COLOR, MINERALOGY AND COMPOSITION
OF UPPER JURASSIC WEST SIBERIAN GLAUCONITE:
USEFUL INDICATORS OF PALEOENVIRONMENT

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ABSTRACT

Changes in color of Upper Jurassic glauconite of the Georgiev Formation, in the West Siberian Basin, in Russia, are related to
changes in physicochemical conditions that caused glauconite maturation and alteration, driven by regional paleoenvironmental
evolution. Maturation produces dark green (bluish) glauconite formed from Fe-rich smectite by increasing the content of Fe$^{2+}$
together with K. Alteration produces brown rims and cracks that are enriched in Al and depleted in Fe and K with respect to the
glaucite cores. The change from a yellowish green to dark green color in progressively more mature glauconite is explained by
light absorption induced by enrichment in octahedrally coordinated Fe$^{2+}$ relative to the total Fe, associated with the progressive
decrease in the proportion of Fe$^{3+}$-rich smectite interleaved with glauconite. The brown color in alteration rims is due essentially
to light scattered by nanometric inclusions of Fe oxyhydroxides. These, together with residual Al-rich glauconite and a subor-
dinate Fe-rich smectite, constitute reaction products formed by leaching of K and Fe$^{2+}$, and by the oxidation of yellowish green
glaucite cores. Berthierine formed later in the brown rims on Al-rich glauconite, and pyrite formed as a result of drowning
of the platform during the latest Jurassic – earliest Cretaceous and sedimentation of black shales under increasingly reducing
conditions.

Keywords: glauconite, color, composition, maturation, alteration, Georgiev Formation, West Siberian Basin, Russia.

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Les changements en couleur de la glauconite de la Formation de Georgiev, d’âge jurassique supérieur, dans le bassin Sibérien Occidental, en Russie, sont liés aux changements des conditions physicochimiques qui ont provoqué la maturation et l’altération de la glauconite, conditions régies par l’évolution du milieu paléo-environnemental. La maturation a produit une couleur vert foncé (bleuté) de la glauconite formée aux dépens de la smectite ferriére par augmentation de la teneur en Fe\(^{2+}\) et en potassium. L’altération a produit une bordure brune et des craquelures qui sont enrichies en Al et appauvries en Fe et K par rapport aux noyaux de glauconite. Le changement de couleur, de vert jaunâtre à vert foncé dans la glauconite progressivement plus mature, serait dû à l’absorption de la lumière induite par enrichissement du Fe\(^{2+}\) en coordonante octaédrique par rapport au fer total, ainsi que la diminution progressive de la proportion de smectite riche en Fe\(^{3+}\) en intercalation avec la glauconite. La couleur brune des lisérés d’altération serait due surtout à la dispersion de la lumière par des inclusions nanométriques d’oxyhydroxides de fer. Ces particules, ainsi que la glauconite résiduelle riche en Al et une proportion subordonnée de smectite riche en fer, constituent les produits de réaction formés par lessivage de K et Fe\(^{2+}\), et par l’oxydation des noyaux de glauconite vert jaunâtre. La berthierine s’est formée plus tard sur les bordures brunes des grains de glauconite alumineuse, et la pyrite s’est formée lors d’une submersion de la plateforme à la fin du Jurassique et au début du Crétacé, et de la sédimentation de shales noirs qui s’en suivit sous conditions de plus en plus réductrices.

Mots-clés: glauconite, couleur, composition, maturation, altération, Formation de Georgiev, bassin Sibérien Occidental, Russie.

INTRODUCTION

Mineral color is a physical property controlled by a wide spectrum of mechanisms, from those studied by geometrical and physical optics to process accounting at the molecular to subatomic scale. Despite this complexity, color is a classical criterion for identifying and classifying minerals and, currently, is one of the features that is systematically observed and routinely catalogued in field and laboratory studies. Marine authigenic green clay minerals are characterized by their color and are usually divided into verdine and glaucony facies (Odin 1988). The use of facies terms (glaucony) instead of mineral names (glauconite) was proposed because many marine green grains, pellets, and crusts are actually not made of glauconite but of other green Fe-rich clay minerals (Odin & Matter 1981).

Textural and compositional observations of many grains of glaucony actually reveal that their mineralogy is very complex in detail, and that a variety of mineral phases occur within each glaucony grain, in addition to glauconite (Lamboy 1976, Odin 1988, Martín-Algarra & Sánchez-Navas 2000, Eder et al. 2007). Such heterogeneity certainly influences the color of glaucony grains, and is related to a complex pattern of sedimentological and chemical evolution before burial. For example, brown grains or brown rims on green grains are commonly found within marine greensands and associated facies, such as phosphorites (Glenn & Arthur 1990, Rao et al. 1993, Zamirailova et al. 1999, Martín-Algarra & Sánchez-Navas 1995, 2000). Commonly, such brown grains are synsedimentarily replaced by phosphate and ferruginous materials. Many brown grains, however, are not phosphatic, but rather are made of other Fe-rich clay minerals (Lamboy 1976, Odin & Lamboy 1988, Odin & Fröhlich 1988) in addition to true glauconite, which is in some cases rich in Al (Eder et al. 2007). Independently of its green or brown color, which has never been systematically evaluated together with associated chemical variations, the origin of Al-rich glauconite is debated, and the physico-chemical changes that have led to such Al enrichment have been interpreted as related to synsedimentary (Berg-Madsen 1983) or postsedimentary processes, the latter corresponding to weathering (Odin & Fullagar 1988), diagenesis (Longuépée & Cousineau 2006, and references therein) or anchimetamorphism (Guimaraes et al. 2000).

The maturation and alteration of Upper Jurassic Siberian glauconite have been studied by petrography, powder X-ray diffraction, several spectroscopic methods and analytical electron microscopy to establish the chemical and mineralogical composition and its relation to changes in optical properties such as color and index of refraction. Here, we discuss the changes in color in relation to Fe\(^{2+}/Fe^{3+}\) increase and K enrichment during glauconite maturation. We also review the microtextural, mineralogical and color changes associated with alteration, and the paleoenvironmental significance of these changes, which produced a brown glauconite rich in Al, among other by-products.

BACKGROUND INFORMATION

The mineral glauconite is referred to as Fe-rich glaucony with a K\(_2\)O content higher than 6% (Odin & Matter 1981, Odom 1984, Stille & Clauer 1994). The IMA Mica Nomenclature Committee has defined glauconite as a dioctahedral interlayer-deficient mica with composition K\(_0.8\)R\(^{3+}\)\(_{1.33}\)R\(^{2+}\)\(_{0.67}\)Al\(_{0.1}\)Si\(_{3.5}\)O\(_{10}\)(OH)\(_2\), with V\(^{I}\)V\(^{II}\)/(V\(^{I}\)Al+V\(^{II}\)Fe\(^{3+}\)) ≥ 0.15 and V\(^{I}\)Al/(V\(^{I}\)Al+V\(^{II}\)Fe\(^{3+}\)) ≤ 0.5 (Rieder et al. 1998). Color and texture in glaucony are related to maturation to glauconite through chemical and mineralogical changes involving increased K and
decreased smectite content (Amorosi 1995). Compositional trends of iron and potassium obtained from electron-microprobe data on glaucony grains have also been interpreted as being due to glauconite maturation (Martín-Algarra & Sánchez-Navas 2000). Other authors have interpreted such correlation as related to burial diagenesis leading to the formation of Al-rich glauconite through Al-for-Fe substitution and K loss (Longuépée & Cousineau 2006). The smectite content of glauconite, as observed by X-ray diffraction, is the traditional parameter used to establish the stage of glauconite maturation (Odin & Matter 1981).

**GEOLOGICAL SETTING**

The glauconite studied comes from Upper Jurassic condensed marine sediments (Georgiev Formation: Shurygin et al. 2000) of the West Siberian Basin (Fig. 1). The marine Georgiev Formation lies on Callovian to middle Oxfordian continental and marine rocks (Vasugan Formation), contains Upper Jurassic pelagic fossils (Vyakhileva et al. 1990), and is covered by Tithonian and lower Berriasian black shales (Bazhenov Formation), which are the main oil source-rocks of the basin (Braduchan et al. 1986). The Georgiev Formation is made of limestones, dolostones, manganese carbonates, claystones, siltstones, sandstones and phosphate rocks (Beizel et al. 2002, Zanin et al. 2003). Sandstones occur predominantly in the lower part of the formation, where phosphate and glauconite concretions, grains, pellets and crusts are ubiquitous (Nesterov 1991, Zamirailova et al. 1999). Glauconite grains are most abundant in its lower part (Barabian Member, Late Oxfordian), where they form fossiliferous glauconitites or sandstones with glauconite pellets and ichnofossils (Eder et al. 2003).

Marine transgressions flooded this large epicontinental basin from the North through a strait connected with the open Boreal Ocean (Fig. 1). According to the type of Upper Jurassic sedimentation, three paleogeographically different areas are distinguished in one north–south section of the West Siberian Basin (WSB): a continental area in the outermost southern part, a transitional area in the south-central part, and a marine region in the remaining central and northern areas (Zakharov et al. 1983). In east–west paleogeographical sections, shallow marine condensed sediments close to the Urals graded laterally to the deepest marine environments. They developed in the central and northern parts of the basin, which evolved toward the east to thick prograding shallow marine and continental terrigenous depositional systems (Vyssotski et al. 2006).

**MATERIAL AND METHODS**

The distribution of glauconite and associated sediments within the Georgiev Formation in different sectors of the basin is related to distinct environments of mineral formation controlled by paleogeography (Eder et al. 2007). Two samples were selected for color study from two cores drilled away from each other in the central (M–11–22: –2666.5 m deep) and northeastern (YAR–10, –1302.5 m deep) areas of the WSB (Fig. 1). Maximum diagenetic temperatures were probably less than 120°C in both cases (Eder 2004). These samples, previously studied by Eder et al. (2007) together with other types of glauconite in the WSB, were selected for this study of color because one contains mature glauconite grains with a bluish dark green color (M–11–22), whereas the other sample (YAR–10) contains texturally complex grains showing a core made of glauconite of yellowish green color (slightly less mature than that of sample M–11–22) and a brown rim formed of glauconite poorer in K and in Fe, and richer in Al, which Eder et al. (2007) interpreted as related to alteration.

Isolated grains of glauconite were extracted from the poorly consolidated whole rock by heavy-liquid separation, and separated, purified, concentrated, and divided into 8–12 mineral fractions by standard roller and isodynamic electromagnetic SIM–1 and electromagnetic universal UCM–1 separators. The most typical (densest) fraction of each sample was used for X-ray diffraction and color studies. In addition, polished thin sections were obtained from the most cemented parts of each core for textural and compositional studies. No major textural or compositional differences were noticed between grains from concentrates and thin sections from these or from any other samples of WSB glauconite (see Eder et al. 2007, for details).

Powder X-ray-diffraction diagrams (PXRD) from concentrates of grains of glauconite (reduced in size with an agate mortar) and oriented aggregates of the <2 μm fraction treated with ethylene glycol, were recorded using a Philips PW1710 diffractometer. A decomposition program (MACDEF) was applied to the low-angle region (5–11°2θ) of the X-ray diffraction pattern of ethylene glycol in order to investigate the superposition of glauconite and smectite mixed-layers on the 1 nm peak for glauconite (Petschick 2000). Peaks were fitted through a Pearson VII function. Various combinations of peaks were checked until we obtained the lowest residuum. Intensity, half-height width and peak position were approximated until they optimally retraced the peak course of the original profile. The fit was improved by the least-squares method.

Back-scattered electron (BSE) images and energy-dispersion X-ray (EDX) analyses of the surface of glauconite grains were made with a field-emission scanning electron microscope (FESEM) LEO GEMINI 1525. A CAMEBAX SX–50 automated electron-microprobe was used for quantitative chemical analysis (EMPA) at an acceleration voltage of 20 kV, a probe current 5 nA, and a beam diameter of 5 μm.

For transmission electron microscopy (TEM), study samples were ion-milled after extraction from selected areas in thin sections, coated with carbon and examined
with a Philips CM20 instrument operated at 200 kV and equipped with an EDX model EDAX system for micro-analysis.

Reflections with \(d\) values \(> 0.4\) nm were used for lattice images. Selected-area electron diffraction (SAED) patterns were acquired for glauconite and berthierine packets; no good SAED patterns were obtained for poorly crystalline smectite. The investigation of glauconite–smectite mixed-layers and smectite layers by TEM involves problems related to the origin of the contrast of lattice images. According to Guthrie & Veblen (1989), smectite and illite interlayers can produce different contrast and can be differentiated only in lattice images made with very well-defined electron beam (001) orientation and defocus conditions corresponding to 1000 Å overfocus in their Philips 420 instrument. In general, contrast differences between mica-like (fringes of dark contrast) and smectite-like (light-contrast fringes) layers are not observed. In some cases, open interlayer spaces produced by contraction due to dehydration in a vacuum may allow the identification of smectite-like layers. The absence or uncertainty of both characteristics cannot be interpreted in terms of the absence of smectite layers. In general, mica and smectite layers have the same appearance and cannot be differentiated because the smectite interlayers collapse in the TEM vacuum to the same 10 Å spacing as mica.

Analytical electron microscopy (AEM) allows quantitative micro-analyses in STEM mode using a beam 5 nm in diameter and a scanning area of \(100 \times 20\) nm. Diverse silicates and oxides were used as standards to obtain k-factors to correct EDX data by the thin-film method of Lorimer & Cliff (1976).

The same powder used for PXRD study was also used later for spectrometric study of color. The existence of \(\text{Fe}^{3+}\) and \(\text{Fe}^{2+}\) in the structure of silicate minerals is responsible for the occurrence of light absorption that affects their color (Rossman 1988). Diffuse reflectance spectra in the ultraviolet – visible – near infrared (UV–VIS–NIR) were recorded from powder samples at room temperature on a Varian Cary 5E UV–VIS–NIR spectrophotometer (200–2000 nm). Infrared (IR) absorption spectra of the samples were recorded with KBr slices on Nicolet–20SX FTIR spectrometer (4000–400 cm\(^{-1}\)). Chromophoric transition elements were studied by electron paramagnetic resonance (EPR). The EPR spectra were recorded using a Bruker ESP 300E ESR spectrometer operating at X-band frequencies. Ferric ion (\(d^5\) configuration) has been determined by this technique; it gives rise to a splitting of three Kramer’s doublets; \(|±5/2>,|±3/2>\) and \(|±1/2>\). These are separated by 4D and 2D, respectively, where D is known as the zero-field splitting parameter. These doublets split further in the presence of an external magnetic field. Owing to the simultaneous population of all three doublets, the \(\text{Fe}^{3+}\) ion gives the number of resonances throughout the X-band region (Rao & Subramanian 1985).

**RESULTS**

**Petrography**

Sample M–11–22 corresponds to a medium- to fine-grained quartz-rich pyritic – phosphatic – glauconitic silty sandstone with a clayey and calcareous matrix (Fig. 2A). Glauconite grains are subrounded to well rounded, with smooth surfaces, and dark green (bluish) colors, in some cases with a lighter green halo (Fig. 2A). Compaction cracks in some grains are filled by diagenetic calcite, quartz or pyrite (Figs. 2A, B), but either cracked or not, these grains have a homogeneous composition, as evidenced by their uniform relectivity (Fig. 2B), gray levels in BSE images (which mimics reflected-light optical images) and X-ray (EDX) profiles (Fig. 2C). Light green halos present in some grains (Fig. 2A) are due to thinner edges of the rounded grains. The intersection of the lower rounded surface of these grains with the plane surface of the slide (glass) produces the darker line that bounds the internal area of the grain, separating it from the lighter rim of lesser thickness.

Sample YAR–10 corresponds to a siltstone including glauconitic grains and aggregates, which usually have variable roundness, cerebrum shape and larger size than grains of sample M–11–22. The glauconitic grains in YAR–10 are systematically cracked and show increased relief (index of refraction) from a yellowish green core to the surrounding brown rim, in which two sets of fissures with lighter brown (older) and darker brown (younger) colors are visible (Fig. 3A). The lighter brown zones show sharp or diffuse contact-areas toward the yellowish green core (Fig. 3B). The darker brown zones show consistently sharp contact-areas either with lighter brown areas or directly with the green core (Fig. 3B).

Uniform gray levels in low-magnification BSE images of M–11–22 glauconite (Fig. 4A) indicate a homogeneous texture and composition, and this is confirmed by single-point EDX and EPMA analyses and profiles (Fig. 2C). Nevertheless, more detailed examination of BSE images reveals a much more complex picture. Many grains show a heterogeneous texture due to the presence of very small inclusions of other minerals (Fig. 5A), in particular pyrite, quartz, feldspars, micas,apatite and calcite, among others. Other grains, however, seem quite homogeneous (Fig. 5B). This contrast emphasizes the difficulty in interpreting the results of chemical analyses performed on

![Fig. 1. Location and simplified stratigraphy of the West Siberian Basin. The glauconite comes from the Georgiev Formation.](attachment:image.png)
glauconite concentrates by magnetic separation, where such small inclusions are impossible to detect. The EPMA results for the sample M–11–22 presented in Table 1 come from carefully selected very clean areas without any inclusion, such as that shown in the grain of Figure 5B.

Complex relationships between texture and composition are clearly shown in BSE images of glauconite in sample YAR–10: the cores are lighter than the rims (Fig. 4B), grains are quite commonly cracked (Figs. 5C, E), and two sets of darker and lighter gray fissure-fillings are visible (Fig. 5E). These correspond to lighter and darker brown colors in optical images, respectively (compare Figs. 3A–B with Figs. 4B and 5C–F). Gradual and diffuse changes from light gray (= yellowish green) cores to darker gray (= light brown) rims are identified, and there are also sharp contacts between lighter cores and a first set of fissures filled with material identical to that of the rims (Fig. 5E, arrow). The second set of fissures is, in some cases, included within the former (Fig. 5C), and is filled by a completely different, denser, Fe-rich material that also includes Fe-poor clay peloids detached from the darker rims (Fig. 5D). The border of the glauconite grains in these fissures is marked by a narrow rim of Fe-poor clays with a similar texture to that of peloids included within the Fe-rich fissures (Fig. 5F). Pyrite framboids are present within the siliciclastic matrix (Figs. 4B, 5C).

**Glaucnite–smectite mixed-layer**

PXRD diagrams of separated fractions of the samples studied are consistent with previously reported data on glauconite. Reflections around 10 Å show slightly higher spacing than expected according to the higher-order (00l) basal peaks (Fig. 6). When samples are ethylene-glycol-saturated, this first basal reflection splits into various peaks. The most intense one shows spacing completely consistent with the rest of the (00l) peaks, producing a rational series of orders corresponding to 10 Å spacing (Fig. 6, left, finer green line). The less intense peaks are in the 11–13 Å zone (Fig. 6, right) and, according to this spacing and its ethylene-glycol-saturated behavior, may be interpreted as a glauconite–smectite ordered mixed-layer, composed predominantly of mica-type layers, but showing slightly different proportions of smectite layers. Therefore, the

![Fig. 2. Optical microphotographs of Siberian glauconite (polished thin sections, plane-polarized and transmitted light (A) and reflected light (B). (A) Glauconite grains of sample M–11–22 cracked by compaction and surrounded by mixed terrigenous clay as well as fine-grained carbonate, plus quartz grains. The black area within the grain at the bottom of the image corresponds to pyrite (py). (B) Reflected-light image of (A). Lighter grains of terrigenous quartz, terrigenous clays (darker areas), micritic carbonate areas (lighter zones of the upper left corner), and compaction cracks within the largest grain are more evident than in the transmitted light image. (C) As is clear from the reflected-light image, the BSE image and the Si, Al, Fe, K and Ca X-ray profiles (dashed line) of the largest grain of glauconite demonstrate that no compositional difference exists from core to rim, and that the light green halos surrounded by a darker line observed in the transmitted light image are optical artifacts produced by the thinner border of the grain and its contact with the underlying glass.](image-url)
Fig. 3. Optical microphotographs of sample YAR–10 (polished thin sections, plane-polarized and transmitted light). (A) Yellowish green cores surrounded by cracked brown rims with lighter (1: older) and darker (2: younger) brown color. (B) Contacts between yellowish green cores and brown rims are in some cases sharp (arrows).
Fig. 4. General views (BSE images) of the glauconite grains and enclosing sediment. (A) Sample M–11–22 is formed by subrounded grains of mature glauconite (g), with a relatively homogeneous texture and composition (cf. Fig. 2C), included within a sandstone matrix that is made of quartz (q) feldspars (f) and detrital illitic clays (c), and contains abundant grains of diagenetic framboidal pyrite (py). (B) Sample YAR–10 mainly consists in this view of a densely packed aggregate of grains of glauconite surrounded by a matrix of fine-grained siliciclastic sediment (s) made of angular silt-sized quartz grains and clay minerals, with some diagenetic framboidal pyrite (py); these glauconite grains show denser light gray cores and darker gray rims, as well as a complex set of fractures. The square areas in both images correspond to the location of insets reproduced in the Figures 5B, 5C and 5E.
Fig. 5. (A) BSE image of glauconite grain including very small quartz grains surrounded by a thin rim of pyrite (sample M–11–22). (B) BSE image of the contact between one homogeneous grain of glauconite (g) with an internal flaky texture and its surrounding terrigenous matrix, including pyrite frambooids (py). Sample M–11–22, see location of this image in Figure 4A. (C) BSE image of sample YAR–10 with light gray glauconite cores (c) and dark gray rims (r); other gray levels correspond to siliciclastic quartz (q), feldspars and terrigenous clays, and to framboidal pyrite (py); the fissures are filled with two different substances, with darker and lighter gray levels; the lighter gray, denser fissure-filling is richer in Fe than rims and than cores. See location of this image in Figure 4B. (D). Inset of the square area in (C), where peloids detached from the darker gray rims are surrounded by Fe oxyhydroxides. (E) Gradual to sharp (arrow) contacts between lighter gray cores (c) and darker gray rims (r); some siliciclastic grains are visible in the matrix (darkest areas correspond to quartz); the fissures filled with lighter and denser material cross-cut both the lighter gray cores and the darker gray rims. See location of this image in Figure 4B. (F) Close-up of the square area in (E); the light gray fissure filling is Fe-rich, but includes dark peloids of Fe-poor clays and Fe-poor rims (arrows) just at the borderline of the fissure cross-cutting both the core and the rim of the glauconite grain.
Fig. 6. Powder X-ray diagrams of air-dried and ethylene-glycol-treated samples of glauconite, with indication of d value. In sample YAR–10, the arrows point to the peaks at 7.09 Å and 3.55 Å, which are absent in sample M–11–22. Deconvoluted peaks at 10 Å of ethylene-glycol-treated samples are shown in the right-hand diagrams. The theoretical curves (thicker line) resulting from the sum of the small peaks (fine lines) show excellent agreement with the experimental diagrams (residuals between 2–3%). In the deconvoluted curves, the most intense component peak on the right corresponds to mica-type layers, and the various smaller component peaks on the left are interpreted as glauconite–smectite mixed-layers.
samples studied are predominantly made of glauconite containing some minor layers of smectite, which is in accordance with EPMA data (K2O content below 8.5%). Unfortunately, only the peaks in the 11–13 Å zone can be recognized, owing to the low proportion of mixed-layer and the high proportion of mica layers, which produces a spacing of the mixed-layer very similar to that of discrete glauconite.

The glauconite in sample YAR–10, however, contains more smectite layers than in sample M–11–22, as evidenced by both the position and relative intensity of the most significant mixed-layer peaks in the deconvoluted ~10 Å peak of ethylene-glycol-treated samples (Fig. 6, right). It also contains another phase, as indicated by a peak at 7.09 Å, which is absent in sample M–11–22. The exact position of the peak was determined adding quartz to the sample as an internal standard to correct the possible error in spacing due both to sample preparation (small differences in height outside the Rowland circle) and the specific diffractometer used. In this way, we can assure accuracy in the measurement of around 0.01 Å. This procedure allowed us to rule out the possible presence of kaolinite (d_001 in the range 7.15–7.17 Å). The second-order peak at 3.55 Å and the absence of possible odd reflections corresponding to 14.2 Å demonstrate that they correspond to a rational series of orders at 7.1 Å, which is perfectly consistent with berthierine. Smectite appears in the PXRD diagrams only as minor interstratified layers, but it is absent as a discrete mineral. No other mineral phases are observed in the PXRD diagrams of glauconite concentrates.

**Mineral compositions**

The EPMA study demonstrates that the composition of different grains and parts of each glauconite grain of sample M–11–22 is quite homogeneous (Table 1, Fig. 7). The content of the heavier atoms (K and Fe) in the darker gray (light brown) rim of sample YAR–10 is lower than in the corresponding lighter gray (yellowish green) core. Moreover, the light brown areas in the rim are not only clearly depleted in K and Fe, but also strongly enriched in Al relative to the green core of the same sample, and to the green grains of sample M–11–22 (Table 1; see also K, Fe and Al X-ray maps in Fig. 10 of Eder et al. 2007). The EPMA data also indicate that bluish green glauconite of sample M–11–22 has a higher K, Mg, and Al content, as well as a lower Fe content, than the yellowish green cores of sample YAR–10 (Table 1, Fig. 7). In addition, both samples show a positive correlation between K2O and Fe2O3 and a negative correlation between VIAl and Fe (total Fe expressed as Fe3+). Structural formulae of the glauconite (Table 1) indicate that VIAl is the same in cores and rims of sample YAR–10, and higher than in grains.

**TABLE 1a. SELECTED RESULTS OF ELECTRON-MICROPROBE ANALYSES OF GLAUCONITE IN SAMPLE YAR–10**

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Total   | 92.52 | 92.81 | 93.13 | 92.67 | 92.91 | 94.44 | 94.76 | 94.71 | 94.00 | 94.36 | 93.96 | 95.80 | 92.51 | 95.37 |

Si (apfu) | 3.55 | 3.57 | 3.54 | 3.57 | 3.56 | 3.56 | 3.57 | 3.57 | 3.56 | 3.56 | 3.53 | 3.56 | 3.55 | 3.55 |

Ti      | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.01 | 0.01 | 0.01 | 0.01 |

VIAl   | 0.45 | 0.43 | 0.46 | 0.43 | 0.45 | 0.43 | 0.44 | 0.44 | 0.43 | 0.44 | 0.44 | 0.44 | 0.44 | 0.45 |

Fe     | 1.52 | 1.50 | 1.50 | 1.58 | 1.58 | 1.64 | 1.62 | 1.59 | 1.60 | 1.53 | 1.30 | 1.30 | 1.33 | 1.33 |

Mg     | 0.24 | 0.24 | 0.25 | 0.25 | 0.24 | 0.24 | 0.24 | 0.24 | 0.24 | 0.24 | 0.24 | 0.24 | 0.22 | 0.22 |

VIAl   | 0.21 | 0.22 | 0.22 | 0.15 | 0.16 | 0.09 | 0.12 | 0.15 | 0.13 | 0.20 | 0.45 | 0.56 | 0.45 | 0.41 |

ΣVIAl  | 1.97 | 1.96 | 1.97 | 1.97 | 1.97 | 1.98 | 1.98 | 1.97 | 1.96 | 1.99 | 1.99 | 1.99 | 1.99 | 2.01 |

K      | 0.69 | 0.70 | 0.68 | 0.68 | 0.70 | 0.70 | 0.67 | 0.67 | 0.68 | 0.70 | 0.60 | 0.57 | 0.60 | 0.61 |

Ca     | 0.03 | 0.03 | 0.03 | 0.02 | 0.02 | 0.02 | 0.02 | 0.02 | 0.02 | 0.01 | 0.02 | 0.02 | 0.02 | 0.02 |

ΣVIAl  | 0.73 | 0.73 | 0.72 | 0.71 | 0.74 | 0.73 | 0.70 | 0.70 | 0.71 | 0.73 | 0.65 | 0.62 | 0.66 | 0.66 |

GC: green core, Br: brown rim. The compositions, expressed in wt%, are recalculated in terms of atoms per formula unit (apfu) on the basis of 22 charges. *Total Fe expressed as Fe3+. M: octahedrally coordinated cations, A: interlayer cations.
of sample M–11–22, and that Fe$^{3+}$ variations are essentially balanced by octahedrally coordinated Al through incorporation of the ferriceladonitic component. Transmission electron images of the yellowish green cores of the sample YAR–10 (Fig. 8A) show the typical lamellar arrangement of glauconite packets. Diffraction patterns (Fig. 8A, inset) indicate that the stacking of packets is subparallel. Lattice images (Fig. 8B) show the existence of a $d = 1$ nm spacing typical of mica and contracted-smectite layers (layer type 2:1). A representative chemical formula obtained from AEM analyses of YAR–10 glauconite cores (Table 2; all Fe expressed as Fe$^{3+}$) is: $K_{0.7}(Fe_{1.7}Mg_{0.3}Al_{0.1})Si_{3.4}Al_{0.6}O_{10}(OH)_2$.

The TEM study performed on the brown rim (Fig. 9A) indicates the presence of glauconite with a chemical composition slightly different from that of the yellowish green glauconite cores (diamonds) and light brown rims (triangles) of sample YAR–10.

![Graph](image)

**Fig. 7.** $K_2O$ versus $Fe_2O_3$ (A) and $VI Al$–$Fe$ (B) bivariate plots for the two samples of glauconite studied. Gray diamonds correspond to dark green grains in sample M–11–22, whereas black symbols correspond to the composition of yellowish green glauconite cores (diamonds) and light brown rims (triangles) of sample YAR–10.
Fig. 8. TEM images of one yellowish green glauconite core (sample YAR–10): (A) Sub-parallel stacking of glauconite packets. Diffraction pattern in the lower left side of the image. (B) Lattice image of one glauconite packet showing 1 nm d value of mica and contracted-smectite layers.
goethite or hematite (Fig. 9C), and minor amounts of smectite, including Fe-oxyhydroxides (Fig. 9A). The formula derived from results of AEM analyses of the glauconite in the rim is: K₀.₇(Fe₁.₄Mg₀.₃Al₀.₃)Si₃Al₀.₆O₁₀(OH)₂.

Berthierine form relatively well-defined packets showing parallel layer with a d value of 0.7 (Fig. 9B), which corresponds to the peak at 7.09 Å detected by PXRD (Fig. 6). The d = 1.4 nm value observed (Fig. 9B) corresponds to a two-layer polytype of berthierine. Its chemical formula, obtained by AEM (all Fe expressed as Fe²⁺), is also perfectly coherent with the chemical composition of berthierine: (Fe₃.₇₂Mg₀.₆₃Al₁.₃₇)Si₃.₁₉Al₁.₃O₁₀(OH)₈.

The presence of smectite mixed with iron oxyhydroxides (Fig. 9A) is also made evident by the low Si content and by the sum of octahedrally coordinated cations in most of the cases. Its chemical formula, normalized to 10 atoms of oxygen and 2 OH (Fe expressed as Fe³⁺) is: K₀.₂(Fe₂Mg₀.₄Al₀.₁)Si₂.₇Al₁.₃O₁₀(OH)₂.

**UV–VIS–NIR and IR spectroscopy**

Optical absorption UV–VIS–NIR spectra of powdered samples of glauconite concentrates are shown in Figure 10. Their most significant feature comes from Fe²⁺, which produces a typical absorption band due to a dd transition around 9260 cm⁻¹ (1080 nm). Another prominent band at 770 nm (~13000 cm⁻¹), observed in both samples, is due to Fe²⁺ → Fe³⁺ electron transfer. This metal–metal charge-transfer (CT) band has been observed at 720 nm in trioctahedral micas by Robbins & Strens (1972). A decrease of both metal–metal CT and Fe²⁺ dd absorption bands, and a differential increase in the intensity of the metal–metal CT band in relation to the Fe²⁺ dd band, are noted from bluish green glauconite (M–11–22) to glauconite with a yellowish green core and a brown rim (YAR–10). The transmission window appears shifted toward higher wavelengths in the latter. The UV–VIS–NIR spectra also show the presence of an absorption band at 1900 nm in the NIR zone (Fig. 10), which is produced by bending motions of H₂O molecules. The existence of bending modes near 1620 cm⁻¹ in the IR spectra of both samples (Fig. 11) confirms the presence of H₂O molecules (Aines & Rossman 1984).

The IR spectra of both samples also indicate the existence of a prominent band at ~3400 cm⁻¹ corresponding to stretching modes of the OH⁻ essential to the structure of the glauconite and of other clay minerals (Fig. 11). Glauconite contains OH⁻ groups, but no molecular H₂O within its structure. If glauconite is the only hydrogen-bearing mineral within the samples studied, then only O–H stretching modes should be observed in the IR spectra, and the bending-related absorption bands should be absent. However, molecules of H₂O do occur within the two samples of glauconite studied, and must be associated with smectite layers included within the glauconite.

**Electron paramagnetic response (EPR)**

The EPR spectra of samples of powdered glauconite are shown in Figure 12. Both samples exhibit a broad resonance with a line width of ~1500 G, having g value close to 2.0 and also a weak signal at g ≈ 4.0. Another weak band at g = 2.787 appears only in sample YAR–10. These three resonance bands are due to the presence of Fe³⁺ (Vedanand et al. 1993) and are marked with an asterisk (*) in each spectrum (Fig. 12). The main resonance band in sample YAR–10 is a broad band at g = 1.927 with a line width of 1560 G, which is due to the dipolar–dipolar interactions of Fe³⁺ in glauconite, resulting from the presence of ferric iron in a high concentration. Two low-intensity resonances are visible in the low-field region at g = 2.787 and at 4.101, and both bands are attributed to the presence of Fe³⁺ as impurities (Reddy et al. 2002). Bluish green glauconite (M–11–22) also exhibits the main broad resonance at g = 1.939 and a weak signal in the low field at g = 4.093. The occurrence of this weak resonance at g ≈ 4.0 in the spectra of both samples is interpreted to be due to the presence of minor amounts of Fe³⁺-rich smectite in layers included in both samples of glauconite. The presence of a weak EPR signal at g = 2.787 in sample YAR–10, but not in the sample M–11–22 (Fig. 12) is attributed to the Fe³⁺ derived from Fe oxyhydroxide inclusions in the brown rims.

**DISCUSSION**

**Change in color associated with maturation**

A color change from pale green to light green and, finally, to dark green has been described from nascent to highly evolved glaucony grains during glauconite maturation [see Amorosi (1995), and references therein]. This sequence agrees with the observed color (Figs. 2, 3) and chemical composition (Table 1, Fig. 7) of the yellowish green cores of glauconite in sample YAR–10.
relative to the more mature dark green grains of sample M–11–22. The color of iron-bearing minerals is dependent on the occurrence of ferrous or ferric ions in the structure. Optical spectroscopy enables determination of the presence of Fe$^{2+}$ and Fe$^{3+}$ in minerals (Burns 1993). Both ions are octahedrally coordinated in the structure of glauconite. In minerals containing pairs of edge-sharing Fe$^{2+}$ octahedra, partial oxidation to Fe$^{3+}$ enhances the Fe$^{2+}$ dd transition band, and the advance of the oxidation process gives rise to the appearance of a Fe$^{2+}$–Fe$^{3+}$ charge transfer (CT) band (Amthauer & Rossman 1984, Rossman 1988). The increase of the intensity of the Fe$^{2+}$ dd transition and of Fe$^{2+}$–Fe$^{3+}$ CT band in the dark green glauconite (M–11–22) relative to the yellowish green to brown glauconite (YAR–10), and the decrease in the ratio of intensities $I_{CT}/I_{Fe^{2+}dd}$ (Fig. 10), are interpreted as due to a greater Fe$^{2+}$:Fe$^{3+}$ ratio in the occupancy of the layer of octahedra for dark green glauconite. Besides of the decrease in these two absorption bands at 770 and 1080 nm, the increase in the intensity of a ligand-to-metal (Fe$^{3+}$) CT band centered at the near-ultraviolet (Fig. 10) explains the observed shift of the transmission window toward lower wavelengths in the optical spectrum of dark green glauconite relative to that of yellowish green to brown glauconite. In brief, the increase in the ratio Fe$^{2+}$/Fe$^{3+}$ controls the change from yellowish green to blue green colors associated with glauconite maturation. This interpretation is also consistent with EPMA data (Table 1, Fig. 7). However, this technique does not allow a direct determination of Fe$^{2+}$/Fe$^{3+}$ value, but rather the consideration of real Fe$^{2+}$

**Fig. 9.** TEM images of brown rims of sample YAR–10. (A) Low magnification TEM image in which other phases are visible in brown rims, in addition to glauconite (Gla): berthierine (Be), smectite (Sm) and massive iron oxyhydroxides (M Fe–O). (B) Lattice image of berthierine showing 0.7–1.4 nm d value, with diffraction pattern in the upper right side of the image B. (C) Iron oxyhydroxides with massive (M Fe–O) and lamellar habits (L Fe–O).

**Fig. 10.** UV–VIS–NIR spectra of the two glauconite samples, with an indication of the absorption bands observed.

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as theoretical Fe$^{3+}$ produces the effect of an artificial decrease of the sum of octahedrally coordinated cations ($\Sigma^{VI}M$) in the phyllosilicate formula (e.g., Nieto 1997). In the glauconite samples studied, $\Sigma^{VI}M$ is slightly lower than two atoms per formula unit in dark green grains of sample M–11–22 and in yellowish green cores of sample YAR–10, but not in the brown rim. In the dark green grains, however, $\Sigma^{VI}M$ is lower than in the yellowish green cores (Table 1, Fig. 7). In addition, dark green grains show the highest interlayer occupancy, which is essentially monitored by K content.

The PXRD data of sample YAR–10 (Fig. 6) do not show the presence of iron oxyhydroxides or of smectite found by TEM in the brown rim (Fig. 9). The smectite determined by PXRD is present as individual mixed-layers and therefore corresponds to smectite precursors of glauconite and interleaved with it, rather than to the minor and discrete Fe-rich smectite packets found by TEM in the brown rim (Fig. 9A, Sm). The Al-rich glauconite of the brown rims has the same K content (Table 2), and, thus, the same amount of interleaved smectite as the yellowish green glauconite of the core. Bertherine of the brown rim is a Fe$^{2+}$ phase and, consequently, this phase increases the Fe$^{2+}$/Fe$^{3+}$ ratio. Nevertheless, the EPMA data on the brown rim (Table 1) show evidence of the highest values of $\Sigma^{VI}M$, which correspond to the lowest Fe$^{2+}$/Fe$^{3+}$ value. Thus, the contribution of bertherine to the color of sample YAR–10 is considered negligible.

The higher Fe content of the glauconite in the yellowish green cores of sample YAR–10 compared to the Fe content of glauconite in dark green grains of sample M–11–22 is balanced by the incorporation of Al and Mg into the glauconite structure in the latter sample, thus producing the chemical substitution of Fe$^{3+}$ for Mg$^{2+}$ plus K$^+$, together with the isovalent substitution Fe$^{2+}$$\rightarrow$ Al$^{3+}$ (Table 1, Fig. 7B). Nevertheless, the Fe$^{2+}$/Fe$^{3+}$ value in dark green grains is higher than in the yellowish green cores, as indicated by the optical spectroscopy study and by the $\Sigma^{VI}M$ (Table 1). Therefore, the difference in color [from yellowish green to bluish green, usually observed in cogenetic samples of glauconite: see Amorosi (1995), and references therein] is interpreted to result from a higher maturity of grains with a darker green color, which is related to the decrease in interleaved smectite layers. This change in the mineralogy of layers produces an overall substitution of Fe$^{3+}$ for Fe$^{2+}$ and an increase in K$^+$. In brief, glauconite maturation is not only monitored by the increase in K, as usually accepted, but also by the increase in Fe$^{2+}$/Fe$^{3+}$ which, moreover, controls the changes toward a darker green color traditionally associated with maturation. Potassium is stabilized at the interlayer by the Fe$^{2+}$ of the octahedral sheet, which explain how a mica-type structure, such as that of glauconite, compositionally close to the celadonite end-member, is stabilized at surface conditions.

**Change in color associated with alteration**

Optical (Fig. 3) and BSE images (Figs. 4B, 5C–F) indicate that rims and cracks in YAR–10 grains are
related to the alteration of the yellowish green glauconitic cores (Eder et al. 2007). The more detailed study performed here shows that alteration proceeded in two stages: the light brown rims and fissures (darker gray in BSE images) were produced first, and dark brown fissures later (the lightest gray fissure fillings in BSE images). The first alteration and fragmentation of yellowish green cores in glauconite produced another glauconite poorer in Fe and richer in Al (Tables 1, 2), but with the same K content (thus the same interleaved smectite content, i.e., the same maturity) as the glauconite of the cores, as demonstrated by AEM data (Table 2), together with other clay phases (probably smectite; Fig. 9A). All degrees of replacement of fresh cores by alteration phases have been observed in optical (Fig. 3) and in BSE images (Figs. 4B, 5C–F), and confirmed chemically (Tables 1, 2). The second set of fissures cross-cuts either fresh cores made of mature glauconite or previously altered zones made of Al-rich glauconite, producing Fe-washing from fissure walls, detachment of Fe-poor clayey peloids and fissure filling with Fe–(Al) oxyhydroxides (Figs. 5C–F). Although the Al-rich glauconite of the rims has the same K content as the glauconite of the cores (Table 2), EPMA data (Table 1) demonstrate that the rims have a lower total content in K and Fe and a higher sum of octahedrally coordinated cations (=lower Fe$^{2+}$/Fe$^{3+}$ ratio) than the cores (see also K, Fe and Al X-ray maps in Fig. 10 of Eder et al. 2007). The lower total K content of brown rims (Table 1, Fig. 7) is explained by the presence of berthierine, smectite, and Fe oxyhydroxides diluted within the Al-rich glauconite matrix of the brown areas (Fig. 9A). Consequently, all these phases are considered to be reaction products after Al-enrichment of a previous yellowish green glauconite similar to that of the cores, and that also constituted the brown rims before alteration. The Al-enrichment of the altered glauconite was accompanied by preferential leaching of Fe (probably Fe$^{2+}$) and K from the rims. As indicated by Courbe et al. (1981) and Środoń (1999), weathering of glauconite quickly seems to reverse the glauconitization process. The formation of a ferrous phase such as berthierine, however, requires a change in redox conditions during alteration, and needs further explanation (see below).

The IR data independently confirm that glauconite in the brown rims results from alteration of a previous glauconite, because it reveals higher amounts of hydroxyl ions and of molecular H$_2$O in the glauconite of the sample YAR–10 (Fig. 11B) than in the apparently fresh, mature glauconite of sample M–11–22 (Fig. 11A). This structural and molecular H$_2$O is not only associated with smectite interleaved with glauconite of fresh yellowish green cores, but also with smectite and Fe oxyhydroxides located in the more hydrated alteration-induced rims.

The weak EPR band at $g = 2.787$, found only in sample YAR–10 (Fig. 12), is an independent confirmation of the presence of Fe oxyhydroxides in this sample and, therefore, in the brown rims. Thus, the color change from yellowish green cores to brown rims is due to the partial transformation of the glauconite to goethite or hematite (plus smectite and other Fe oxyhydroxides). In minerals with only ferric iron such as goethite and hematite, all Fe$^{3+}$ sites are always surrounded by Fe$^{3+}$ sites with oxygen (or hydroxyl) linkage in the sheet of octahedra. This induces reddening by absorption of light (Rossman 1996). On the contrary, minerals containing other elements in addition to Fe$^{3+}$ in the sheet of edge-sharing octahedra, such as glauconite and smectite, cannot produce red colors. Therefore, the brown color in the rims is due to the presence of Fe oxyhydroxides. Nevertheless, the amount of Fe oxyhydroxides observed under TEM in the rims is too small to produce a reddening of the color exclusively due to light absorption, as evidenced by the lower Fe content of the dark gray areas in BSE images (EPMA data: Figs. 4B, 5C–E), which correspond to light brown areas in optical images (Fig. 3). Thus, other physical causes must be invoked to explain the observed change in color together with the increase in relief (index of refraction) from the yellowish green cores to the brown rims.

The scattering of light, produced by small, nanometer-sized inclusions of mineral or fluid, is generally responsible for reddening in minerals (Bohren & Huffman 1983). Scattering effects are particularly
enhanced in relation to absorption in the brown rims of the glauconite studied, owing to very different index of refraction of the nanometric inclusions of iron oxyhydroxides in relation to the index of refraction of the surrounding glauconite. A similar example of change from green to brown colors in phyllosilicates was studied by Mellini et al. (1991) for altered chlorite containing nano-inclusions of iron oxyhydroxides and smectite at the TEM scale. Dirty brown colors usually found in many altered minerals (e.g., Franceschelli et al. 1986) can be interpreted in light of the present results as due to the scattering effects of nanometric inclusions.

Glaucotic color as useful paleoenvironmental indicator

Color changes during maturation and alteration of glauconite are useful paleoenvironmental indicators. Dark green (bluish) mature glauconite (like that of YAR–10 cores) is found of the basin, whereas yellowish green and slightly less mature glauconite (like M–11–22) was studied by Mellini et al. (1991) for altered chlorite containing nano-inclusions of iron oxyhydroxides and smectite at the TEM scale. Dirty brown colors usually found in many altered minerals (e.g., Franceschelli et al. 1986) can be interpreted in light of the present results as due to the scattering effects of nanometric inclusions.

Enhanced in relation to absorption in the brown rims of the glauconite studied, owing to very different index of refraction of the nanometric inclusions of iron oxyhydroxides in relation to the index of refraction of the surrounding glauconite. A similar example of change from green to brown colors in phyllosilicates was studied by Mellini et al. (1991) for altered chlorite containing nano-inclusions of iron oxyhydroxides and smectite at the TEM scale. Dirty brown colors usually found in many altered minerals (e.g., Franceschelli et al. 1986) can be interpreted in light of the present results as due to the scattering effects of nanometric inclusions.


PETSCHICK, ?, ? (2000):


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