within the 0.1 to 0.7 range reported by Weaver and Pollard (1973). In the Wilcox glauconite, average Al values are 0.51 for glauconite in concretions and 0.37 for glauconite in ferroan calcite-cemented and uncemented sandstone. Weaver and Pollard (1973) reported an average of 0.35 for glauconite from various settings. No simple correlation between the amount of tetrahedral Al substitution and the percentage of expandable layers is apparent for the present samples.

Accompanying the increase in K in the interlayer is an increase in average octahedral charge ranging from -0.12 in the octahedral layer of glauconite from the concretions to -0.26 in glauconite from the ferroan calcite-cemented sandstone to -0.33 in glauconite from the uncemented sandstone. The tetrahedral charge ranges from an average of -0.51 for glauconite in the concretions to -0.37 for both of the other glauconites. The total layer charge ranges from -0.63 in the concretions and the ferroan calcite-cemented sandstone to -0.70 in the uncemented sandstone. Charge balance appears to be fairly well maintained for all the glauconite samples, thus supporting the assumption that Fe<sup>3+</sup> is dominant.

# DISCUSSION

Hypotheses on the genesis of glauconite range from co-precipitation or crystallization of Fe-Mg-Al-Si-rich gels (Murray and Renard, 1891; Twenhofel, 1936) to the "layer lattice" hypothesis proposed by Burst (1958a, 1958b) and further developed by Hower (1961). According to the "layer lattice" hypothesis, glauconite forms by the transformation of degraded "layer-lattice" silicates. Under reducing conditions and in contact with Fe- and K-rich solutions, the degraded silicate substrate, preferably a 2:1 clay, transforms by structural ion exchange and uptake to glauconite. Odin (1975), Odom (1976), and Odin and Matter (1981) showed that a precursor phyllosilicate may not be required and proposed a "neoformation" hypothesis, in which glauconite forms by direct precipitation of ions from solution. Degraded materials in the pellet may be the source of the ions in the solution or the ions may have originated elsewhere. In both hypotheses, the nascent material (Odin and Matter, 1981) is presumed to be a low-K, highly smectitic mixed-layer clay containing more than 50% expandable layers.

Pelletal glauconite generally occurs in sediments deposited on the continental shelf; its morphology suggests a fecal pellet or other biogenic origin (Odom, 1984). The lobate to rounded shape of the glauconite pellets in the first Wilcox sandstone, their homogeneous internal appearance, and their abundance in the littoral bar sandstone is consistent with the origin suggested above. The low expandability of the mixed-layer clay and the predominance of Al in the octahedral laver of the pellets in the early formed concretions suggests that the precursor material was illite/smectite having about 20% expandability. These factors and the absence of shrinkage features indicate that transformation may have been the most important process responsible for the observed compositions of the Wilcox glauconite pellets.

That the mineralogy of the pellets continued to evolve during burial diagenesis is suggested by the different composition of the three diagenetic types found in the Wilcox. The range of composition for each diagenetic type is probably due to: (1) differences in the original composition of the precursor minerals in the pellets and/or (2) lack of complete equilibration during diagenesis. Some pellets may have been somewhat isolated from migrating pore fluids. Although the timing of glauconitization can not be established directly, clearly only those samples from pellets in the uncemented zone contain more Fe than Al in their octahedral layers. Thus, glauconitization must have been accomplished after the cementation with ferroan calcite and at depths at which the porosity had been reduced to <25%. Empirical porosity-depth curves constructed by Bond and Kominz (1984) for delithifying miogeoclinal sequences suggest that this porosity may be obtained at burial depths of 0.6 to 1.8 km.

Although glauconite is not an abundant mineral in the first Wilcox sandstone, glauconitization may have had a minor influence on the chemical evolution of the formation water because K, Fe, and some Mg must have been extracted from porewater, and Al must have been added as glauconitization progressed. K-feldspar and detrital muscovite have generally been recognized as potential sources for K (Hower *et al.*, 1976), but they have not been specifically identified in the present study. Fe and Mg in the glauconite were probably provided by the dissolution of the carbonates or the recrystallization of ankerite as a less Fe- and Mg-rich carbonate. Either of these carbonate-related processes could have occurred at depth and presumably produced Fe<sup>2+</sup> in solution, but the mechanism for the incorporation and oxidation of this Fe in the glauconite can not be identified. Al supplied by glauconitization may have precipitated as kaolinite.

The glauconite pellets in the first Wilcox sandstone have undergone compositional changes in the same sandstone unit similar to those found in glauconite of different age from various localities. Glauconite pellets preserved in ankerite-cemented areas, ferroan calcitecemented zones, and the open pore system thus illustrate a progressive substitution of  $Fe^{3+}$  for octahedral AI, a decreasing expandability of the mixed-layer clay, and increased K in the interlayer position.

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