Development of Sulfuric Acid Speleogenetic Deposits within Cavernous Middle Eocene Beds: Inference on Hydrocarbon Gas Seepages, Giza Pyramids Plateau, Egypt

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Abstract: Development of sulfuric acid speleogenetic mineral deposits within cavernous middle Eocene beds of the Pyramids plateau is linked to hydrocarbon gas seepages. The work carried out field observations, binocular, polarizing, scanning and transmitted electron microscopy investigation, X-ray diffraction and X-ray fluorescence analyses. The morphological and petrographic features and chemical composition of the studied mineral deposits reveal a hypogene sulfuric acid speleogenesis. A model comprised of the following stages can be used to explain the presence of these features. (1) Ascent of hydrogen sulfide (H\(_2\)S) gas associated with hydrocarbon seepages from the Cretaceous reservoirs under reducing conditions followed by oxidation to sulfuric acid (H\(_2\)SO\(_4\)), (2) descent of carbonic acid (H\(_2\)CO\(_3\))-rich solution generated from surface sources, (3) reaction of H\(_2\)SO\(_4\) and H\(_2\)CO\(_3\) with the calcareous and argillaceous host rocks, (4) formation of H\(_2\)SO\(_4\) speleogenetic by-products represented by natroalunite, aluminium-phosphate-sulfate, hydrated halloysite and Fe/Mn oxides within the replacive gypsum, and (5) subsequent stresses due to the formation of nearby stratiform cavities gave rise to the development of fractures/veinlets filled with displacive fibrous satinspar gypsum. The study sets the paleokarst features of the Giza Pyramids plateau within a hypogene sulfuric acid karst system developed by the action of groundwater containing H\(_2\)S, H\(_2\)SO\(_4\) and H\(_2\)CO\(_3\).

Key words: karstification, natroalunite, aluminium-phosphate-sulfate, hydrated halloysite, enterolithic growth, satinspar gypsum

1 Introduction

The Eocene limestone plateau belongs to the Paleocene-Eocene carbonate platforms, which represent the largest and oldest extension of carbonates all over Egypt. Because of their wide extension in the Western Desert of Egypt, all types of karstification are shown on these platforms (Embabi, 2018). The studied cavernous middle Eocene beds exposed on the western side of the Giza Pyramids plateau, which constitutes a part of the Eocene plateau, are characterized by the occurrence of mineral deposits represented by natroalunite (Na-alunite) nodules and its finely intergrown aluminium-phosphate-sulfate (APS) and hydrated halloysite, Fe/Mn oxide inclusions and gypsum veinlets. APS minerals represent the structural analogues of natroalunite; they collectively belong to the so-called alunite supergroup that comprises the alunite, beudantite and crandallite groups (Jambor, 1999). They have a general formula AB\(_3\) (XO\(_4\)\(_2\)) (OH)_x, where A sites represent large cations of the elements Na, U, K, Ag, NH\(_4\), Pb, Ca, Ba, Sr and REE in 12 fold coordination. B sites are occupied by cations of the elements Al, Fe, Cu and Zn in octahedral coordination. In nature, the anion (XO\(_4\))\(^{2-}\) is dominated by P and S (Scott, 1987a, b). The minerals of alunite group, including natroalunite, are free of phosphate, but contain sulfate (SO\(_4\))\(_2\) (Dill, 2001), while the other two groups are either free of sulfate and contain phosphate or contain sulfate that is partially replaced by PO\(_4\) or AsO\(_4\) (Wilson, 1985; Rasmussen, 1996; Pe-Piper and Dolansky, 2005; Galán-Abellán et al., 2013). The intermediate sulfate-phosphate composition yields the APS minerals (Bayliss et al., 2010).

In Egypt, alunite/natroalunite has been recognized in several rock units of the Phanerozoic rock sequence (Said, 1990). It has been studied in detail from several localities (e.g., Shukri, 1954; El Shazly et al., 1963; El-Sharkawi, 1977; Goldberg, 1980; Hilmy et al., 1983; Rouchy and Pierre, 1987; Hassan and Baioumy, 2007). Afterward, disseminated APS minerals were reported in the upper Cretaceous shallow marine ooidal ironstone in the Aswan area by Salama (2014). The genesis and paleoenvironments of the natroalunite in Darb El Fayum (the area of the present study), located on the western side of the Giza Pyramids plateau have been inferred by Hilmy et al. (1983) based on the texture and morphology of the nodules. The present contribution aims to give an account about the geologic setting, mineralogical and geochemical characterization as well as the genesis and paleoenvironments of whole sulfate, including natroalunite, APS minerals and gypsum and the host...
cavernous middle Eocene rock sequence.

The Eocene carbonates overlie the Cretaceous rock succession, which in many oil fields of the Western Desert are developed constituting a potential source rock and good reservoir for economic oil and gas production (e.g., Aadland and Hassan, 1972; El Gezeery and O’conor, 1975; Deibis, 1976; Barakat et al., 1987; Taher et al., 1988; Hantar, 1990; Darwish, 1994; Khaleed, 1999; El Nady et al., 2003; Maky and Saad, 2009; El Nady and Harb, 2010; El Nady and El-Naggar, 2016; El Nady and Mohamed, 2016). In the light of this geologic setting, the study discusses the origin of the studied mineral deposits within the host middle Eocene rock sequence in relevance to hydrocarbon gas seepages from the subsurface accumulations. The associated H₂S to the petroleum is probably bacterial in origin, oxidized upon reaching groundwater, and resulting in highly concentrated sulfuric acid that can then result in the formation and precipitation of sulfate (Khalaf, 1990). The present work also suggests that the paleokarst features observed in the carbonate rocks of the Giza Pyramids plateau were formed via the action of sulfuric acid-rich solutions beside the action of carbonic acid-rich solutions. To achieve the aim, the present work focuses on field observations, petrography, mineralogical and geochemical composition of the studied mineral deposits and the host rock sequence.

2 Materials and Analytical Techniques

Morphological features of the studied mineral deposits and the host middle Eocene rock sequence, including dissolution features, color, geometry, growth pattern, texture and composition were examined in the field at an exposure located in Darb El Fayum, western side of the Giza Pyramids plateau (Fig. 1a). Nodules, gypsum veinlets and host rocks were sampled. The nodules were carefully extracted from the host rocks and the surrounding gypsum and iron oxide coating.

To determine microtextures and microscopic features of the studied mineral deposits and the host rocks, 18

![Fig. 1. Location of the studied section and dissolution features.](image)

(a) The location of the studied mineral deposits and the host rock sequence exposed on the western side of the Giza Pyramids plateau; (b) dissolution fissures, voids and undercutting; (c) karst pocket consists of allochthonous sediments. Note the squeezing and pinch out of the karst filling near a minor fault.
representative fresh samples were selected from the collected samples for the preparation of thin sections, and then examined using transmitted plain and polarized light. The staining technique of Dickson (1966) was used to stain the carbonate thin sections with alizarin red-S to discriminate between calcite and dolomite, and then the thin sections were stained with a mixture of potassium ferrieánide and alizarin red-S to indicate the presence of ferroan or non-ferroan calcite and/or ferroan dolomite.

The fresh samples of the studied nodules were investigated using a binocular microscope to examine the surface features and the nature of the contact with the host rocks. The internal structures of the nodules were investigated using a high-resolution transmitted electron microscope (HRTEM) Model Jeol 2100 JEM. The samples were ground using an agate mortar, and then suspended in distilled water and dispersed using ultrasonics and dropped on high-resolution transmitted electron microscope grids. Operating conditions were 200 kV accelerating voltages and a beam current ranging from 109 μA to 112 μA. Rock slabs of selected nodules and the host rocks were cut and coated with platinum for micromorphologic and geochemical analyses. They were examined using a scanning electron microscope (SEM) Model Jeol 6510 JSM LA equipped with an energy dispersive X-ray analyzer (EDX). The operating conditions were 10-30 kV accelerating voltages, and 7-14 mm working distance.

The mineralogical composition of the nodules and the associated rock samples was studied by X-ray diffractometry (XRD). The samples were disintegrated by mechanical crushing and ultrasounds. A PAN analytical XRD equipment model X’Pert PRO with Secondary Monochromator, Cu-radiation (l = 1.542 Å) at 45 kV, 35 mA and scanning speed 0.02°/s was used. The diffraction peaks between 2θ = 2° and 60°, corresponding spacing (d), Å and relative intensities (I/I0) were obtained. The diffraction charts and relative intensities are obtained and compared with ICDD files.

The X-ray fluorescence (XRF) technique was used for the analysis of major and trace elements with a very high accuracy; it covers a range of elements from Beryllium to Uranium by using ceramic Rh wide range tube. The XRF analysis was conducted for powder (< 74 mm) samples of nodules and the host rocks using XRF equipment PW 2404 with six analyzing crystals. Crystals (LIF-200), (LIF-220) were used for estimating Ca, Fe, K, Ti, Mn and other trace elements from Ni to U, while crystal (TIAP) was used for determining Mg and Na. Crystal (Ge) was used for estimating P and crystal (PET) for determining Si and Al and PXI for determining Na and Mg. The concentration of the analyzed elements was determined by using software Super Q, and Semi Q programs with accuracy of 99.99% and confidence limit of 96.7%. The estimation of the major and trace elements were done as powder pellets (Pellets method), which were prepared by pressing the powder of the sample in Aluminium Cup using Herzog presser and 10 ton pressure. On the other hand, using the fusion method in platinum crucible (Bead method) gave better results for light elements measurements, but owing to the presence of P as phosphate and S as sulfide in the studied samples, which led to corrosion in platinum crucible, we used the pellet method as mentioned above. The XRD and XRF analyses were carried out in the Central Laboratories Sector of the Egyptian Mineral Resources Authority (EMRA), Dokki, Giza, Egypt. Binocular and optical microscopy investigations and SEM and TEM analyses were carried out in the analytical labs in the Faculty of Science, Beni-Suef University.

3 Analyses and Results

3.1 Stratigraphy and field observations

The middle Eocene rock sequence in the study area has been subdivided into the Mokattam, Qurn and Wadi Garawi Formations (Strougo, 1985). Generally, the exposure displays several paleokarst features such as brecciation, dissolution fissures, cavities, voids and undercutting as well as pockets of karst filling consisting of allochthonous sediments within the carbonate beds (Fig. 1b, c).

The occurrence of the studied mineral deposits characterizes the argillaceous beds of the Wadi Garawi Formation (Fig. 2). The host argillaceous horizon measures about 2.5 m thick and starts with brown, calcareous, wrinkly- and thinly-laminated mudstone (Fig. 3a) displaying a papery weathering surface. The basal part is irregular and marked by dissolution cavities/fissures (Fig. 3a). The laminated mudstone grades up to gray, hard, saliferous and gypsiferous massive mudstone (Fig. 3b). It displays mottled green and red coloration and is invariably intersected by gypsum veinlets. The massive mudstone beds are extensively fractured and dissected into blocks that slide and rotate in response to the growth of the gypsum veinlets (Fig. 3c). Generally, the mudstone beds are poorly fossiliferous and enclosed by sandy/gravelly dolostone beds, being intercalated with thin/medium gypsum interbeds in the upper part. The phosphatic dolostone beds exhibit variable shades of brown color as earthy, greenish and yellowish as well as being mottled with green tint. Phosphate grains are represented by bone fragments, shark teeth, coprolites and peloids.

Mineral deposits consisting of natroalunite and gypsum are recognized along two horizons (Fig. 2). The natroalunite is fine-grained, hard compact rock, breaking with a conchoidal fracture and exhibiting a dull porcelain-like appearance (Fig. 3d), and sometimes displays a powdery appearance. It is characterized by a distinct white color within the surrounding varicolored host rocks and displays a nodular and banded appearance. The lower horizon exists at the base of the thinly-laminated mudstone and displays a nodular form. The nodules reach up to 5 cm in diameter and are distributed in a relatively discontinuous manner, being incorporated within prismatic gypsum crystals. Thin ochreous materials surround the lower and upper boundaries of the nodular horizon. The upper horizon occurs near the base of the massive mudstone and comprises densely packed nodules, which form a more or less continuous band ranging in thickness from 3 cm to 15 cm (Fig. 3b). The nodules reach up to 10 cm in diameter and exhibit a distinct growth pattern consisting of contorted, ribbon-like layer (Fig. 3c).
They are developed parallel to the bedding planes and are usually incorporated into colorless prismatic gypsum crystals. The top of the nodules shows distinct polygons (Fig. 3d, e). Patches of ochreous materials (Fig. 3b) and concretions of iron and manganese oxides associate the natroalunite band.
The natroalunite band is irregular and dissected with a mesh-work of gypsum veinlets that are obliquely oriented in several directions (Fig. 3b, c). The gypsum is colorless, being locally stained by black spots and exhibits some tarnish coloration (Fig. 3f). It is translucent and displays fibrous texture and silky luster. In a single vein, the gypsum is usually zoned and made up of two or more parallel layers separated by a central straight parting that is parallel to the fracture surface (Fig. 3f). The crystal fibers are elongate and oriented with their long axes perpendicular to the fracture walls and have a uniform width.

3.2 Petrography of mineral deposits and the host rock sequence

The binocular microscopy investigation shows the varied textures of the studied nodule surfaces. On the exposed surface, it shows a white color and a fine crystalline texture with scattered colorless coarse crystals. It is coated with a salty crust exhibiting a glassy luster. Locally, patches of the underlying deposits protrude through irregularly dissolved zones of the salt crust. Brownish-orange lumps and black spots are spread randomly on this surface. The nodules are affected by several minute fractures, being perpendicular to the surface and exhibit two directions. The fractures are mostly unfilled, but are filled with a black bituminous matter near the contact with the host rocks. On the other hand, the surface opposite to the host rocks is characterized by a dark tint and the presence of wide fractures filled with a fibrous gypsum. Salt forms wide patches rich in disseminated black spots. Inclusions of
ochreous clay materials are commonly observed on this surface. The host clays, gypsum and salt are abundantly observed on this surface.

Investigation of the studied nodules microfacies reveals the cryptocrystalline aggregate habit of the natroalunite (Fig. 4a) and very low birefringence colors. Brown fibrous aggregates showing moderately high relief and low birefringence colors are also scattered in the interstices. The natroalunite aggregates contain abundant inclusions represented by iron oxide, pyrite, organic matter and clays (Fig. 4b). The inclusions are very fine in size, being disseminated through the aggregates with a random orientation. Sometimes, they display a network pattern ranging in color from opaque (pyrite, organic matter and iron/manganese oxides) to translucent (clay relicts). Corroded quartz and feldspar grains of fine silt size are also distributed within the natroalunite aggregates (Fig. 4b).

Microscopically, the host mudstone consists of quartz grains and clay aggregates. Quartz grains are silt-sized and having an angular outline and a brecciated appearance. Clay minerals form silt-sized aggregates rich in iron oxide and calcareous matter. The mudstone is characterized by a microscopic lamination consisting of light, thin/thick algal laminae alternating with dark, thin clay laminae. The algal laminae show a high order of interference colors, while the clay laminae are pale-yellow under plain light and isotropic under crossed nicols. Discrete patches of iron oxide occur within the algal laminae. The laminae are intersected by obliquely oriented veinlets, being filled with halite and/or bituminous matter. The halite is colorless and shows a very low relief in a polarized light, and appears isotropic with crossed nicols. Thin irregular planar fenestrae arise within the undulatory algal laminae and are filled with calcareous matter.

Petrographically, the beds underlying and overlying the host mudstone consist of sandy dolostone. The facies mainly consists of dolomite rhombs with dispersed quartz and phosphate grains. It exhibits a coarse sand/gravel grain size and a massive texture (Fig. 5a) alternating with...
fine sand grain size and a laminated texture (Fig. 5b). Dolomite rhombs are non-ferroan, euhedral with planar crystal boundaries and range from coarse silt to medium sand size. Mostly, the rhombs are scattered in the matrix of the rock, but some cavities and fissures are occluded with coarser dolomite rhombs (Fig. 5c). Occasionally, the rhombs coalescence and merge together in a tight interlocking pattern (Fig. 5c-e). Reworked quartz grains displaying a pebble grain size and angular to subrounded edges are scattered through the matrix. They are weathered, altered, fractured, and rich in inclusions and sometimes are corroded with the surrounding dolomite rhombs (Fig. 5d, e). Patches of coarse calcite rhombs are distinct as a cavity filling. The rock displays several microscopic dissolution features represented by cavities and fissures, which are either filled or coated with iron oxides. Phosphate grains are mainly represented by peloids and coprolites (Fig. 5d-f) with rare bone fragments (Fig. 5f) and sediment intraclasts.

Secondary electron images (SEI) support the binocular microscopy investigation. They demonstrate the exposed surface of the studied nodules is encased with a thin halite crust that is partially dissolved (Fig. 6a, b) and shows the tubular crystal aggregates of the underlying materials (Fig. 6b, c). The halite displays a homogeneous smooth texture with well-distinct crystals on the surface (Fig. 6d). Cylindrical and oval-shaped minute (2 mm) bodies protruding from the underlying aggregates are attached to the halite crust. Locally, clusters of tiny (< 2 mm), well-formed, equigranular pseudocubic crystals are also observed on the top of the halite crust and dispersed

Fig. 5. Microphotographs of the sandy dolostone.
(a) Coarse-grained massive sandy dolostone consisting of dolomite rhombs (1), quartz and phosphate grains represented by bone fragments (2), P.L.; (b) fine-grained laminated sandy dolostone, P.L.; (c) cavity filled with coarse dolomite rhombs coalescence and merged in a tight interlocking pattern, P.L.; (d, e) fractured quartz grains (1) corroded with dolomite rhombs, being coalescence and merged in a tight interlocking pattern. Note phosphate grains are represented by peloids (2) in (d) and coprolites (3) in (e), P.L.; (f) phosphate grains represented by peloids, coprolites (1) and fish scales (2). Note prismatic habit and well-preserved organic structure of the fish scale, C.N.
within the rock matrix (Fig. 6e, f).

SEI images of the natroalunite surface opposite to the host rocks show the character of the sheet-like structure of the associated clay (Fig. 7a). The tubular crystal aggregates are also covered with a partially dissolved halite crust that displays a blocky form. On the other hand, gypsum commonly occurs on this surface and displays a fibrous texture (Fig. 7b). The SEI of the host mudstone shows coarse globular and irregular shaped bodies scattered within the fine-grained granular matrix (Fig. 7c). The dolostone beds overlying the upper mineral deposits horizon show many dissolution cavities (Fig. 7d) and are characterized by hollow rhombs with dissolved cores and dissolution resistant outer zones (Fig. 7d). The hollow cores are filled up with insoluble residues of the clay matrix (Fig. 7d). High-resolution transmitted electron microscopy (HRTEM) investigation shows the pseudocubic form of the natroalunite, tubular form of the halloysite (Fig. 7c, f) and euhedral pseudohexagonal shape of the kaolinite crystals (Fig. 7f).

3.3 Mineralogy of mineral deposits and the host rock sequence

The mineralogy of the studied nodules and the host rock sequence has been identified by X-ray diffraction (Table 1). The bulk sample of the studied nodules is composed of 40% Na-alunite, 30% mixed chlorite-vermiculite-montmorillonite clay, 20% gypsum and 10% halite. The diagnostic reflections of the studied nodules are at 4.94, 3.49, 2.82, 2.47, 2.26, 1.90, 1.74, 1.66 and 1.64 Å. The present values are notably similar to natroalunite peaks discussed in the literature (e.g., Gaied et al., 2015). The XRD results give the following chemical formulas: (K$_{0.805}$ Na$_{0.132}$ (H$_2$O)$_{0.063}$) Al$_3$ (SO$_4$)$_2$ (OH)$_6$ and Na$_{0.5}$ Al$_6$ (Si, Al)$_8$ O$_{20}$ (OH)$_{10}$ H$_2$O for the studied natroalunite and the associated mixed clay minerals, respectively. X-ray diffraction of the host rock sequence reveals the presence of quartz (22.85%-40%), calcite (11.42%), kutnoklorite (77.14%), dolomite (68.15%-76.24%), iron phosphate (23.75%), gypsum (15%-20%), halite (10%-37.61%), montmorillonite (30%) and kaolinite (10.95%-20%).
Fig. 7. (a–d) Secondary electron images of natroalunite surface opposite to the host rock. (a) Sheet-like structure of the associated clay; (b) gypsum displays a fibrous texture (arrows); (c) coarse globular and irregular shaped bodies (arrows) scattered within fine-grained granular matrix; (d) dolostone hollow rhombs (1) with dissolved core (black arrow). Note the dissolution cavities (white arrow) and the hollow cores are filled with insoluble residues of clay matrix (2); (e, f) plan view images of high-resolution transmitted electron microscope of powder sample; (e) the pseudocubic form of natroalunite (1) and the tubular shape of the halloysite clay (2); (f) euhedral pseudohexagonal shape of the kaolinite crystals (1) and the tubular shape of halloysite (2).

Table 1 Semi-quantitative mineralogy of the studied natroalunite nodules and the host rock sequence

<table>
<thead>
<tr>
<th>Minerals</th>
<th>M1 (%)</th>
<th>M3 (%)</th>
<th>M8 (%)</th>
<th>M9 (%)</th>
<th>Upper Natroalunite horizon (%)</th>
<th>M10 (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz</td>
<td>31.84</td>
<td>-</td>
<td>40.00</td>
<td>35.00</td>
<td>-</td>
<td>22.85</td>
</tr>
<tr>
<td>Montmorillonite</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>30.00</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Kaolinite</td>
<td>-</td>
<td>-</td>
<td>10.95</td>
<td>20.00</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Chlorite-vermiculite-montmorillonite</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>30.00</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Na-alunite</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>40.00</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Kutnohorite</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>77.14</td>
</tr>
<tr>
<td>Calcite</td>
<td>-</td>
<td>-</td>
<td>11.42</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Gypsum</td>
<td>-</td>
<td>-</td>
<td>15.00</td>
<td>20.00</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Halite</td>
<td>-</td>
<td>-</td>
<td>37.61</td>
<td>-</td>
<td>10.00</td>
<td>-</td>
</tr>
<tr>
<td>Dolomite</td>
<td>68.15</td>
<td>76.24</td>
<td>-</td>
<td>-</td>
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<td>-</td>
</tr>
<tr>
<td>Iron phosphate</td>
<td>-</td>
<td>23.75</td>
<td>-</td>
<td>-</td>
<td>-</td>
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</tr>
<tr>
<td>Total (%)</td>
<td>99.99</td>
<td>99.99</td>
<td>99.98</td>
<td>100.00</td>
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</tr>
</tbody>
</table>

Note: M1 and M3 represent the sandy phosphatic dolostone. M8 represents the host laminated mudstone of the lower natroalunite horizon. M9 represents the host massive mudstone of the upper natroalunite horizon. M10 represents the sandy dolostone overlying the upper natroalunite horizon.
3.4 Geochemistry of mineral deposits and the host rock sequence

The chemical composition obtained from the EDX analysis of the studied nodules is given in Table 2 and the chemical composition of the host rocks is given in Table 3. The geochemical results of the pseudocubic crystals support a natroalunite composition. They show higher Al₂O₃ ranging from 28.35% to 51.17% and SO₃ ranging from 26.29% to 37.07% with a positive correlation between K₂O and SO₃ and a negative correlation between Na₂O and SO₃. Also, SiO₂ and Cl are detected (spots no. 1, 2, Table 2).

The major constituents of the tubular-shaped aggregates are represented by Al₂O₃ and SiO₂ and the minor constituents are represented by CaO, K₂O, Na and Cl (spot no. 3, Table 2). Cylindrical, platy and oval-shaped bodies attached to the halite crust and protruded from the underlying aggregates are composed of SiO₂, Al₂O₃, K₂O and CaO. They are rich in S, Na and Cl with a negative correlation between S and Cl (spots 4-7, Table 2). The matrix contains a high content of Al₂O₃ and SiO₂ with traces of K₂O, Na and Cl (spot no. 8, Table 2). Commonly, the halite crust shows higher sodium and chlorine content, while in the present study it is contaminated by SiO₂ and Al₂O₃ (spots no. 9, 10, Table 2).

The results of the surface opposite to the host rocks show that the sheet-like structures contain CaO and SiO₂ as major oxides mixed with Al₂O₃, Na₂O, SO₃, FeO, K₂O and Cl (spot no. 11, Table 2). Commonly, patches of matrix found in the fibrous gypsum (spots no. 12, 13, Table 2) are similar in chemical composition to that of the tubular-shaped aggregates (spot no. 17, Table 2). Halite blocks show traces of SiO₂, Al₂O₃, CaO, SO₃ and FeO (spots no. 14-16, Table 2). Oxides in the host rocks are mainly represented by SiO₂ and Al₂O₃ mixed with minor CaO, K₂O, FeO and Na₂O (spots no. 1-6, Table 3). Oxides of Fe₂O₃, MgO, P₂O₅ and TiO₂ are also detected in the host rocks (spots no. 7-11, Table 3). Traces of carbon are measured in the globular bodies that are dispersed within the rock matrix. Na, Cl and S are detected in the host mudstone (Table 3).

Major and trace elements of the studied nodules and the host mudstone obtained from the XRF analysis are listed in Table 4. The results show the major elements forming natroalunite are represented by SiO₂, Al₂O₃ and SO₂ with traces of CaO, Fe₂O₃, TiO₂, MnO, MgO, P₂O₅, Cl (Table 4). The concentrations of Na₂O and K₂O in the studied nodules are almost the same (Table 4), but Na₂O is less and K₂O is much compared to the chemical composition of an ideal natroalunite (Na₂O = 7.8% and K₂O = 0%). The analysis demonstrates the presence of an assortment of trace elements represented by V, Cr, Ni, Zr, Zn, Co, Cu, Rb, Sr, Y, Nb, Ba, La, Th and Pb. Results from the studied nodules show high concentrations of Sr and Th relative to the host mudstone, and low concentration of Zr relative to the host mudstone (Table 4). On the other hand, trace elements of V, Cr, Ni, Cu, Co, Rb, Ba and La have high concentrations in the host mudstone relative to that in the studied nodules (Table 4).

4 Discussion

4.1 Morphological and petrographic features

Lithologic attributes of the host rock sequence provide an account about the paleoenvironments. The laminated nature of the mudstone enclosing the lower mineral deposits horizon, lack of fossil diversity and the predominance of oxidizing colors, salt crust and gypsum

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Table 2 Chemical composition (%) of the studied natroalunite nodules

<table>
<thead>
<tr>
<th>Spot no.</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
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<tbody>
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<td>SiO₂</td>
<td>-</td>
<td>6.74</td>
<td>39.23</td>
<td>24.40</td>
<td>13.12</td>
<td>8.41</td>
<td>11.13</td>
<td>47.69</td>
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<tr>
<td>Al₂O₃</td>
<td>51.17</td>
<td>28.35</td>
<td>35.08</td>
<td>27.97</td>
<td>13.50</td>
<td>8.42</td>
<td>36.28</td>
<td>43.56</td>
</tr>
<tr>
<td>CaO</td>
<td>-</td>
<td>-</td>
<td>0.27</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
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<tr>
<td>K₂O</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>25.86</td>
<td>24.84</td>
<td>15.97</td>
<td>-</td>
<td>-</td>
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Note: Spots 1, 2: Pseudocubic crystals. 3: Tubular-shaped aggregates underlying the halite crust. 4-7: Cylindrical, platy and oval-shaped tiny bodies. 8: Matrix.

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veinlets support mudflat environments. While the lack of laminated and mottled coloration in the mudstone enclosing the upper mineral deposits horizon could be attributed to homogenization by burrowing organisms. The wrinkled nature and calcareous composition of the thinly-laminated mudstone indicates the presence of algal filaments. The upper region of the intertidal zone is usually characterized by the presence of both convoluted and folded mats, which experience prolonged periods of desiccation (Riding and Awramik, 2000). The laminated intertidal algal mat peats may be dissected to form algal polygonal mats (Warren and Kendall, 1985). The polygonal form of the scattered nodules within the replacive gypsum was probably inherited from the pre-existing algal polygonal mats. However, the replacive gypsum can preserve bedrock texture and grain shapes (Palmer and Palmer, 2012).

Microscopic features of the studied nodules and the host mudstone, including the presence of discontinuous, wavy to crenulate lamination patterns, planar fenestral fabric, well-distinct microbial structures and clotted microtexture of thrombolite (Fig. 4) support the morphological features (Fig. 3a) and confirms the presence of biogenic remnants of the host algal laminated mudstones. Their dominance in the microfossils usually reflects proximity to the host rocks. The fenestrae were formed in soft sediments and they are common in peritidal microbial mats either from biogenic gas or by bridging (Scholle and Ulmer-Scholle, 2003). The association of microbial structures with traces of poikilotopic halite cement and the prevalence of gypsum veinlets within the studied nodules indicate upward groundwater seepages within the topmost part of the intertidal (mudflat) zone. The fluids flowing through the interstitial pores of the carbonate host sediments occur above the water table (Rosen and Warren, 1990; Schreiber and Tabakh, 2000).

Sulfuric acid plays the same role as carbonic acid in promoting the rate of chemical dissolution as well as its role in the alteration of argillaceous minerals. The dissolution of calcareous matter via sulfuric acid leads to concentration of Ca$^{2+}$ and SO$_4^{2-}$ and the precipitation of replacive gypsum, while the alteration of claystone results in the formation of alunite, hydrated hallosyrite, sulfur and Fe/Mn oxide inclusions within the replacive gypsum (Palmer and Palmer, 2012). The diagenetic growth of natroalunite nodules in the host sediments caused the deformation of the host layers. Such growth pattern resembles the enterolithic structure of ancient anhydrite described by Shearman (1978). Presence of the hollows at the contact between the carbonate rock and the replacive rocks. The fenestrae were formed in soft sediments and they are common in peritidal microbial mats either from biogenic gas or by bridging (Scholle and Ulmer-Scholle, 2003). The association of microbial structures with traces of poikilotopic halite cement and the prevalence of gypsum veinlets within the studied nodules indicate upward groundwater seepages within the topmost part of the intertidal (mudflat) zone. The fluids flowing through the interstitial pores of the carbonate host sediments occur above the water table (Rosen and Warren, 1990; Schreiber and Tabakh, 2000).

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gypsum enclosing the natroalunite nodules (Fig. 3a) indicates intense dissolution. However, the development of subsurface/surface dissolution features and accumulation of ochers, calcareous mudstones and salt minerals within the middle and upper Eocene limestones represent the main karst products of the Giza Pyramids plateau under certain climatic and hydrologic conditions, where the movement of water in the plateau area is mainly upward (El Aref and Refai, 1987).

Fibrous varieties of gypsum have been identified in several settings. The cross-cutting nature of the studied gypsum veinlets (Fig. 3b, c) supports the displacive growth pattern where the gypsum infilled subhorizontal to oblique fractures formed after the consolidation of the host rocks. Moreover, the attributes of the veins crossing the host mudstone, including the mesh-work texture, fibrous and zoning nature of the filling, straight central paring (Fig. 3b, c, f), perpendicular orientation of the crystal on the fracture walls, uniform fibers width and monomineralic composition (i.e., gypsum) of the filling confirm the affinity of the studied gypsum to satinspar fracture fillings. Central parings in veins commonly form in satinspar veins (El Tabakh et al., 1998). The stresses set up in the beds due to the formation of nearby stratiform cavities gave rise to the development of the fractures and veins filled with fibrous satinspar gypsum (Warren, 2006). The sliding and rotation of adjacent blocks to the fibrous-filling fractures (Fig. 3c) reflect the change in nearby dissolution cavities. The fibrous morphology, the symmetry of the fractures/veins filling and absence of anhydrite relics within the gypsum indicate a primary fracture fill material. The consistency of gypsum crystal fibers width and their elongate form, indicate that fracture opening and crystallization occurred at the same rate (El Tabakh et al., 1998). The formation of bordered gypsum veinlets confirms that the clear middle part had been open cracks filled with gypsum, and the dark outer margin was formed by replacement of the host rocks and hence the formation of inclusions (Misik, 1998).

Features of the mineral deposits and clay minerals are more readily observed by SEM than by other conventional means. The tubular morphology of crystals in the studied aggregates is typically of hydrated halloysite. According to Millot (1970), the mineral is assigned to the kaolin group, in which individual layers are separated by sheets of water. It is characterized by a kaolin-like EDX spectrum that shows nearly equal peak heights of Al and Si. The SEM investigation of the studied nodules shows that pseudocubic natroalunite crystals are disseminated within the halloysite clay. The sheet-like texture of the host mudstone is similar to the cantilevered stacking sequence pattern of platelets in kaolinite stacks, which show pseudohexagonal plates during the TEM investigation. The coexistence of pseudohexagonal kaolinite plates with natroalunite indicates intensive leaching via the action of acidic circulating water (Khoury, 2002). The dominance of globular/irregular-shaped bodies (Fig. 7c) rich in carbon within the host mudstone could be attributed to microbial structures. The presence of dissolution cavities within the associated dolostone beds as well as the dominance of hollow rhombs with dissolved core confirm extensive dissolution related to kaaristification processes. The partial dissolution of the salt crust casing the nodules can be attributed to undersaturation of the pore water with respect to halite (Warren, 2006).

4.2 Mineralogical and geochemical evidences

The XRD spectra of the studied nodules and the host rocks support the petrographic and chemical analysis as well as show the type of the present impurities. Natroalunite (NaAl₃(SO₄)₂(OH)₆) formed if the Na:K, atomic ratio equals or exceeds unity (Hall, 1978). However, alunite (KAl₃(SO₄)₂(OH)₆) rarely exhibits the pure potassium end-member composition and the partial substitution of Na for K (up to greater than 95 mol% Na) is common in all alunite forming environments (Stoffregen et al., 2000). The detection of kutnohorite (CaMn(CO₃)₂) in the XRD pattern of carbonate beds overlying the host horizon (Table 1) reflects enrichment in manganese. The silicate weathering provides a significant source of Mn²⁺ to surface and ground waters (Gross, 1965; Post, 1999). However, when there are high levels of soluble Mn²⁺, this divalent ion can substitute for Ca²⁺ in authigenic carbonate phases (Johnson et al., 2016). On the other hand, the detection of significant amounts of iron phosphate within the dolostone beds underlying the host horizon (Table 1) is supported by the petrographic investigation that shows an abundance of phosphate grains intermixed with dolomite rhombs (Fig. 5a, d-f). Generally, mudflats are known to be important phosphorus reservoirs. Locally, bacteria may increase PO₄³⁻ and/or Fe through metabolic activity, and hence the required critical saturation state is exceeded and precipitation occurs (Cosmidis et al., 2014).

Geochemical analyses of the studied nodules provide support for the genesis of the mineral deposits. The detection of Si, Cl and excess of Al in EDX spectra of the studied nodules (spots no. 1, 2, Table 2) supports the presence of clay mineral impurities that were also confirmed by the mineralogy and petrography, while traces of Fe₂O₃ may correspond to the presence of iron oxide. The expected amount of alkali cations of an ideal natroalunite equals 7.8 wt% and that of an ideal alunite equals 11.40 wt%, while the amount of K₂O and Na₂O in the studied samples equals 3.66 wt %. This confirms that the studied sample belongs to natroalunite and is far from the alunite end member. The enrichment of the studied natroalunite nodules with major and trace elements strongly supports a hydrocarbon-associated water activity. The presence of hydrocarbons leads to reducing conditions, and hence the associated groundwater is characterized by low EH values and contains H₂S, NH₄, Mn and elevated CO₂ content (Matthess, 1994). The high content of Sr constitutes a supplementary indicator of oilfield water (Chilingarian et al., 2005; Satyanarayana, 2011).

Diagenetic processes that took place in the host sediments yield a variety of minerals and fabrics. The interaction of montmorillonite with H₂SO₄-bearing vadose solutions leads to the transformation of this clay to hydrated halloysite (Hill, 1987, 1990). The mechanism of
halloysite formation is attributed to the enrichment of Al due to the mobility of Ca, Na, Fe, Si and Mg elements in meteoric geochemical environments. Such a mechanism occurs in either hot or cold water environments (Keller and Hanson, 1969; Keller et al., 1971).

Individual APS crystals have not been visually identified in the present study. The small crystal size (<10 μm) and low concentration (ca. 0.05 wt%) hampered the detection of individual APS minerals by conventional mineral analysis techniques such as X-ray diffraction, and low magnification optical microscopy (Rasmussen, 1996; Gaboreau et al., 2005). The detection of \( \text{P}_2\text{O}_5 \) in the geochemical results leads to the suggestion that APS has formed amongst the natroalunite nodules. The predominance of marine phosphate grains within the carbonate and mudstone beds encasing the natroalunite nodules (Fig. 5) supports the occurrence of an APS phase within the studied mineral deposits. Weathering of phosphates leads to depletion of F, Ca, Na and Sr (Dill, 2001). The depletion of Ca raises the acidity of the meteoric water, which causes total removal of apatite and mineralization of the APS (Mordberg, 1999; Dill, 2001; Mordberg et al., 2008). At sufficiently high dissolved phosphate concentrations, apatite is stable down to a pH of about 6, though at low phosphate activities (\( \text{H}_3\text{PO}_4 \) less than 8) the apatite is already converted to APS phases at pH 7 (Dill, 2001). Moreover, PO₄ and Cd are commonly present in the hydrocarbon-associated water, which is also characterized by a higher salt concentration than that of sea water (Matthess, 1994). Perched water tables and connate and/or formation waters control APS mineralization in karst cavities formed via the ascent of \( \text{H}_2\text{S} \) from deep oil reservoirs. The detected Sr and P in the studied nodules might represent the intergrown goyazite phase of the APS aggregates. Ba and Ca elements are also assigned to APS mineral inclusions; they collectively indicate the hypogenic origin of alunite (Rye et al., 1992). Moreover, APS minerals represent an important basin for mineralization in karst cavities formed via the ascent of \( \text{H}_2\text{S} \) from deep oil reservoirs. The depleted Ca raises the acidity of the meteoric water, which was produced at depth beneath the surface rather than at the surface or soil \( \text{CO}_2 \) or near other surface acid sources (Klimchouk, 2009). This could take place due to the ascent of acidic fluids, which is related to deep oil deposits (Hill, 1987; Auler and Smart, 2003; Tisato et al., 2012), while epigenic karstification is formed by meteoric water (Ford and Williams, 2007).

Spearoogenesises by \( \text{H}_2\text{S}:\text{H}_2\text{SO}_4 \) reactions with carbonate bedrock yields the so-called speleogenetic by-products. Gypsum, elemental sulfur, hydrated halloysite and alunite/natroalunite are amongst the reported primary sulfuric speleogenetic by-products. The precipitation of APS solid solution series also occurs via superegen and hypogene processes (Dill, 2001). They are usually associated with the alteration of phosphorite deposits or weathering profiles of tropical soils (Mordberg, 1999; Dill, 2001; Mordberg et al., 2008). Furthermore, Polyak and Provencio (2001) indicated that the alteration of clays above hydrocarbon reservoirs can yield similar mineral associations, where the water table movement has been active, and it can also occur in non-cave areas as well. A model comprised of the following stages can be used to explain the genesis of the studied sulfate mineral deposits. (1) Ascent of hydrogen sulfide gas associated with hydrocarbon seepages from the Cretaceous reservoirs under reducing conditions folowed by oxidation to sulfuric acid [Eq. 1], (2) descent of carbonic acid-rich solution generated from surface sources [Eq. 2], (3) reaction of \( \text{H}_2\text{SO}_4 \) and \( \text{H}_2\text{CO}_3 \) with the calcareous and Al/K-rich clay host rocks [Eqs. 3-5], (4) formation of \( \text{H}_2\text{SO}_4 \) speleogenetic by-products represented by natroalunite and its intergrown APS and hydrated halloysite along with Fe/Mn oxide inclusions within replacive gypsum, and (5) subsequent stresses due to the formation of nearby stratiform cavities gave rise to the development of displacive fibrous satinspar gypsum veinlets.

\[
\begin{align*}
\text{H}_2\text{S} + 2\text{O}_2 & \rightarrow \text{H}_2\text{SO}_4 \\
\text{H}_2\text{O} + \text{CO}_2 & \rightarrow \text{H}_2\text{CO}_3 \\
\text{CaCO}_3 + \text{SO}_4^2\text{−} + 2\text{H}^+ & \rightarrow \text{CaSO}_4 \cdot 2\text{H}_2\text{O} + \text{CO}_2 + \text{H}_2\text{O} \\
\text{CaCO}_3 + \text{H}_2\text{CO}_3 & \rightarrow \text{Ca}^{2+} + 2\text{HCO}_3^− \\
3[\text{K},\text{Na}][\text{Al},\text{Mg,Fe}][\text{Si},\text{Al}][\text{O},\text{OH}][\text{H}_2\text{O}] + 2\text{H}_2\text{SO}_4 & \rightarrow 9\text{SiO}_2 + \text{Na}^+ + \text{K}^+ + \text{Fe}^{2+} + \text{Mg}^{2+} 
\end{align*}
\]
5 Conclusions

The mineral deposits association described in the present contribution reveals new insights into the speleogenesis of the Giza Pyramids plateau. The formation of natroalunite, APS minerals, hydrated halloysite, Fe/Mn oxides and replacive gypsum indicates deposition in acidic environments in which sulfuric acid-induced dissolution of the carbonate bedrock and alteration of the associated clays are the predominant processes. Formation of hollows at the contact between carbonates and replacive gypsum, cavities within dolostones and hollow rhombs with dissolved core and accumulation of ochers, calcareous mudstones and salt minerals represent the main dissolution features supporting the speleogenetic genesis of the present mineral deposits. The coexistence of pseudohexagonal kaolinite plates with natroalunite indicates intensive leaching via the action of acidic circulating water. The association of microbial structures, traces of poikilotopic halite cement and the prevalence of gypsum veinlets indicate upward groundwater seepages within the topmost part of the host mudflat. The enrichment of the studied natroalunite with major and trace elements strongly supports a hydrocarbon-associated water activity. The detection of P2O5 in the geochemical results confirms the growth of fine APS within the natroalunite nodules. The attributes of the studied mineral deposits are well-fit with the features that were documented in the other H2SO4 systems around the world.

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References


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