EXPERIMENTAL TAPHONOMY OF AVIAN EGGS AND EGGSHELLS : EFFECTS ON EARLY DIAGENESIS

by

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SUMMARY

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ABSTRACT

We experimentally explore the early taphonomic stages involving the decay and biodegradation of buried eggs and eggshells. Unfertilised commercial chicken eggs and eggshell fragments were buried in plastic containers and were kept under controlled conditions for eight months. Half of the containers were filled with marl, and the remainder with sand. All were saturated with fresh tap water, acidified water, sulphated water, or seawater. They were kept in the dark at 23.4-26 ° C, except one, which was kept in a heating chamber at 37.4°C. We expected that different burial conditions would produce distinct taphonomic outcomes. Instead, the taphonomic alterations of buried eggs parallel that of the alteration of egg proteins (i.e., denaturation and/or putrefaction) with an additional role played by the eggshell. Mummification, encrustation, distortion and fragmentation, and necrokynesis (vertical displacement) depend on organic matter decay. The experiment identifies environmental conditions that may favour or actively promote these taphonomic processes. Of these, early pyritization is one of the most relevant. For comparative purposes, samples of fossil and extinct eggshell representing three distinct environmental burial conditions were examined. These included *Megaloolithus*, *Caiman crocodilus*, and *Struthio camelus* ootypes. The geochemical analysis of these eggshells showed no significant differences among the chemical variables of these fossil and extant ootypes. Eggshells exhibited a stable composition over a range of experimental conditions.

RESUMEN

En el presente trabajo se explora los estados tafonómicos tempranos relacionados con procesos descomposición y biodegradación de huevos aviarios y cáscaras que fueron previamente enterrados. Huevos de gallina sin fertilizar y algunas cáscaras de gallina y avestruz se enterraron en contenedores plásticos y se mantuvieron bajo condiciones controladas. La mitad de los contenedores se rellenaron con margas, los demás contenedores fueron rellenos de arena. Todos ellos se saturaron bien con agua de grifo, agua ácida, sulfatada o agua marina. Se colocaron en un lugar oscuro a temperatura constante, excepto uno que se introdujo en una estufa a 37,4°C. Esperábamos que cada uno de los diferentes ambientes de enterramiento dieran lugar a características tafonómicas propias. Por el contrario, la comprensión de las alteraciones tafonómicas que sufrieron los huevos enterrados depende de la alteración de las proteínas del huevo (desnaturalización y /o putrefacción) más el papel que juega la propia cáscara. Momificación, encostramiento, distorsión y fragmentación y necrocinesis (desplazamiento vertical) están regidos por la descomposición de la materia orgánica. Los parámetros ambientales actúan ajustando o potenciando estos procesos propios de la descomposición de la materia orgánica. La experiencia llevada a cabo detecta posibles condiciones ambientales que favorecen o promueven estos cambios tafonómicos. La piritización es uno de los procesos más relevantes que han ocurrido por su prontitud. En esta expereincia también se seleccionó una muestra de ootipos fósiles (*Megaloolithus*) y actuales (*Caiman crocodilus* y *Struthio camelus*) representando distintas condiciones ambientales de enterramiento. El análisis geoquimico de las cáscaras revela que no existen diferencias significativas entre las variables quimicas de los ootipos fósiles y actuales. Las cascaras mantienen una composición química estable dentro de las presentes condiciones experimentales.

INTRODUCTION

Fossil sauropsid eggs and eggshells have been discovered in a wide spectrum of sediments and environments: fluvial, lacustrine, near-shore, and eolian (Hirsch & Quinn, 1990; Sanz *et al.*, 1995; Sahni *et al.*, 94, Norell *et al.*, 95). These palaeoenvironments also determine the burial conditions and provide a set of biases operating on a short temporal span affecting the differential preservation of eggshells, eggs and embryos during early to late fossil diagenesis. Experimental taphonomy, provides a way to understand patterns of egg preservation in the fossil record. Carried out under laboratory or natural conditions, experiments can address questions of taphonomic preservation and bias. Importantly, results might be useful in predicting depositional environments favouring the preservation of embryonic remains.

Experiments that analyse the resistance and degradation of eggshells have shown that they are resistant to breakage during taphonomic transport (Tokaryk & Storer, 1991), and that high level of moisture and bacteria can alter the eggshell structure (Clayburn & Hayward, 1997; Smith & Hayward, 1997). Observations made after an environmental catastrophic event indicate that factors such as behaviour and ecology also influence the preservation of eggs (Hayward *et al.* 1989; 1991). The above-mentioned experiments show that, in fact, early alterations do play a role in preservation. Here we explore the early taphonomic stages involving the decay and biodegradation processes of avian eggs and eggshells by an experiment with controlled environmental conditions. Our initial premise was that the type of soil or sediment, the chemical composition of the water, and the ambient temperature would be decisive variables in the differential preservation of eggs, eggshells and embryos. We expected that different milieus would give rise to distinct taphonomic regimes. Therefore, we simulated particular environmental conditions during egg burial, and analysed a set of quantitative and qualitative taphonomic variables potentially affecting egg preservation or eggshell degradation. We compared preservation biases and environmental parameters, with the aim of answering the question: Are better-preserved eggs associated with particular environments?

Here we describe the alterations that arise in avian egg during decay of organic matter, and explore the morphological and chemical degradation of the eggshell. We also analyse geochemical data from extant and fossil archosaur eggshells in order to assess differing burial environments and their ecological and taphonomic signals.

MATERIAL AND METHODS

Thirteen unfertilised commercial chicken eggs and isolated eggshell fragments were buried in plastic containers, and kept under controlled conditions of light and temperature. Each cage contained one complete egg and the two halves of a single chicken egg plus small pieces of ostrich eggshells. Plastic containers had 3000 ml of capacity. Six of the containers were filled with marl (coming from the fossil site of

Biscarri, Upper Cretaceous, Tremp Basin), six with sand (coming from different localities, Uña, Lower Cretaceous Fm El Collado, and Madrid, Miocene, Arcosas de la Unidad Intermedia). Each container was saturated with fresh tap water (pH 8.52), acidified water (acetic acid in distilled water), sulphated water (distilled water and Na_2SO_4) or seawater (Table 1). The containers were covered but not sealed air tight, and were kept in the dark at room temperature (between 23.4ºC and 26ºC). One additional container was kept in an oven at 37.4ºC. Containers were checked every week and after 40 days eggs were dug up, weighed and photographed. Isolated eggshell fragments were examined using a binocular microscope and samples fragments were glued to aluminium stubs, coated with gold, and examined under a Scanning Electron Microscope (Philips XL30).

Following the examination, eggs and eggshells were reburied and kept under the same conditions for seven months. At this point the eggs were again uncovered, weighed, photographed, and finally broken up to provide eggshell samples. These samples and some isolated chicken and ostrich eggshells were studied by binocular and SEM. The pH and salt content of the sediments were measured at the beginning and at the end of the experience using standard edaphological methods. Eggshells and eggs were taphonomically analysed by means of a set of qualitative and quantitative variables. These variables describe parameters in egg preservation: shape, weight, fractures, orientation and egg content. The alterations of eggshells were described using structural and chemical variables: surface morphology, colour and eggshell chemical composition.

Geochemical analyses were carried out using an electronic microprobe using WDS technique (JEOL, JXA-8900). The electron accelerating voltage applied to analyse the specimens was 15 k V, the electron beam having a diameter between 1 and 5 mm. It was analysed a sample of Megaloolithus siruguei parataxon from two Catalonian localities, Faidella and Biscarri (Late Campanian, Tremp Fm), as well as the chemical composition of the sediment from each site. Extant specimens included *Caiman crocodilus* and *Struthio camelus*. Fossil eggshells were collected at the nesting site of Faidella, and from the matrix that embedded the clutch from Biscarri. The extant eggshells were from farm animals. The sample gathers six fossil eggshells fragments: three from Faidella (FDL1, FDL2, and FDL3) and three from Biscarri (BIS1, BIS2 and BIS3), plus one fragment of ostrich and crocodile.

For the geochemical analysis, eggshell fragments were ultrasonically cleaned. Each sample was analysed taking values from an external, middle, and inner point of the eggshell. Thus, table 2 contains 24 cases in which major and minor elements were measured (major elements expressed as oxides are measured as percentage of the total weight; trace or minor elements in ppm). Chemical elements analysed correspond to Si, Ti, Al, Fe, Mn, Mg, Ca, Na, K, P, F, Cl, and S. Trace or minor elements correspond to Sr, Ba, Cu, Zn, Co, Ni, V, and Cr.

Table 1 includes a brief description of eggs, and eggshells' taphonomic features (external surface and burial states), it also reflects changes in sediment and water lamina both at 40 days, and eight months later of the experiment.

Egg preservation

Taphonomic alterations of the experimental eggs were visible very soon after burial, starting as early as the fourth day. The eggs underwent a number of different qualitative changes during the eight months of burial. The range of notable macroscopic changes included eggs that were dehydrated, empty or holed, burst and with cracks, deformed, reoriented, and encrusted with crystals or pyrite minerals. Significantly, none of the environmental conditions resulted in the refilling of the egg by the embedding material.

A simple model is useful in the discussion of varying taphonomic alterations. In the model (Figure 1) the egg is pictured as a protein pouch enclosed within a permeable, but hard and continuous eggshell. The primary alterations of the egg appear to be based on protein alteration (i.e., denaturation or putrefaction) with an additional role played by the eggshell. The model presents two pathways each with three stages. The initial stage involves the release of organic matter from the egg to the surrounding sediment. Subsequently the taphonomic processes can follow divergent paths. They may lead to "mummified eggs" if the protein is denatured, or they may turn into "rotten eggs" if the organic matter decays and undergoes autolysis. The final stage is characterised by gas release, a by-product of denaturalisation and putrefaction. Gas release results in the cracking and sometimes reorientation of eggs. Below we define and describe the main taphonomic alterations found at each stage.

The release of organic matter. The organic matter flooded the sediment at early stages of egg burial (the first week in sands). Organic matter had the appearance of a blackish liquid with spots of redish and yellow fungi and bacteria dense colonies. The emergence of organic matter was retarded in eggs embedded in marl (after the first month) relative to those in sand. In sand, the organic matter sank to the bottom, whereas in marl the organic matter mixed in the water column or remained at the top of the sediment.

The halos of organic matter could come from either the inner content of the egg, the outer organic layer or colonisation of bacteria and fungi around the egg. We did not analyse the organic matter, to determine its source (e.g. protein composition). The conductivity of the sediment, measured at the beginning and end of the experiment (Table 1) provided a means of measuring the amount of organic matter released. Conductivity decreased in sand and in marl except in those cases with a high salt content, those with either sea or sulphated water. Sediment pHs were also measured (pH 8.01, 8.29 and 8.91 for sand and pH 8.08 for marl) at the beginning and end of the experiment. Although the water was acidified with acetic acid in one group of containers (numbers 1 to 3 and 7 to 9), the final pH in all the cases was alkaline ranging from 9.15 to 7.39 (standard mean 8.25).

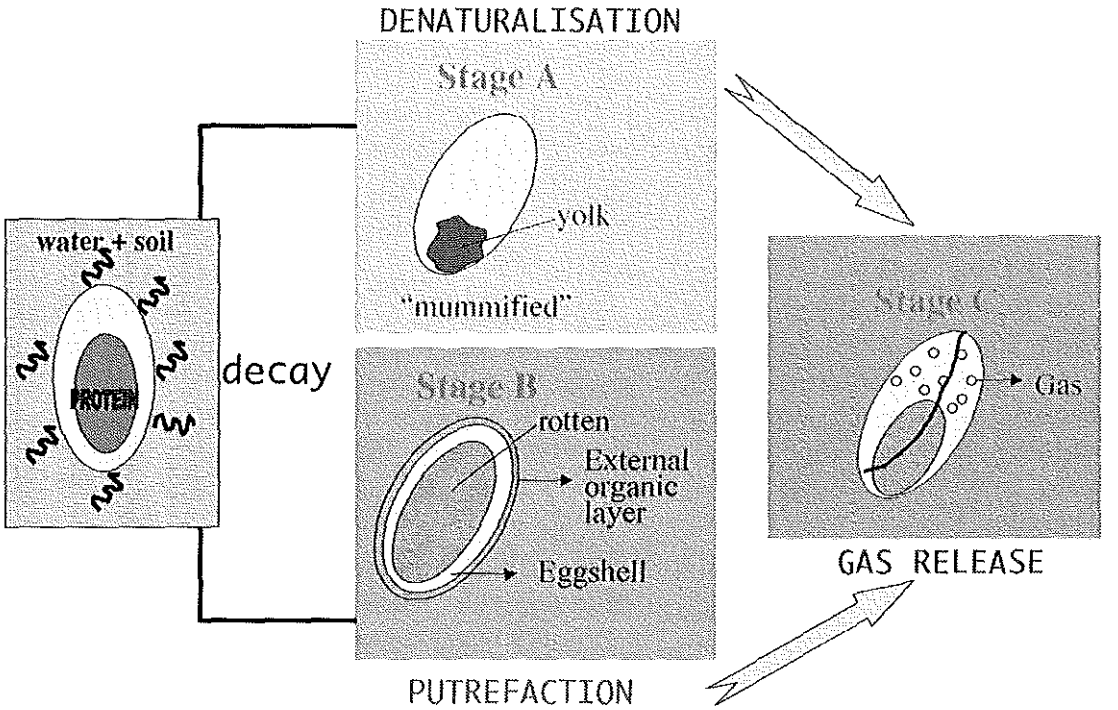


Figure 1.— Schema showing the model of an egg depicted as a protein pouch enclosed in a permeable but continuous hard matrix. Egg alterations are based mainly on protein denaturation with an additional role played by the eggshell. The initial stage involves the release of organic matter and subsequently two different paths can be follow: A- "mummified" eggs (denatured proteins) and B- "rotten eggs" (decay and autolysis). The final stage is characterized by gas release as a by-product of denaturalisation and putrefaction.

Mummified eggs. Mummified eggs are those with preserved membranes (eggshell or vitelline membrane) and that retained the original colour of the yolk. These eggs lack the albumen, resulting in the collapse of yolk and membranes. Yolk protein becomes denatured and dried, thereby taking on a rather solid appearance. Yolk is rich in lipids, proteins, and antioxidant substances. We associate yolk preservation with the alteration of lipids and protein denaturation (known as yolk coagulation or gelation), which inhibits bacterial biodegradation. Mummification resulted from two conditions, either from desiccation, exemplified by the egg placed in the heating chamber (Egg #13) at 37.4 °C, or from those eggs that were placed in containers with acidified water (pH 4.5 – 5) in sand or marl (Eggs #7 and #2). On egg #13, placed at the heating chamber, the sediment dried out earlier than in the remainder. However, eggs #7 and #2 kept the water level along the experience (see Table 1).

Rotten Eggs. Decay and autolysis (i.e. cell breakdown by self produced enzymes) of the eggs' organic content occurred both in sand and marl environments. Eggs were filled with putrid black liquids, but the autolytic process was not studied in detail, since the distinct properties found in rotten eggs (i.e., putrid liquids inside or outside the pouch, or even maintenance of their original appearance) could not be recorded. However, one parameter, egg weight, showed an interesting variability that differed from the expectation of continuous weight loss. Although all the eggs in the sample did

					AFTER 40 DAYS				AFTER 8 MONTHS			
N°	SEDIMENT	SOLUTIONS	PH	COND. (dS/m)	WATER	SEDIMENT	BURIAL	EGG SURFACE	WATER	SEDIMENT	BURIAL	EXTERNAL SURFACE OF THE EGG
1	Sand	Acidified water (pH= 4.5)	8.15	0.37	Present	Black halo under the egg.	Egg partially unburied	Black spots at the surface in contact with the sediment. No fractures.	Present	White coloured at the first centimeter and below an orange coloured layer. Sediment under the egg black coloured	Egg partially buried (approx. 1/2)	Some dark brown spots at the surface in contact with sediment. Pores dark coloured. No fractures.
2	Sand	Acidified water (pH= 5)	8.63	0.15	Present	Black halo under the egg.	Egg partially unburied	Egg with black spots and fractures.	Present	Surface ochre coloured. Yellowish colour at the lateral faces of the recipent. Black sediment surrounding the egg.	Egg partially buried (approx. 1/2)	Area in contact with sediment black coloured. No dark pores. Fractures along the longitudinal axis of the egg.
3	Sand	Acidified water (pH= 6)	8.93	0.11	Present	Black halo under the egg.	Egg partially unburied	Some black spots at the surface in contact with sediment. No fractures.	Absent	Sediment orange coloured (except the surface)	Egg partially buried (approx. 1/2)	Fungi colonies and black pores at the exposed surface (none at the buried surface). No fractures.
4	Sand	Marine water (pH= 7.84)	7.56	2.08	Present	No spots.	Egg buried	No spots. No fractures. Weight= 34 gr.	Present	Sediment black coloured except the outer surface (brown-orange coloured)	Egg on the surface	No spots or dark pores. Fungi colonial mats covering surface. No fractures.
5	Sand	Sulphated water (pH= 5.95)	8.11	0.67	Present	No spots.	Egg partially unburied	Black spot at the surface in contact with the sediment. No fractures.	Present	Sediment black coloured except outer surface and at the sides of the recipent	Egg on the surface	No spots or dark pores. Fungi colonial mats covering surface. No fractures.
6	Sand	Tap water (pH= 8.52)	7.39	0.22	Present	No spots.	Egg buried	No spots. No fractures.	Present	Black and orange extense spots in the sediment	Egg on the surface	Dark brown spots over the whole surface. Dark pores. No fractures.
7	Marls	Acidified water (pH= 4.5)	8.06	1.03	Present	No spots.	Egg buried	Some scattered white spots. No fractures.	Present	Sediment black coloured with some orange and yellow spots near of the egg. (Water also appeared black coloured with white and orange spots	Egg buried	No spots. Dark pores. Egg fractured along the longitudinal axis of the egg (only one fracture).
8	Marls	Acidified water (pH= 5)	8.31	0.30	Present	No spots.	Egg buried	No spots. Black pores. No fractures.	Present	Grey coloured. No spots.	Egg buried	Some scattered and small dark spots at the exposed surface. Dark pores. No fractures.
9	Marls	Acidified water (pH= 6)	8.18	0.82	Present	No spots.	Egg buried	No spots. Black pores. No fractures.	Present	Extense black and orange spots on the surface and the first half of the sediment. (Water black coloured).	Egg buried	Some black spots at the surface in contact with the sediment. Dark pores.Some fractures along the short axis of the egg.
10	Marls	Marine water (pH= 7.84)	8.33	1.25	Present	No spots.	Egg buried	Egg slightly discoloured. No fractures.	Present	Grey coloured. No spots.	Egg buried	Eggshell show some discoloured patches. No dark pores. No fractures.
11	Marls	Sulphated water (pH= 5.95)	8.41	1.71	Present	No spots.	Egg buried	No spots. Black pores. No fractures.	Present	Grey coloured with some black and orange spots near of the egg.	Egg buried	Black spots over the whole surface of the egg. Dark pores. No fractures.
12	Marls	Tap water (pH= 8.52)	8.06	0.80	Present	No spots.	Egg buried	No spots. Black pores. No fractures.	Present	Grey coloured. No spots.	Egg partially buried	No spots. No dark pores. No cracks.
13	Sand	Sulphated water (pH= 5.95)	9.15	0.90	Present	Black halo around the egg.	Egg partially unburied	Fungi colonies on the surface exposed. Black pores and some scattered dark spots. No fractures.	Absent	Homogeneus ochre coloured. No spots.	Egg on the surface	Some dark brown spots at the surface in contact with sediment. Pores dark coloured. No fractures.
	Sand (initial sample)		8.29/ 8.01/ 8.91	0.83								
	Marls (initial sample)		8.08	1.06								

Table 1.— Experimental conditions and values of pH and conductivity of the sediment (initial sample and after 8 months). Taphonomic features of eggs and eggshells as well as changes in sediment and water lamina at 40 days after burying and eight months later (end of the experiment) are also provided.

loose weight, the process was not time-dependent. Rotten eggs were able to maintain their original weight (mean 56 g), while a few lost weight drastically. After eight months, egg #4 in seawater and sand became rotten and lost about 70% of its weight.

Reoriented and Cracked Eggs. Aged eggs release water and carbonic anhydride and show an increase in the size of the air cell (Burley & Vadhera, 1989). Necrokynesis (mechanism of taphonomic alteration that consists in lateral, or downward or upward shifts of elements before definitive burial, Fernández-López, 2000) and fragmentation occurred in association with gas production. Eggs buried in sand behaved differently than those in marl. Early in the experiment, eggs buried in waterlogged sands tended to rise and reorient themselves; some became completely unearthed after a few days (1 to 4). The two cases with retarded flotation occurred in containers with alkaline water and seawater; e.g. egg #13 (kept at 37.4°C with sulphated water) floated after 18 days. Eggs in marl did not become unearthed at any point in the experiment and remained in their original position, although two eggs (#7 and #9) broke as a result of gas expansion. In the first case, a conspicuous cap with a unique fracture was produced along the long axis of the egg; in the latter, the cracking took the form of circular fractures near the poles (Table 1).

Eggshell degradation

The morphological alterations of the eggshell, in isolated eggshell fragments and complete eggs, were analysed at 40 days and eight months later. The most common taphonomic alteration of the eggshell surface was the formation of organic and mineralized crusts and coats.

Isolated eggshell fragments. After 40 days, the only eggshell alteration occurred on the cuticle near pores. At the end of the experiment, pores were well defined and somewhat enlarged. Pores became dark-brown in a similar way as in complete eggs, although the change was less pronounced. In general, the eggshell surface turned discoloured, and with a rough appearance. No bacterial or fungal colonies were observed at the inner surface of the shell. The eggshell membrane appeared to be attached to the inner shell surface even after several months of burial. Calcite degradation was not observed on the inner surface.

Eggshell of complete eggs. The ultrastructure of the eggshell showed no significant changes in eggs embedded either in sand or in marl. At the end of the experiment, the eggshell layers and their ultrastructure were enhanced in a similar way as they were treated with acetic acid but no significant degradation of calcite was seen. Only egg #13 (kept at 37.4° C) showed a loss of clear boundaries between the different eggshell layers. Instead, all layers appeared continuous, although this requires further examination.

Eggshell surface degradation was different in eggs buried in sand compared with those buried in marl. In general, those in sand exhibited more eggshell alterations. Eggshell surfaces had a pale colour and a scaly appearance. Even greater degradation occurred in locations where the calcite had been scratched by the action of fungi and bacteria and began to be altered adquiring a scaly appearance.

In sand immersed in distilled water with acetic acid (#1 – #3), the eggshell areas

in contact with the sediment showed a black coloration after 40 days. Shells in alkaline water retained their original colour. In marl, some eggs that remained buried throughout the whole experiment (see burial section in Table 1) did not turn black in colour (#8 and #10).

The membrane of the eggshell was resistant to degradation, remaining attached to the inner surface in most of the samples. Only in two cases (#5 and #6, eggs buried in sand, and with sulphated and tap water) the calcite got dissolved at the inner mammillae cores (Plate 1 A-B). Calcite mammillae dissolution is a feature associated with reproductive behaviour. The embryo's calcium intake modifies the eggshell structure, producing craters at the base of the mammillae. Similar absorption craters have been described in many fossil eggshells (Hirsch & Quinn, 1990; Carpenter, 1999). Our experimental results show that dissolution processes could produce similar craters (Plate 1 A-B). Further research is needed to establish the true differences between craters produced by biological calcium mobilisation and those produced by erosion (physical or chemical).

Organic and mineral crusts. Eight months later the most remarkable alteration of the eggshell surface of complete eggs was the presence of organic deposits, which eventually formed mineralised coats principally around or inside pores. In the isolated chicken eggshell fragments that were buried in container 6 (filled with alkaline tap water), dendrites of manganese radiated from the pores (Plate 1 C). In the complete chicken eggs the release and breakdown of the organic matter (either from the egg or from the microorganism colonies) produced a boundary around the egg surface. This thin coat was also enriched in elements and salts from the water content and sediment: a) sulphate and iron (natural sulphide mineral), or b) sulphur and phosphate, or c) manganese. A clear distinction can be made between eggs buried in marls and those buried in sand.

As early as four days after burial, eggs in sand showed external halos of organic matter. Egg #4 (in seawater and sand) showed a dense black coating composed of a microbial mat (enriched with P, S, and K) with associated fungi (Plate 1 D). Egg #13 (in sand and sulphated water, and kept in the oven) dried out early, and a crust of crystals of sulphur and gypsum had formed on its surface (Plate 1 E-F).

In eggs buried in marl, pores were surrounded and partially filled with bacterial colonies, and fungal hyphae. In marls, pyrite formation took place in several containers (#8, #9, #11 and #12) waterlogged with either acidified or salty water. The deposits of iron sulphide minerals had different compositions (as pyrite precursors and pyrrhotite). In container #8 a crystal was found (16.2% S and 27.84% Fe) in an enriched area of Fe, Si, and Ca around the pores. Mineral iron sulphite aggregates had a similar morphotype to that described by Clark & Lutz (1980) in the shells of living bivalves. This type of aggregate, observed in eggs #9 and #12 (Plate 1 G-H), consisted of tiny, loosely organised flakes of about 1 micrometer in diameter.

Pyrite formation was confined to the external surface of eggs. No pyrite was found on the inner membranes, mammillary cores, and interstices. Thus, the sulphate reduction of the organic matter took place outside of the eggs in a microenvironment that enveloped the whole egg and in an aqueous alkaline milieu, which was poorly

oxygenated in the case of marl (see discussion).

Geochemical composition of eggshell

To characterise differences in the degree of chemical preservation of the eggshells we compared two samples of Megaloolithus siruguei parataxon from the sites of Faidella and Biscarri (Fm Tremp, Late Campanian) with eggshells of two extant archosaurs: *Caiman crocodilus* and *Struthio camelus*. Fossil eggshells show no significant gain or loss of chemical elements when compared with extant ootypes (Figure 2). With the exception of the Sr, the geochemical analysis revealed no significant differences in the amounts of major and trace elements between fossil and extant eggshells (Figure 2 and Table 2). The increase in the percentage of Sr in fossil eggshells is directly related with the substitution of Ca during fossildiagenesis.

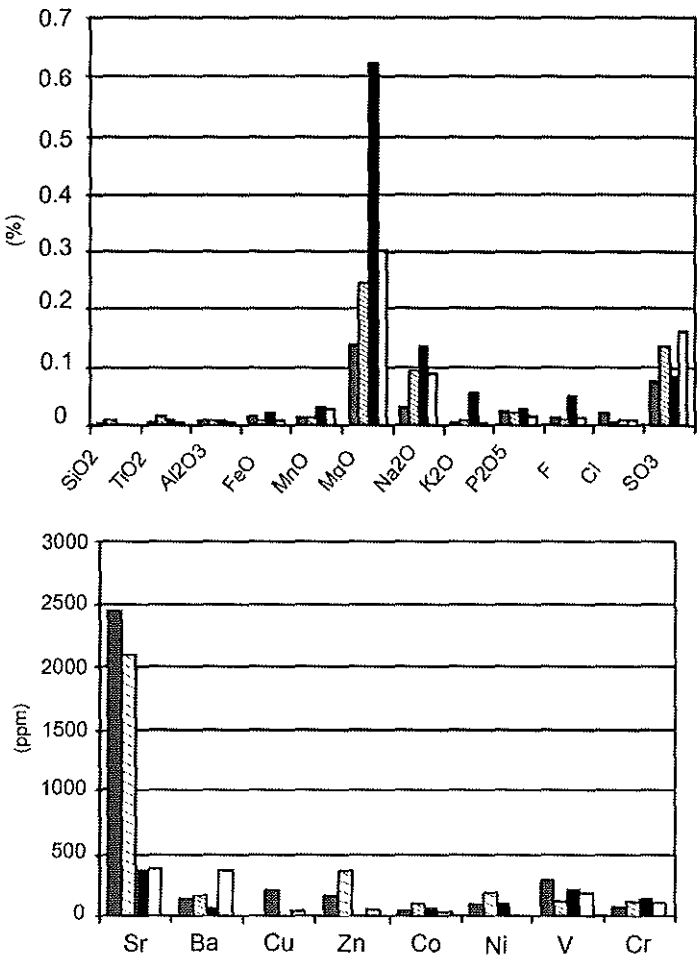
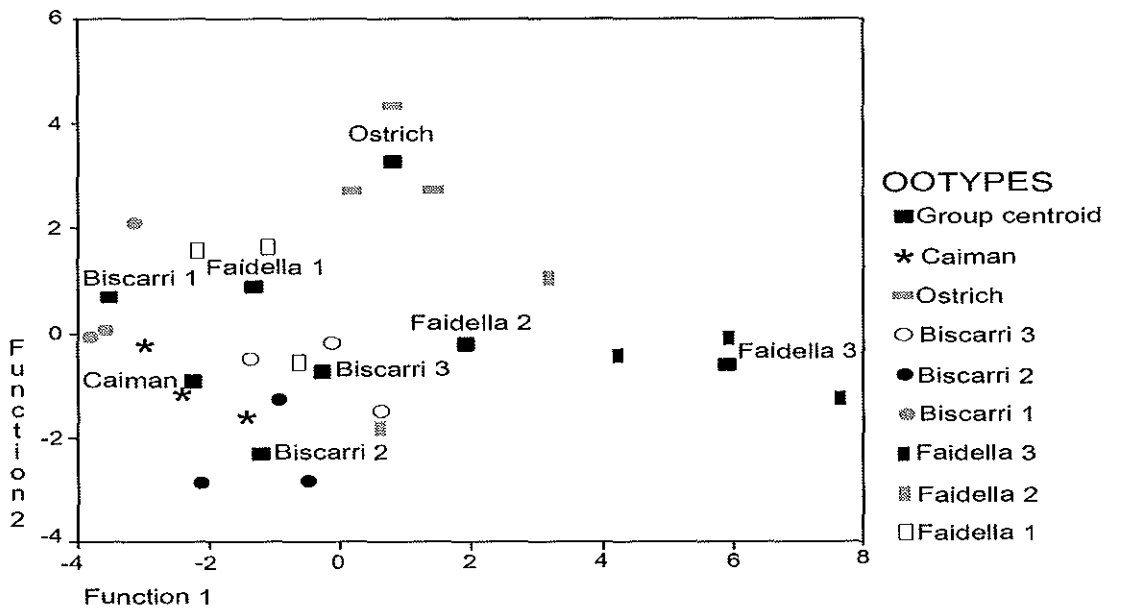


Figure 2.— Bar representation of geochemical data. Bars show the major elements (above) as a percentage of total weight, and traces elements (below) in ppm. The sample is composed of Megaloolithus siruguei from Faidella (grey), Biscarri (striped), and by two extant archosaurs: *Struthio camelus* (black), and *Caiman crocodilus* (white). With the exception of Sr there is no significant change in the amount of the elements in fossil eggshells.

To compare the chemical content of ootypes it was also applied a discriminant analysis (SPSS, version 10.0). The objective was to detect a set of chemical variables able to discriminate: a) fossil and extant ootypes, or b) environmental parameters related with burial conditions. We used discriminant analysis to identify the strongest correlation between variables using all the cases collected in Table 2. The identification of correlated variables prompts the definition of discriminant functions as a way to classify groups (see Figure 3). The Wilk's Lambda test hypothesis for discriminant functions shows that the means listed for the functions are equally across groups, that is, without significant group values. The classification power for the chemical variables according with the prior probability test for groups stresses that a specimen is equally likely to be a member of any group.



Structural matrix

Function

	1	2	3	4	5	6	7
Na_2O	-0.143	0.135	*.415	0.316	-0.119	-0.057	-0.211
SO_3	-0.16	-0.282	*.355	-0.001	-0.212	0.255	-0.22
CaO	-0.102	0.095	-0.073	*-.535	0.293	0.056	0.379
Cl	0.121	0.035	-0.091	*-.496	-0.109	0.132	0.025
Al_2O_3	0.092	-0.039	0.045	0.257	*.453	0.251	-0.076
SiO_2	0.022	-0.04	0.176	0.113	*.296	-0.178	-0.156
MnO	-0.024	0.222	0.028	0.45	-0.073	*.577	-0.036
FeO	0.099	0.119	-0.011	0.158	0.177	*.222	0.084
K_2O	0.016	0.408	0.286	0.1	0.017	-0.042	*-.711
TiO_2	-0.006	-0.178	0.477	0.083	0.108	-0.19	*.675
FeO	0.059	0.215	0.122	0.11	-0.109	-0.126	*-.632
P_2O_5	0.038	0.371	0.286	0.117	-0.086	-0.004	*-.589
MgO	-0.01	0.171	0.188	0.097	-0.046	0.053	*-.557

Wilks' Lambda

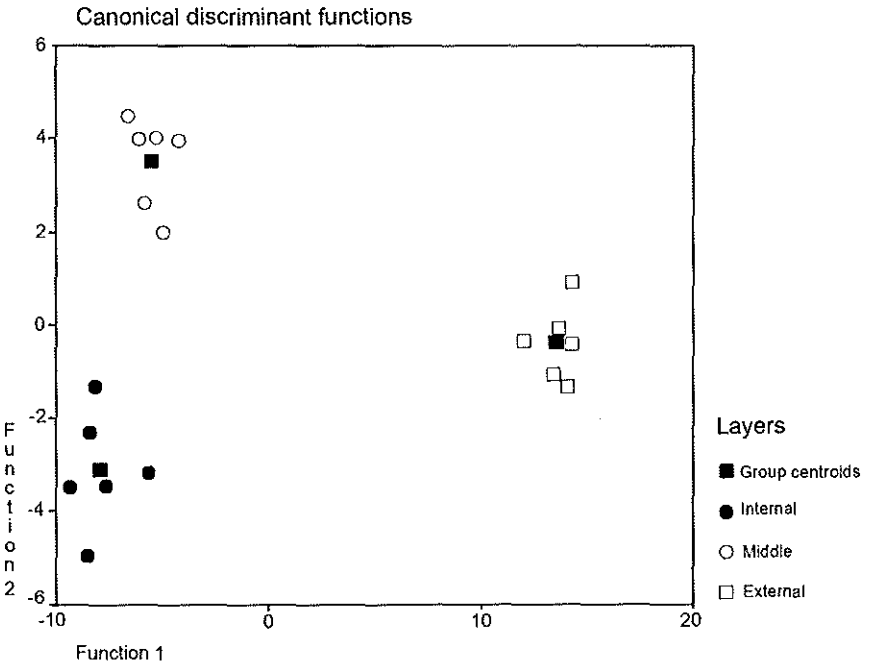
Test of functions	Wilks' Lambda	Chi-square	df	Sig.
1 through 7	0.001	92.334	91	0.441
2 through 7	0.008	60.958	72	0.82
3 through 7	0.035	42.003	55	0.901
4 through 7	0.116	26.966	40	0.943
5 through 7	0.29	15.49	27	0.962
6 through 7	0.554	7.38	16	0.965
7	0.842	2.147	7	0.951

Figure 3.— Factorial discriminant analyses and structural matrix. Ootypes are from caiman, ostrich and *Megaloolithus siruguei* from Faidella and Biscarri. Ootypes show no significant difference when Function 1 is plotted against Function 2. Asterisk in structural matrix represents the strongest correlation between each variable and any discriminant function. The strongest correlation of Na and S occurs with function 3 (see also text). Seven functions have bee used in the discriminant analysis. The Wilk's Lambda table represents the test of functions. Significance < 0.10 indicates that group means differ.

SAMPLE	SIO2	TIO2	AL2O3	FEO	MNO	MGO	CAO	NA2O	K2O	P2O5	F	CL	SO3	SR	BA	CU	ZN	CO	NI	V	CR
FDL1E	0	0,014	0	0,012	0	0,098	60,29	0,07	0,001	0,048	0,009	0,044	0,147	2714,4	0	0	281,2	86,5	0	0	0
FDL1M	0	0	0	0,008	0,021	0,169	53,846	0,059	0,023	0,027	0	0,023	0,077	2528,3	0	0	0	149,4	172,9	314,8	239,5
FDL1I	0,002	0	0	0	0	0,374	53,383	0	0	0	0	0,004	0,092	2452,2	0	24	466	0	0	267,6	0
FDL2E	0,019	0	0,046	0	0,021	0,109	54,106	0,03	0,017	0,005	0,016	0	0,042	2071,7	0	0	216,9	0	0	0	0
FDL2M	0,002	0	0,011	0,02	0	0,163	53,592	0,043	0	0	0,022	0,018	0,114	2841,2	53,7	0	0	0	15,7	165,3	47,9
FDL2I	0	0	0	0,039	0,028	0,444	52,536	0,006	0,006	0,04	0	0,019	0,025	4185,7	241,8	0	0	0	0	149,5	0
FDL3E	0,01	0,023	0,011	0,035	0,024	0,063	50,638	0,016	0	0,047	0,037	0,002	0,086	668	0	0	0	0	0	204,3	0
FDL3M	0	0	0	0,005	0	0,174	52,837	0,005	0	0	0	0,023	0,055	297,1	349,3	0	514,2	0	298,6	487,9	88,9
FDL3I	0	0	0,013	0,007	0	0,28	52,698	0,03	0,004	0,021	0,02	0,037	0,029	2519,9	546,4	0	0	0	0	543	328,4
BIS1E	0,002	0,01	0	0	0	0,018	54,44	0,074	0,006	0,003	0	0	0,021	431,2	53,7	0	8	173	0	102,3	75,3
BIS1M	0,008	0	0,005	0,016	0,041	0,223	53,204	0,112	0,007	0,012	0,016	0,01	0,149	1987,2	385,1	495,4	144,6	101,1	0	0	0
BIS1I	0	0	0	0	0	0,368	53,301	0,051	0,004	0,004	0,01	0	0,074	2088,6	0	175,8	0	0	94,3	31,5	369,5
BIS2E	0,024	0,023	0	0	0	0,163	52,418	0,127	0,014	0,034	0,026	0,009	0,226	3982,8	367,2	719,1	0	275,3	0	133,8	136,8
BIS2M	0,004	0	0,019	0,003	0	0,227	53,301	0,046	0,006	0,004	0,006	0	0,097	2071,7	313,5	0	538,3	0	267,2	0	0
BIS2I	0	0,042	0	0	0	0,523	53,977	0,124	0	0,023	0	0,016	0,231	2240,8	0	0	674,9	47,2	243,6	511,5	390
BIS3E	0,02	0,007	0	0,048	0,031	0,096	52,933	0,042	0,011	0,016	0	0,01	0,149	3004,9	0	0	491,1	0	0	212,5	0
BIS3M	0	0,039	0,026	0	0	0,187	53,005	0,115	0	0,008	0	0	0,105	794,9	421	399,5	1406,1	250,5	110	102,3	0
BIS3I	0,003	0,022	0,02	0	0,041	0,386	52,894	0,137	0	0,027	0	0	0,156	2190,1	0	151,8	0	0	0	0	61,6
HAE	0	0,005	0,013	0,007	0,021	1,566	51,495	0,25	0,116	0,61	0,142	0,014	0,169	710,3	107,5	0	0	23,6	275	31,5	0
HAM	0	0,008	0	0,009	0,031	0,132	52,245	0,058	0,018	0,069	0,009	0,012	0,046	219,8	35,8	0	0	0	0	275,4	75,3
HAI	0,02	0,014	0,014	0,041	0,028	0,165	54,501	0,09	0,034	0,091	0	0	0,042	126,8	35,8	0	0	149,4	0	314,8	328,4
HCE	0	0,008	0,012	0,021	0,041	0,243	52,456	0,103	0	0,023	0	0,01	0,226	600,4	331,4	0	0	0	0	314,8	294,2
HCM	0	0	0	0	0,017	0,22	52,1	0,116	0	0	0	0	0,096	473,5	662,8	98,9	136,6	47,2	0	0	0
HCI	0,001	0	0	0	0,021	0,441	52,343	0,044	0,008	0,026	0,03	0,01	0,157	67,6	71,6	0	0	47,2	0	212,5	41,1
FDL (SE)	23,109	0,142	11,341	1,86	0,065	1,222	27,633	0,108	1,881	0,106	0	0	0,026	1057	0	151,8	0	0	0	181	13,7
BIS (SE)	12,665	0,118	6,836	0,657	0,017	0,768	34,406	0,101	0,595	0,042	0	0,007	0,087	1445,9	179,1	0	0	306,7	0	802,7	0

Table 2.— Geochemical results of major elements (oxydes, % of total weight), and trace elements (ppm) in fossil and extant eggshells. Abbreviations: FDL, Faidella; BIS, Biscarri; HA, Ostrich; HC, Crocodile; SE, Sediment. Fossils are composed by three samples (1, 2 and 3), and the values correspond to E, external, M, middle and I, internal layers. Note that ostrich has an extremely high value of Mg in its outer layer.

Function 1 and 2 show no significant correlations between any major elements to sort out differences between extant and fossil archosaurian eggshells (ostrich, crocodile, Faidella 1, 2, 3, and Biscarri 1, 2, 3). Instead, variables strongly and significantly correlated occur for other functions (see structural matrix functions 3 to 7, Figure 3) discriminating in such cases particular samples. For instance, function 3 discriminates, due to their Na and S content, the group composed by Biscarri 2, Biscarri 3, plus ostrich (see structural matrix in Figure 3). None of the groups defined by these discriminant functions was coherent. For instance, in the group above mentioned BIS1 is excluded from the remainder Biscarri samples.



Structural matrix

Function

	1	2
SiO_2	*.124	0.073
F	*.055	0.007
FeO	*.034	0.021
CaO	*.033	0.02
Cl	*-.008	-0.003
MgO	-0.272	*.528
P_2O_5	0.04	*.218
TiO_2	0.015	*.088
K_2O	0.049	*-.085
Al_2O_3	0.014	*-.073
Na_2O	0.001	*-.027
MnO	0.006	*.024
SO_3	0.011	*.019

Figure 4.— Discriminant analysis by eggshell layer (internal, middle and external) of *Megaloolithus siruguei* eggshells from Faidella and Biscarri sites. Internal and medial layers are characterised by magnesium content, while external layers are characterised by silica (note the structural matrix for other elements involved in the functions). Two functions have been used in the discriminant analysis.

Discriminant analysis was effective surveying the chemical composition by layers (external, middle, and internal). Eggshell layers can be distinguished by their content. Using only the fossil sample (Faidella 1, 2, and 3 plus Biscarri, 1, 2 and 3), function 1 discriminates between external and internal plus middle layers (Figure 4). The external layer is characterised mainly by silica values. Function 2 is characterised mainly by the magnesium content, discriminating between middle and internal layers. The greater silica content of the external layer might be due to the terrigenous deposits adhered to the eggshell surface, while magnesium is one of the inorganic constituents of eggshells. The sampled dinosaur, Megaloolithus siruguei concentrates the magnesium content at the inner layer. Extant avian eggshells posses also a differential distribution of Mg, e.g., in Galliformes magnesium is concentrated in the mammillary layer and in the outer part of the shell, whereas in other avian orders (including the ratites) it concentrates only in the mammillary layer (Board & Love, 1980).

DISCUSSION

Biologically speaking, the sauropsid egg is a complete and discrete female reproductive cell. To fulfil their function, eggs contain the genetic and epigenetic information and the chemical nutrients needed for embryogenesis and growth. These processes occur within the protective biomineralised shell that is permeable to gases and water (Burley & Vadhera, 1989).

The main constituents of an egg are a variety of proteins with vastly different properties, sulphydril groups, and lipids. These are located predominantly in the yolk, although a small amount has been found in the eggshell. Trace elements such as iron (in the albumen and yolk) and manganese (in the whole egg) are also present. Some of the proteins are arranged in densely packed layers, giving rise to organised membranes such as the eggshell membrane and the vitelline membrane.

The mechanisms of taphonomic alteration acted very early, and depended principally upon the nature and composition of the egg. That is, necrokynesis (vertical displacements), distortion and fragmentation, encrustation and mummification depend on organic matter decay and protein denaturation. However, environmental variables fine-tune these processes. The experiment detected possible environmental conditions that favour or actively promote these taphonomic alterations.

Necrokynesis is due to the release of gas derived from putrefaction, and allows the eggs to unearth themselves when the process occurs in porous saturated sandy sediments. Eggs shifted vertically in waterlogged sands. Therefore, it is probable that in saturated sandy sediments fossil eggs could experience reorientation and unearthing. It might be predicted as well a potential lateral movement accomplished with transport. In such a case those eggs would be allochthonous.

On the other hand, gas release can produce the breakage of the egg. Large neat cracks were only produced in marl; fragmentation is, once again, influenced by the nature of the sediment. Large openings present in many dinosaur eggs have been

interpreted as hatching windows (Cousin *et al.*, 1994). In a recent work, Mueller-Töwe *et al.* (2002) considered hatching windows to be those openings that cover at least 45% of the egg area. In contrast, our experiments show that such large openings can be produced by gas expansion arising from the decay of organic matter.

Yolk mummification seems to be a rather frequent event (in 3/13 of the cases, 23%). Once the yolk has collapsed and coagulated it may become mineralised. Possible fossilised yolk has been reported in elongated eggs from the Late Cretaceous of China (Carpenter, 1999). Furthermore, mummification is affected by environmental factors, requiring either dry conditions (heat) or acid environment during the early phase of diagenesis. Mummification in sub aqueous conditions is neat in marls, where egg #7 never unearthed. Mummification in sand could happen likely in sub aerial conditions, because eggs (#2 and 13) floated during the first 40 days (Table 1).

Encrustation consists of the addition of minerals onto the eggshell surface and the filling of the pores. Floated and dried eggs in sand, where organic matter had sunk to the bottom, provide the ideal conditions for forming crystal crusts of gypsum and sulphur (rich elements in the water). Conversely, pyritisation takes place only in marl. Encrustation in marls is related with the release of organic matter.

In our experiment, we expected pyrite to form on the inner side of the eggshell next to the source of sulphide production during egg putrefaction. Iron support may even come from egg content, enriched in this mineral (Burley & Vadehra, 1989). In Catalonia has been described dinosaur eggs with framboidal pyrite encrusting the inner side of the shell (López-Martinez *et al.*, 2000). There is no detailed description of the position of pyrite on this dinosaur egg, and the presence of pyrite was associated with root colonisation following egg burial. Similar conditions prevailed in Pleistocene bones of equids and bovids (Pfretzschner, 2000), whereby a pyrite coat covered the lining of the marrow cavity and traveculae. Pyrite is produced during the first phase of bone diagenesis, combining the decay of collagen and iron that was freed from the haemoglobin in the bone marrow. However, we ruled out any profuse diffusion of organic matter from inside the egg since no pore canal was stained or coloured by putrid liquids, and the inner layers had no organic or inorganic coats. Also the maintenance of weight strengths this observation.

Although the origin of the organic matter around eggs was not analysed, we argue that the major source of organic matter is the decomposition of the outer organic layer of avian eggs, and probably from microorganisms. It is reasonable to suppose that the most reliable mechanism involved in pyritisation of eggs is that mentioned by Canfield and Raiswell (1991), as "pyritisation of organic matter". An analogue of shell pyritisation occurs in living mollusc shells in salt marsh sediments (Clark and Lutz, 1980). In sediments with iron (0.66%), alkaline water and with a shell surface enriched in metabolisable organic matter (formed by the outer organic layer to which fungi and bacteria adhered) iron sulphides formed that plugged the pores.

Our results indicate that fossil and extant eggshells posses a highly stable chemical composition. Dinosaur eggshells and the extant crocodile form a cluster, showing equality of means for the chemical variables. In addition, the cluster of fossil and extant eggshells also implies that the ecological signal in the chemical composition of these eggshells is feeble. Extant and fossil eggshells represent three environmental

conditions. The environmental interpretation of Biscarri indicates the presence of water-saturated sediments in a reducing coastal environment (López-Martínez *et al.*, 2000) whereas Faidella has been interpreted as fluvial or deltaic floodplain deposits (Bravo *et* al., 2000). Ostrich built nests in dry soils far from water. Caiman eggs come from a farm where eggs are removed from the sand and placed in a heating chamber. Irrespective of the burial conditions (coastal, lacustrine, or terrestrial) there are no significant differences in major and trace elements. Dauphin (1990, 1991a, b, 1992) raises the same considerations, demonstrating the difficulty of using the chemical composition of dinosaur eggshells to reconstruct palaeoenvironments. She used a large dataset from the Lower Rognacien of the Aix-en-Provence Basin. The ecological signal in the eggshell chemical composition is not strong.

Isolated eggshell fragments have fewer taphonomic alterations and less variability than complete eggs. Previous experiments carried out on eggshell fragments showed resistance to breakage and degradation during lateral transport (Tokaryk and Storer, 1991). Once dissociated from the whole egg, shells are resistant calcitic elements, and eggshell fragments tend to show rather similar and homogenous alterations independent of the burial environment (i.e., sediment, nature and temperature of water).

The experiment has yielded a description of the earliest diagenetic changes. Similar changes have also been reported in dinosaur eggs by Mueller-Töwe *et al.* (2002). The authors described variations in dinosaur eggs (e.g., coloured shells, deformed eggs, large windows or cracks, yolk preservation, and reoriented eggs) that can be explained within the taphonomic alterations described in this study. We propose that systematic experimental taphonomy will lead to improve taphonomic and palaeobiological interpretations of the amniote fossil record. Further research should be done, clarifying the mechanisms involved in these taphonomic alterations. Furthermore, if we wish to obtain a better understanding of the fossil preservation and bias, more detailed taphonomic data must be acquired during fossil extraction fieldwork.

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PLATE 1

Scanning electron photographs of eggshell surfaces and deposits.

A-B: inner surface of egg #5 (sand and sulphated water). Left, the eggshell membrane is lost and craters appear at the centre of mammillae cores. Note rests of membrane fibres running between the cores. Right, detail of a mammilla showing dissolved calcite crystals.

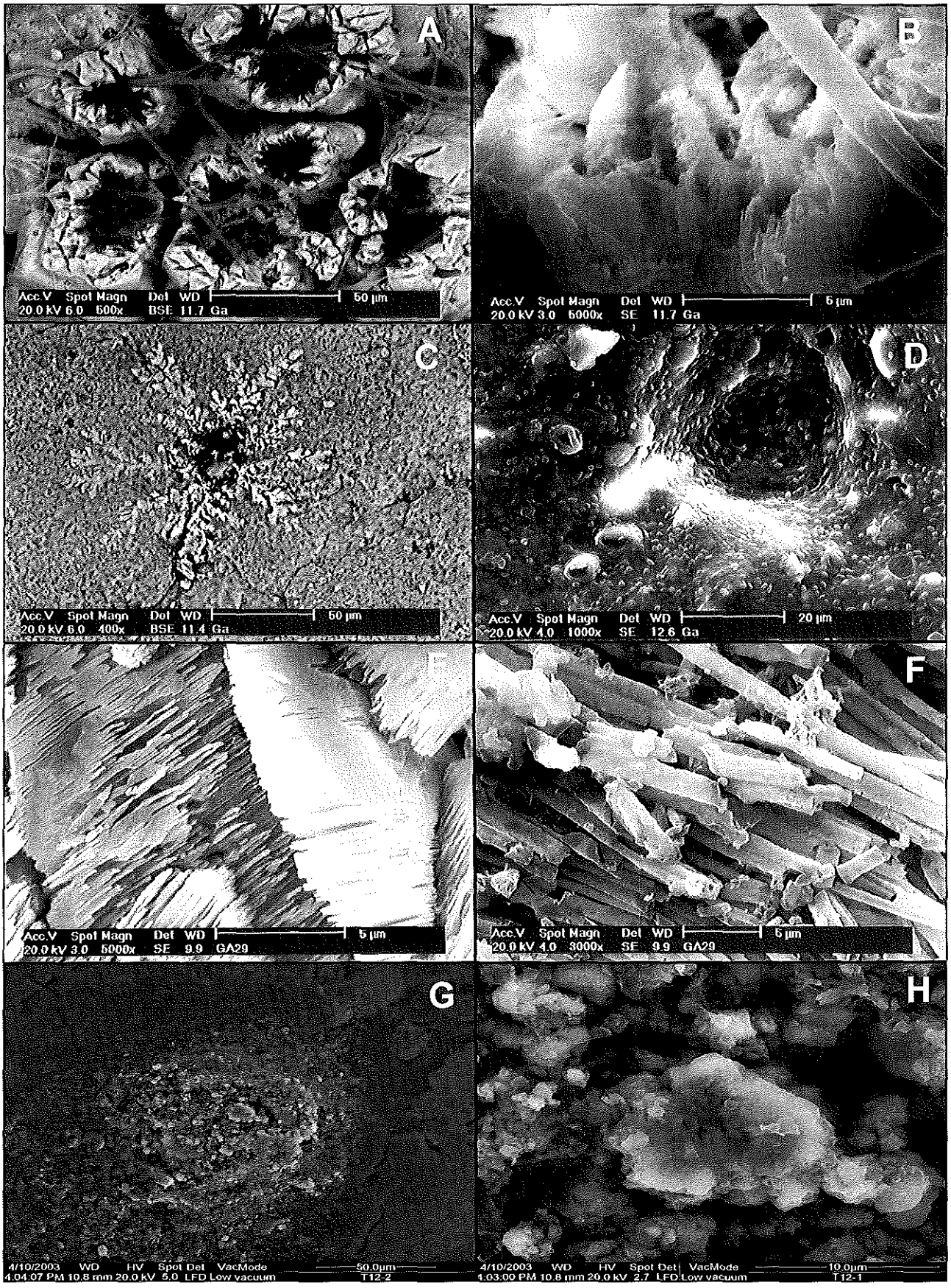
C: manganese dendrites radiating from a pore in a fragment of hen eggshell. Photograph corresponds to egg #6 (container with sand and tap water).

D: microbial mat with fungi associated (see arrow) around and filling a pore of hen eggshell. Photograph corresponds to egg # 4 (in sand and marine water).

E-F: Encrustation of crystals of gypsum (left), and sulphur crystals (right), both detected at the surface of egg #13. It corresponds to the container filled with sand and sulphated water kept in the heating chamber at 37.4°C.

G-H: Aggregate of iron sulphide formed by loose flakes filling a pore of egg #12 (container filled with marl and tap water).

PLATE 1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20.0 kV 3.0 6000x SE 11.7 Ga
C
Acc.V Spot Magn Det WD 50 µm
20.0 kV 6.0 400x BSE 11.4 Ga
D
Acc.V Spot Magn Det WD 20 µm
20.0 kV 4.0 1000x SE 12.6 Ga
E
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F
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20.0 kV 4.0 3000x SE 9.9 GA29
G
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H
4/10/2003 WD HV Spot Det VacMode 10.0µm
4:03:00 PM 10.8 mm 20.0 kV 2.7 LFD Low vacuum T12-1