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AGE OF THE OCMULGEE LIMESTONE (GEORGIA COASTAL PLAIN) BASED ON REVISED METHODOLOGY FOR THE K-AR AGE OF GLAUCONY

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ABSTRACT

Grains (0.15-1.0 mm) composed mostly of well-evolved glauconite, based on X-ray diffraction, visual data, and potassium measurement, are found dispersed within the Ocmulgee Limestone (latest Eocene) 20-22 feet above the basal contact at Taylor's Bluff, its type locality, on the Georgia Coastal Plain. These grains are botryoidal and dark grey-green to green-black in color. Some were separated from the limestone for K-Ar age study and yielded an age of 33.7 ± 1.0 Ma from a small sample, 18 mg, used for both potassium and argon-isotope measurements. This K-Ar age for the glauconite places the deposition of this interval of the Ocmulgee Limestone and its characteristic Eocene fossil assemblage close to the time of the Eocene-Oligocene transition (33.9 ± 0.1 Ma). The techniques described herein make it possible to measure the K-Ar age of glauconite present in small amounts using one weigh-out and thus decreasing analytical error. This technique makes it possible to determine numerical age values for limestone containing only small amounts of dispersed but well-evolved glaucony grains.

INTRODUCTION

The terms "glauconite" and "glaucony" are often used interchangeably to refer to a facies composed of green spheroidal pellets and green amorphous clays (Odin and Matter, 1981). Glauconite refers to an iron-rich dioctahedral mica whose tetrahedral sites may contain more than 0.2 trivalent cations (Al or Fe) per formula unit and whose octahedral sites contain 1.2 trivalent cations per formula unit (Bailey and others, 1979). Herein, we refer to the facies and grains as glaucony while reserving the term glauconite for the authigenic phyllosilicate mineral within glaucony pellets.

Glaucony pellets form at the sediment-water interface at depths typically between 50 and 500 meters water depth (Odin and Matter, 1981; Bornhold and Giresse, 1985; Harris and Whiting, 2000). Where slow sedimentation provides a favorable environment, glaucony pellets grow in size and change in both mineralogy and chemical composition from a glauconite-smectite to a glauconitic illite with high (6-8 wt%) potassium (K_2O) content (Bailey and others,

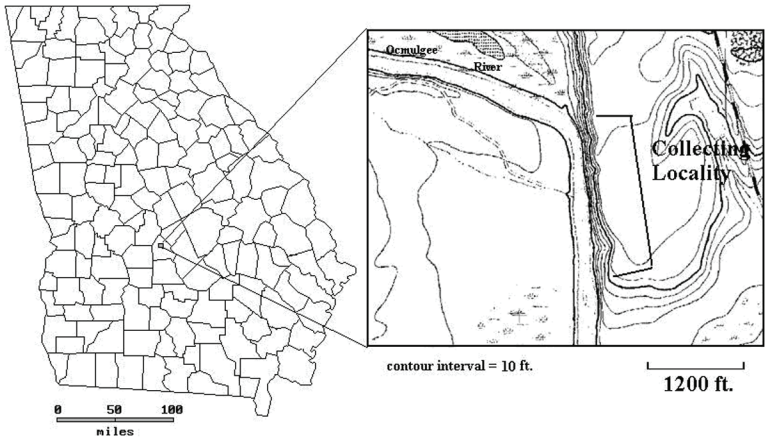

Figure 1: Locality map for Taylor's Bluff, east of the Ocmulgee River, north of Hawkinsville, Georgia., Hawkinsville, 7½ minute Quadrangle.

1979; Odin and Matter, 1981). Odin and Matter (1981) recognized four stages of evolution of the glaucony pellets (nascent, little-evolved, evolved, and highly evolved stages). In this evolution, both potassium and iron contents increase and the glaucony grains change from powdery or crumbly aggregates to larger grains with botryoidal shapes and smooth surfaces. Most of the constituents needed to form glauconite come from the glaucony facies or grains themselves (Odin and Matter, 1981).

During the development and early use of the K-Ar method, glauconite was considered a promising phase for determination of absolute geologic time given the authigenic growth of potassic phases within the glaucony pellets (e.g., Wasserburg and others, 1956; Lipson, 1958; Evernden and others, 1961; Odin, 1982a). Early K-Ar and Rb-Sr age values were typically about 5% less than the values expected from the biostratigraphic positions of the host rocks, which was then thought to be a consequence of diffusional loss of radiogenic Ar (Lipson, 1958; Hurley and others, 1960). Lipson (1958) noted a positive relationship between potassium content and "retention" of radiogenic argon. In some cases, K-Ar age values for glaucony have been larger than the expected values, which may be due to the presence of "inherited argon" within potassic detrital clays in "less evolved" glaucony (Lipson, 1958; Odin, 1982a). "Highly evolved" (6-8 wt.% K_2O) glaucony pellets are found more useful than "less evolved" glaucony pellets (< 4 wt.% K_2O) for determining numerical ages of strata by virtue of being less susceptible to losing radiogenic argon and having less inherited argon (Odin and Velde, 1975; Odin and others, 1977; Odin and Matter, 1981; Odin, 1982a; Odin, 1982b). Age values less than those expected from stratigraphic position may result from argon loss due to increase in temperature after burial to significant depths or from addition of potassium (Thompson and Hower, 1973). Where sediments have not been buried deeply, as for example in the Atlantic Coastal Plain, "evolved" glauconite has been shown to be an excellent K-Ar and Rb-Sr clock for determining the numerical ages of sedimentary strata (Harris and Bottino, 1974; Harris and Fullagar, 1989).

In this study, we describe a method for determination of the K-Ar age of glaucony whereby one weighed sample is used for both potassium and argon-isotope measurements. The K-Ar age of 33.7 ± 1.0 Ma for well-evolved glaucony grains from the Ocmulgee Limestone at its type locality on the Georgia Coastal Plain indicates that the limestone was deposited near the time of the Eocene-Oligocene transition, in the context of the currently accepted time scale for the Paleogene Period (Luterbacher and others, 2004).

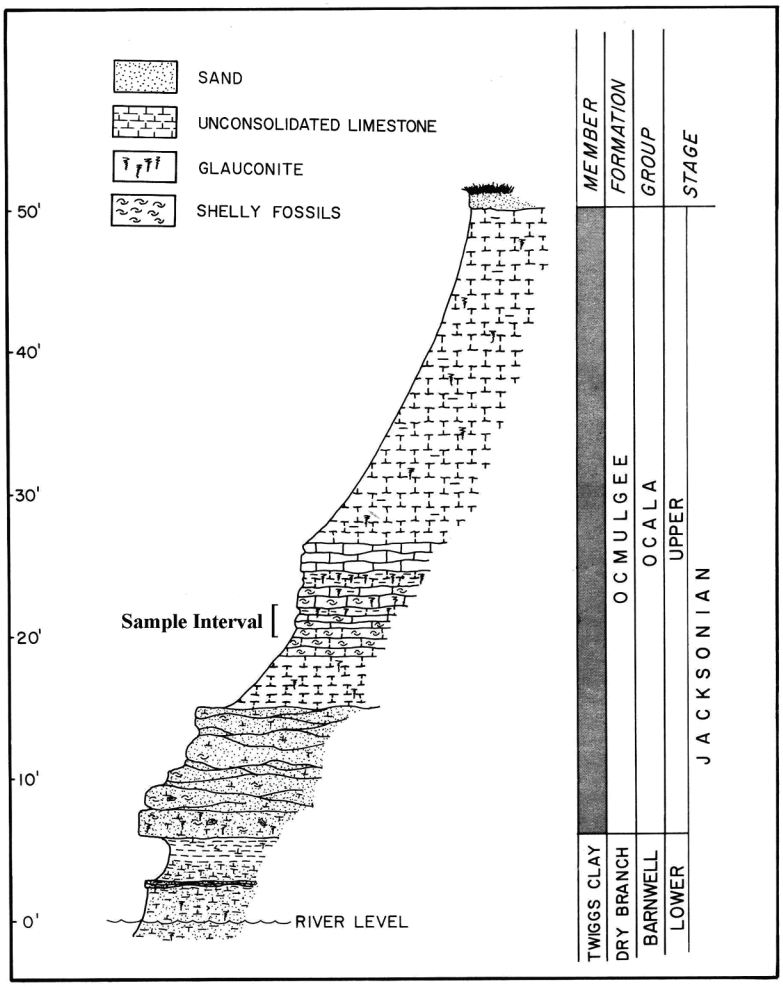

Figure 2: Stratigraphic column of the Ocmulgee Limestone at the type locality at Taylor's Bluff modified from Huddlestun and Hetrick (1986) to show the location of glaucony samples.

GEOLOGICAL SETTING AND LITHOLOGY

The Ocmulgee Limestone, as defined by Huddlestun and Hetrick (1986), extends from exposures in Houston and Pulaski counties eastward at least to the Savannah River. Its character in the east is known from exposures in northern Jenkins County and from Screven County drill cores. Its southern extent is poorly known. The type locality of the Ocmulgee Limestone is at Taylor's Bluff, three miles north of Hawkinsville in Pulaski County (Figure 1). The Ocmulgee Limestone's lithology varies with depth (Figure 2). In some places the limestone near the upper surface of the formation is more argillaceous and much softer than that of the lower section, possibly because of weathering or because that part of the limestone was never well consolidated. The lower section of the limestone is more compact and contains a higher abundance of sand-sized grains and glaucony grains (Huddlestun and Hetrick, 1986).

The Ocmulgee Limestone is highly fossiliferous. The presence of *Chlamys cocoana* (a scallop), was used by Pickering (1970) to suggest an Early Oligocene age for the formation. He noted that *Flabellum cuniformae* (a coral) is found at all outcrops of the formation that are

rather pure calcarenite. Although *F. cuniformae* had been often cited as a Late Eocene guide fossil, Pickering wrote that it occurs throughout the Upper Eocene-Lower Oligocene of the Coastal Plain wherever clean calcarenite occurs. Huddlestun and Hetrick (1986) indicated that *Chlamys cocoana* is no longer thought to be restricted to early Oligocene and placed the Ocmulgee Formation in the uppermost part (upper Jacksonian Stage) of the Eocene epoch. Bryozoans are found throughout the formation. Among these *Ochetosella jacksonica* was used to place the Ocmulgee Limestone in the Late Eocene epoch (Canu and Bassler, 1920; Wortman and others, 2004). The echinoids *Periarchus quinquefarius*, *Paraster americanus,* and *Brissopsis blanpiedi* and the foraminifer *Globigerina sp.* are also present.

In Pulaski County, the Ocmulgee Limestone is overlain by the Marianna Limestone of the Vicksburg Group and is underlain by the Twiggs Clay Member of the late Eocene Dry Branch Formation (Huddlestun and Hetrick, 1986; Huddlestun, 1993). To the east, the Eocene Sandersville Limestone Member of the upper Eocene Tobacco Road Sand has been correlated to the Ocmulgee Limestone based on the presence of the echinoid *Periarchus quinquefarius* in both units (Pickering, 1970; Huddlestun and Hetrick, 1986; Suurmeyer and others, 2003).

METHODS

The original sample was collected at the type locality from an interval 20-22 feet above the base of the limestone (*i.e.*, above the Twigg's Clay) in 2003 (Figure 2). Glaucony grains were separated magnetically from the insoluble residues (primarily quartz) produced by reacting the limestone with 10% hydrochloric acid. The sample was further purified by hand-sorting under a stereomicroscope (40× magnification) using a paintbrush dampened with de-ionized water. Dark grayish green and greenish black botryoidal grains that ranged in size from 0.15 mm to 1.0 mm were picked for analysis in this study. The selected grains were washed on a 0.15 mm sieve to remove contaminants too small to maneuver with a paintbrush or probe.

A small amount of the glaucony grains (~20 mg) was powdered in an agate mortar and pestle. An oriented mount of the powdered sample was scanned in a Philips Norelco X-ray diffractometer equipped with a Bragg-Brentano geometry diffractometer and the MDI Databox to run the scans and plot the diffraction data. The sample was solvated in ethylene glycol vapor for 24 hours and re-scanned to determine the presence of expandable minerals. The X-ray diffraction patterns were interpreted from diffraction data of Moore and Reynolds (1997). Throughout the course of this work, representative glaucony grains were set aside to be examined with a LEO 1450 scanning electron microscope (SEM) at the Georgia State University's Department of Biology.

Grains weighing 30.1 mg in total were selected for K-Ar work. These were crushed in a small mortar under distilled water. The resulting powder was treated for 40 minutes with 0.1 M hydrochloric acid and then separated from the liquid by centrifuging and decantation. The powder was washed twice with ethanol (decanted away after centrifuging) and finally dried at 50ºC.

The dried glauconite powder was weighed into a copper-foil capsule, which was then closed by folding. The folded capsule and a small fused-quartz test tube were placed inside a horizontal glass tube that included a section of fused-quartz glass. The horizontal tube, part of a vacuum line for argon extraction, was then sealed by glassblowing. After overnight evacuation, the central part of the fused-quartz section of the horizontal tube, where the sample was to be heated, was pre-heated for twenty minutes with a small external electrical resistance heater to a temperature near 1100ºC. The fused-quartz test tube was then moved into the pre-heated area and heated by the protocol used later for heating the glauconite. This heating of the empty test tube yielded a small amount (0.25 pmol) of argon having the isotopic composition of atmospheric argon. The copper-foil capsule containing the glauconite was then moved into the fused-quartz test tube and placed at its closed end, and the external heater

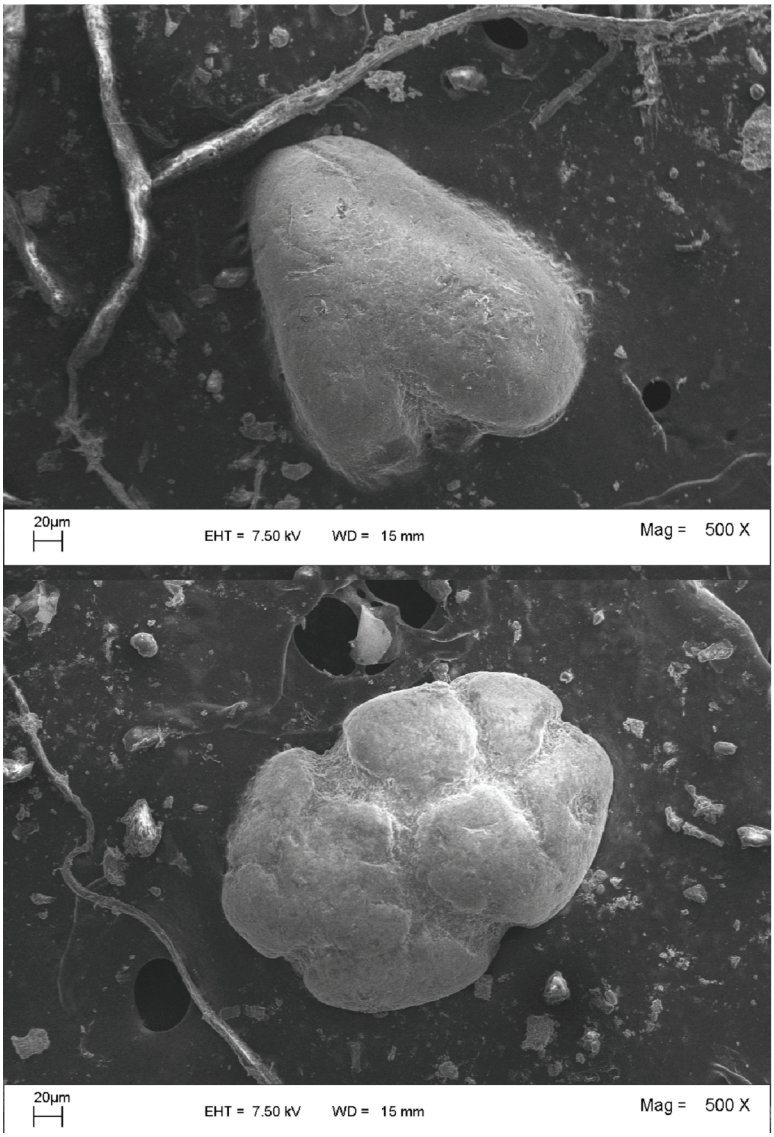

Figure 3: SEM images of representative grains separated by hand for K-Ar measurements.

was centered over the capsule. Power was brought up gradually over 15 minutes and held constant for an additional 7 minutes at a value expected to hold the capsule at a temperature between 1000ºC and 1050ºC. The actual temperature of the capsule exceeded 1050ºC and the copper melted, but the heated (partially melted) powder was retained in the closed end of the fused-quartz test tube. Any potassium evaporated during heating would have condensed in a cooler portion of the fused-quartz test tube that extended beyond the heater. The gases released by heating the glauconite were mixed with a known amount of ^{38}Ar. After less-volatile gases had been removed from the mixture in cold traps, heated titanium in two stages was used to remove reactive gases. The isotopic composition of the argon (a mixture of argon from the sample and the added ^{38}Ar) was measured with an AEI Model MS-10 mass spectrometer attached to the argon extraction line.

The test tube containing the residual solid

was then removed from the argon extraction line and its contents (copper, the silicate residue of the glaucony, and any potassium that may have evaporated from the silicate residue) were transferred with a 10:1 mixture of hydrofluoric acid and perchloric acid into a fluorocarbon (FEP) container. Nitric acid was added to fully dissolve the copper, and the FEP container was closed and heated gently overnight to digest the silicate residue. Then the FEP container was opened and heated more strongly to drive off excess acid and SiF_4. The residual solids were taken up in a measured amount of an acidic (0.1 mol/kg nitric acid) cesium chloride (0.01 mol/kg) solution. After further dilution with the same solution, the mass fraction of potassium in the solution was determined in reference to standard potassium solutions by flame atomic absorption spectrophotometry (FAAS) with a Perkin Elmer Model 3100 spectrophotometer.

Because the silicate residue had reacted slightly with the test tube, the inside of the tube was washed (etched) several times with the hydrofluoric-perchloric acid mixture to extract potassium that had diffused into the fused-quartz glass. During each wash, the hydrofluoric acid was allowed to dissolve silica from the inner walls of the tube for about 15 minutes. Each wash solution was transferred to its own FEP container and evaporated to dryness. The residues were taken up in measured small amounts of the acidic CsCl solution for potassium determination by FAAS. Small but significant amounts of potassium were found in the first two wash solutions (8 µg and 7 µg, respectively, out of 1010 µg total for the glaucony). The third wash solution had virtually no potassium (1 µg), so no further washes were done.

Subsequent work (to be published elsewhere) with other glaucony samples, including the interlaboratory reference sample GL-O, has confirmed that >99.8% of the argon in well-evolved glaucony is extracted when it is held for 10 minutes between 1000°C and 1050°C and that >99.95% of the potassium remains within the copper-foil capsule (which does not melt in that temperature range).

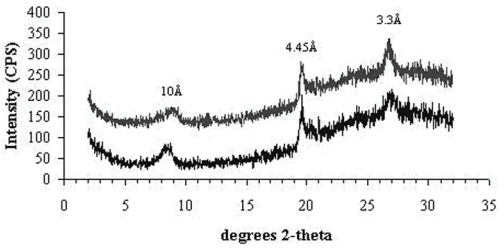

Figure 4: Diffraction patterns of the powdered glaucony. The upper pattern shows the diffraction pattern after solvation with ethylene glycol vapor. The lower pattern shows the diffraction pattern after drying in air. The presence of glauconite is based on the presence of the peaks at 10 Å and at 3.3 Å and the absence of a peak at 5 Å (17.7° 2θ).

RESULTS

Glaucony grains usually range in color from yellow-green to dark green, however, the grains selected for K-Ar measurements and mineralogic characterization were the dark green variety, 2.5/2 5G, 3/2 5G, and 2.5/1 5G on the Munsell Color Chart. These grains were dark grayish green and greenish black and were botryoidal in habit (Figure 3). The grains ranged from 0.15 mm to 1.0 mm in diameter along the longest axis. Before crushing and reaction with 0.1 M HCl, the glaucony grains selected for the K-Ar work had a mass of 30.1 mg. Afterward, the mass of the remaining powder was 18.1 mg. More of the dark green grains described above, about 20 mg, were crushed and analyzed by X-ray diffraction. Glauconite was the primary mineral observed from X-ray diffractometry (XRD) analyses. The XRD scans of both air-dried and glycol-solvated oriented mounts show the presence of a broad 10 Å peak and a more intense 3.3 Å peak and the absence of a 5 Å peak (Figure 4). The peak at 4.45 Å near 20º 2θ is a characteristic *hkl* reflection for phyllosilicates. The background intensity increases from 20º 2θ to the end of the scan at 32º 2θ due to scatter from the glass slide. The age calculated from measurements of potassium and argon isotopes in the glauconite is 33.7 ± 1.0 Ma (Table

Table 1: Results of Potassium-Argon Measurements of Glauconitic Grains from the Ocmulgee Limestone.

Sample Mass (mg)	K (µg)	K (% by mass)	$^{40}Ar^*$ (% of ^{40}Ar)	$^{40}Ar^*$ (nmol kg^{-1})	K-Ar Age (Ma)
18.09	1010†	5.58† ± 0.06‡	73	325 ± 9‡	33.7 ± 1.0‡

* The symbol $^{40}Ar^*$ stands for radiogenic argon.

† Includes 16 µg of potassium etched from the fused-quartz test tube in three washes.

‡ Uncertainty ranges are based on estimates of the analytical error at the 95% confidence level (2σ).

1). The amount of potassium used in calculating the age includes 16 µg of potassium from the three washes (etches) of the fused-quartz test tube, since that potassium is assumed to have entered the glass by reaction of the glaucony residue with the glass. The 1.0 Ma uncertainty is an estimate of the effect of analytical error on the age value at the 95% confidence level (2σ). The argon isotopic composition shows a high percentage of radiogenic argon ($^{40}Ar^*$), 73%. The K content of the glauconite powder was 5.58 weight percent (6.72 wt.% K_2O).

INTERPRETATION AND DISCUSSION

The presence of 10 Å diffraction peaks and the absence of 5 Å diffraction peaks on XRD scans of both air-dried and glycol-solvated glaucony powder indicate the presence of glauconite within the glaucony grains separated from the Ocmulgee Limestone. The increase in background intensity between 20° and 32° 2θ is interpreted to represent scatter of X-rays from the glass slide and/or the presence of amorphous matter within the grains. In aggregate, grains composed of glauconite can be considered to be "evolved" glaucony. Given that the time required for evolution of glaucony is typically 10^4-10^5 years (Odin and Matter, 1981), the 33.7 ± 1.0 Ma age value of the glaucony grains is interpreted to date the deposition of the upper portion of the Ocmulgee Limestone collected in this study. This interpretation assumes that these glaucony grains are authigenic rather than epiclastic. As shown in Figure 5, the numerical age range 32.7 Ma to 34.7 Ma for the glaucony grains is consistent with the latest Eocene (Jacksonian) age for the Ocmulgee Limestone proposed by Huddlestun and Hetrick (1986) and also with the early Oligocene biostratigraphic age proposed by Pickering (1970). Insofar as the analytical errors are random, the most probable value within that range is the central value 33.7 Ma, which is close to the currently accepted date of 33.9 ± 0.1 Ma for the Eocene-Oligocene transition (Luterbacher and others, 2004). Additional K-Ar analyses of glaucony grains are needed from throughout the Ocmulgee Limestone to refine further the range of depositional ages of the Ocmulgee Limestone in central Georgia.

The K-Ar age for the glaucony grains from the Ocmulgee Limestone is in accord with other radiometric ages from rocks in the region. The central value, 33.7 Ma, is 0.6 Ma less than that for the K-Ar age of dark green glaucony grains (34.3 ± 0.4 Ma) from the underlying Twiggs Clay Member of the Dry Branch Formation reported by Albin and Wampler (1996). The difference is not statistically significant, but it is large enough to suggest that a numerical age difference consistent with the difference in stratigraphic position could likely be confirmed with more precise measurements. The K-Ar age obtained in this study is also consistent with the age inferred for a thin sand layer at the base of the Twiggs Clay Member that contains shocked quartz grains thought to have been ejected from the Chesapeake Bay impact event (35.7-36.0 Ma, Harris and others, 2004).

This work also shows that it is possible to obtain an accurate K-Ar age using the methods described herein. Despite the small sample mass of 18 mg, the amounts of radiogenic argon and potassium in that sample were sufficient for accurate measurements. The error for the K-Ar age is lessened by use of one weighing (Dalrymple and Lanphere, 1969), because sample inhomogeneity does not contribute to error in

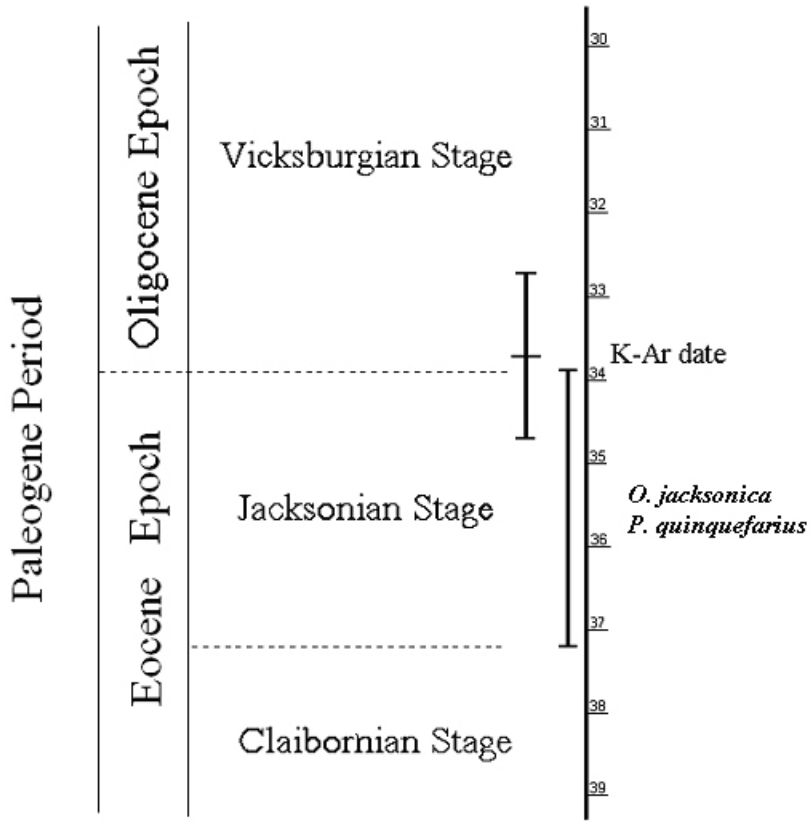

Figure 5: Geologic time scale showing numerical ages of relevant stages of the Eocene and Oligocene epochs, the known ranges of *O. jacksonica* and *P. quinquefarius* (spanning the Jacksonian Stage), and the K-Ar age (33.7 ± 1.0 Ma) of glauconite from the Ocmulgee Limestone.

the age value, nor does any error in weighing. The method described herein makes possible the determination of K-Ar age values where only small amounts of glauconite are available.

CONCLUSIONS

Dark green grains found in the Ocmulgee Limestone are identified as being composed of glauconite, and this glauconite contains sufficient potassium and radiogenic argon for good K-Ar measurements. The external appearance and the potassium content of the grains are those of evolved glaucony. The material remaining after argon extraction was used for the potassium measurement, a method that requires only a small amount of sample and provides a K-Ar age value unaffected by sampling error and weighing error. These results indicate that the interval of the Ocmulgee Limestone studied herein was deposited 33.7 ± 1.0 million years ago. This K-Ar age places the depositional age of the layers studied near the Eocene-Oligocene boundary of 33.9 ± 0.1 Ma (Luterbacher and others, 2004). This age is also consistent with a recently determined K-Ar age value for dark green glaucony from the underlying Twiggs Clay Member of the Dry Branch Formation. This result raises a question regarding the range of depositional ages represented by the Ocmulgee Limestone and its characteristic fossil assemblages (e.g. *Ochetosella jacksonica*) in the Georgia Coastal Plain.

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