

DETERIORATION OF ROCK-TOMBS, SAQQARA AREA, EGYPT*

ABSTRACT

The present work focuses mainly on the deterioration of the mother rock (wall painting support) of rock –tombs at Saqqara area, e.g. Idut tomb; Old Kingdom, 6th Dynasty, which have been hewn in the Upper Eocene (Maadi Formation) rocks.

Petrographic characteristic and mineralogical composition of the mother rock and the plaster ground layer (preparatory layer) are carried out using polarizing microscope, X-ray diffraction (XRD) and differential thermal (DTA) analysis. Cross-Section of the mother rock, plaster layer and the painted layer are also investigated.

The examined samples are mainly marlstones and argillaceous limestones. They are related into two main microfacies associations, fossiliferous micrite (mudston) and clayey biopelsparite (bioclastic pelloidal grainstone), respectively

Rock texture, mineralogical composition and diagenetic process contribute, to great extent, the deterioration recognized by the mother rock and the plaster preparatory layer. The rocks of the mother rock are too weak to resist the interaction between exogenic (climate) and the endogenic (related to the nature of the rock types) conditions since their construction, thousands years ago.

The durability of the mother rocks is mainly conditioned by their clay content and their porosity

INTRODUCTION

Saqqara area as known is a plateau formed mainly of well-stratified yellowish, argillaceous limestone, marl and calcareous claystones, with higher content of gypsum veinlets, and in part highly fractured with some cavities. They are related to lower part of the Maadi Formation, early Late Eocene ⁽¹⁾. The plateau shows a cover of Quaternary gravel, sand and conglomerate of varying thickness. In this vast Egyptologic area many rock –tombs, mostly related to the Old Kingdom, have been hewn within the Upper Eocene rocks. The walls of these tombs (mother rock) are cased by plaster ground layer (preparatory layer), on which the ancient paintings had been carried out. Most of those tombs show many signs of decaying.

The most important stage of any stone conservation is the estimation of the nature of stones, their deterioration, and the causes of the decay. If the chemical, mineralogical and petrographical properties of the stones are neglected and not taken into consideration, the choice of the intervention technique may be

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inappropriate and consequence for the appearance or integrity of the stone may be very damaging. If the caused of the decay are not evaluated and corrective measures taken to get rid of them, successful intervention may in fact be only a temporary palliative.

The present work focuses mainly on the causes of the deterioration of some rock-tombs, e.g. Idut tomb; Old Kingdom, 6th Dynasty; from the point of view of investigation the petrographic characteristics and the mineralogical composition of the mother rock (wall painting support) and the plaster layer. This may help in the estimation of the nature of the stones and their future conservation.

OCCURRENCE

The Idut tomb and its shaft and burial chamber, the main subject of the present work, was hewn within a local hill situated on the southern part of the enclosure wall of Zoser complex (Fig.1A&B). It was discovered by Firth (1927)². According to Firth⁽²⁾, the tomb is considered as mastaba related to the Early 6th Dynasty (2420-2280 B.C.); it was originally built for Ikhy. But, later on it is used as a tomb by a lady named Idut, a royal daughter or princess⁽³⁾.

The study tomb suffered from extensive deterioration of the mural paintings especially the paintings in the burial chamber (Figs.2-4). Such deterioration has been accelerated rapidly since its discovery. Thus, could be detected through comparing the recently observed deterioration (Figs.5-7, photos which have taken two years ago) with that recorded in 1935⁽³⁾. A glance to these photos, one can deduce that large areas of the plaster together with the painted layer have been fallen down from its wall support.

The examined samples have been collected from inside the tomb, from the walls of burial chamber, which represent the mother rock and from the plaster ground layer (preparatory layer). The lithologic characters of the rocks that formed the mother rock (walls of the tomb) could be seen through an exposed face; lies to the back of the Idut tomb and at the entrance of the southern Zoser tomb. This exposed face represents rhythmic succession, as it composes of three rhythmities, each consists of, well stratified (Fig.8), four beds showing continual repeated changes of lithology, but having different thickness. The upper bed of each rhythmite is formed of yellowish, to green, shale with high content of gypsum (0.40-0.75m). This followed downward by Grey, semi-hard argillaceous limestone (0.65-1.0m), then by greenish, soft, thinly laminated marl (1.0-1.5m) and then by sandy, calcareous, massive and nodular claystone (0.3—0.45m). Fractures (Fig.9), cavities and the gypsum veinlets are the main diagnostic features observed.

METHODOLOGY

Thin sections representing the mother rock and the plaster layer are petrographically examined using polarizing microscope. The carbonate ones are stained with Alizarin red-S⁽⁴⁾ to reveal the presence of calcite and dolomite.

The studied samples are subjected to X- ray diffraction analysis to determine their mineralogical composition. The powder diffraction patterns of the samples

are obtained using Cu $k\alpha$ radiation and Ni filter. The scanning speed is $2\theta=1$ degree/min. at constant voltage 40kV, and 30mA using PW 1390 X-ray diffractometer.

Both bulk samples and the clay fractions separated from the marlstones and argillaceous limestones were X-rayed. Oriented aggregates of the clay particles (<2 μ m) are mounted on glass slides by allowing drops of clay suspension, separated by sedimentation dry up on the slides⁽⁵⁾. Three specimens of each sample are subjected to X-ray analysis, one is used as it is, the second is treated with ethylene glycol and the third is heated to 550°C for two hours. The identification of the minerals is carried out using the data given in the ASTM cards by measuring the d-values of the diffraction planes and their relative intensities. Semi-quantitative estimation of the recorded clay minerals was achieved, through the X-ray diffraction of the glycolated oriented clay fraction mounts, following the procedure given by Shultz (1964)⁶ and Pierce & Siegel (1969)⁷.

Acid – insoluble residue, AIR, is determined by treating the samples by dilute, 10%, HCl. The residue was thoroughly washed by distilled water, dried, weighed and its percentages are calculated. Then, they were examined using the binocular microscope⁽⁸⁾.

To prepare cross- sections, a suitable part of sample containing all the layers, is embedded in a transparent resin at room temperature for two days. Then ground on grinding machine using silicon carbide paper (1000-1200). Then the cross-section is examined by using a Leith Microscope at magnification of 200-400 times.

RESULTS

PETROGRAPHY

Mother rock

The rocks compose the mother rock of the Rock-Tombs are related mainly into two lithotopes, argillaceous limestones and marlstones. The determined AIR of argillaceous limestone range from 14-19%, whereas in the marlstones ranges from 39-53%. Binocular microscope examination for the obtained AIR revealed that clay, subordinated by sand in addition to few amount of gypsum are the dominant acid- insoluble constituents. AIR of argillaceous limestone composed of 75-90% clay, 5-9% sand and 3-5% gypsum. On the other hand, AIR of marlstones formed of 77-84% clay, 8-13% and 6–9% gypsum.

Petrographic analysis revealed that the carbonate component, forming the argillaceous limestone of the mother rock, are related to same microfacies association, biopelsparite⁽⁹⁾ or bioclastic pelloidal grainstone⁽¹⁰⁾. Two types of allochemes built up the framework, peloids and bioclastic with various proportions (Fig. 10). The bioclastic constitute (15-25%) of the rock volume and are formed of mollusc fragments, echinoid debris, benthic and planktic foraminifera together with algae. Some of fossil chambers are recrystallised into and/ or filled with sparite, but mostly have been leached out. Peloids (20-35%)

composed of structureless micrite, ranging in size from 20-110 μm and are rounded, ovoid, well-sorted and organic rich (Fig.11). They are formed due to fecal piling by certain organisms such as molluscs and worms. The allochems are embedded in sparitic matrix that is generally subtranslucent with faint brownish cast in thin section. Pore spaces (both inter- and intraskeletal) are abundant (average 13%) having various size and shapes (Figs.12&13). They may be developed due to post-depositional diagenetic dissolution processes. The non-carbonates are represented by fine nodules of clays and fine detrital quartz grains (~8%). Few fine, idetopic, illzoned dolomite crystals (30-55 μm) are observed in some thin sections.

The marl lithotypes have higher percentage of terrigenous material represented by clays that admixed with the micritic matrix. The latter composed of very fine microcrystalline carbonate, which is difficult to distinguish from the terrigenous materials. These rocks can be tentatively classified as fossiliferous micrite⁽⁹⁾(Fig.14) or mudstone⁽¹⁰⁾, where they contain <10% fossil fragments scattered in the groundmass. Some of these fragments are leached or replaced by recrystallized sparry calcite. They have many micro-cracks that some of them were filled with sparite.

Plaster layer

The plaster layer (preparatory layer) was executed directly onto the mother rock with varying thickness (2-4mm), over which a finer painting (0.025mm) layer has been laid, as indicated from the cross-sections investigation (Fig.15). The plaster layer was used to treat the irregularity and smooth the surface of the bed rock (support).

Microscopically, the plaster layer is texturally heterogeneous, as the groundmass consists mainly of fine-grained gypsum in which coarse gypsum crystals are widely distributed (Fig.16). These crystals are occasionally dehydrated into anhydrite; characterized by higher relief and stronger birefringence. Voids (Fig.17) having different sizes and shapes with sharp outlines, occasionally delineated by fine gypsum grains, are additionally present. Some fines to medium quartz grains are also observed. Patches of iron oxides, hematite, occur as staining materials having red to blood-red colour.

MINERALOGY

Plaster layer

X-ray diffraction pattern of plaster layer (Fig.18) indicates that gypsum is the main component with little amount of anhydrite and rare quartz. Gypsum is investigated by its main peaks, occurring at d-values 7.63 Å, 4.28 Å and 3.06 Å (ASTM card No.33-0311). On the other hand, the peaks at 3.49 Å, 2.84 Å, 2.32 Å and 2.20 Å (ASTM card No.6-0226) identify anhydrite.

The differential thermal analysis, DTA of the plaster layer is shown in Fig.19. The DTA curves show the presence of two large endothermic peaks, one small endothermic peak and one small exothermic peak. The first large endothermic peak occurs at 141°C, implying the formation of hemihydrate phase⁽¹¹⁾. The

weigh loss corresponding to this peak as revealed from TGA curve is about 12.04%, that represent about 75% of the total weigh loss (~ 16%) of the sample; i.e. about 1.5 moles of the combined H₂O). The second large endothermic effect occurs at 180°C, indicate a dehydration of hemihydrate to soluble anhydrite and loss of the remaining 0.5 mole of H₂O. Such total loss (~ 16%) implies that the plaster layer is not formed of pure gypsum (total weigh loss ~ 20%) but contains an amount of anhydrite; corroborating the results obtained from the petrographic studies as well as the X-ray analysis. The very small endothermic effect occurring at 240°C may be attributed to the loss of the last traces of water held in the hemihydrate structure ⁽¹²⁾. The small exothermic effect occurring at 365°C is may be related to the transformation from soluble to insoluble anhydrite.

Mother rock

X-ray analysis of the mother rock indicated, that calcite and quartz are the main non-clay minerals recorded in all samples (Fig.20). Whereas, halite, gypsum, orthoclase and microcline are revealed in some samples.

The mineral assemblages identified within the clay fractions (Figs.21a,b&22, as an examples) include smectite, kaolinite and illite, in order of decreasing abundance. Smectite is identified by its moderately sharp peak occurring at 14 Å of the untreated clay fraction which shifts to ~18 Å (with increase in intensity) and ~10Å upon glycolation and heating, respectively (i.e. Ca- montmorillonite). Kaolinite is characterized by its basal reflections at d-spacing 7.13Å and 3.58 Å. These reflections are not affected by glycolation, while disappeared upon heating treatment. Illite is identified by a peak exists at 10.04 Å, which neither affected by heating nor glycolation treatments.

The semi-quantitative estimation of the recorded clays implies that the montmorillonite (the expandable clay mineral) is the predominant, ranging from 61%- 70% with an average 66%. Kaolinite is the second in abundance ranges from 23%-31% and averaging 26%, whereas illite is 7% in average.

DISCUSSION AND CONCLUSION

The petrography and mineralogical characters of the rock used to construct the rock-tombs at Saqqara area contribute to great extend in their deterioration. Those rocks are too weak to resist the interaction between exogenic (climate, include rain water, moisture, humidity, and temperature variation) and the endogenic (related to the nature of the rock types) conditions, since their construction, thousands years ago. These agents of deterioration often acted in conjunction and often potentiate one another ⁽¹³⁾.

The preceding results indicate that the limestones of the mother rock are composed of grainstones with relatively low content of mud (~12% in the AIR) as compared with the marlstones. Grainstones (sparites) are sufficiently permeable and porous facies that facilitate fluids to inter within the rock and act on their component ^{(14)&(15)}. Moreover, the rocks had been suffered from high diagenetic process, dissolution process. It is believed that dissolution was mainly by the action of circulating meteoric in an active zone ^{(16)&(17)}. Dissolution resulted in the

development of inter-and intraskeletal pores through removal of peloids, shell fragments and / or leaching of their cores (Fig.12). This process also produced irregular voids and vugs (Fig.13) with various size and shapes or caverns (observed in the field). Such features might be led to drastic increase in the rock porosity ^{(17)&(18)}.

Such porous rocks with high porosity and high pore connectivity favours the diffusion of rain water and humidity and help in water sorption, increasing the capillary pressure inside the rock and finally giving rise to its disintegration ⁽¹⁹⁾. It is also evident that the higher the rock porosity is in the rock, the higher the tendency to disaggregate is ⁽¹⁹⁾.

It is worth mentioning that, in Saqqara area, humidity fluctuated from 100%- 34% in the early morning and in the afternoon, respectively in winter and from 88% - 26% in the morning and in the afternoon, respectively in the summer ⁽²⁰⁾. On the other hand the determined moisture content of the argillaceous limestone samples ranges from 0.9-1.6% and ranges from 1.7-2.1% in the marlstones, that is in accordance with the results given by Soliman (1998) ²⁰, indicating a relatively higher percentage.

The mother rocks (both marlstones and argillaceous limestones) contain a considerable quantity of clays, but with highly expanding clay minerals. Presence of higher percentage of the expandable clay (Ca-montmorillonite, average 66%), as revealed from the mineralogical investigation, indicates a destructive phenomenon. It has the ability to expand under the action of water or high humidity and may be caused a lot of decaying.

It is known that the expandable clays have different amount of water due to the negative charge and the concomitant presence of exchangeable cations. These cations tend to form hydrates through attracting water molecules, whenever some water is present. Ca and Mg ions retain H₂O more strongly (VanRanst, 1993) ²¹. According to VanRanst (op. cit.), at every temperature, equilibrium exists between the amount of water found in the interlayer space and the atmospheric water (relative humidity). As the relative humidity increase, the mineral will absorb water out of the air until a new equilibrium is reached. The swelling of the expandable clay minerals by water uptake is due to osmotic pressure. Smectites (especially Ca- montmorillonite) can take much more water, as they have variable basal spacing. They swell, giving a volume, which may be several times the volume of the dry clay, depending on the amount of water available. In dry conditions, most of the adsorbed water is lost through evaporation, causing a strong shrinking.

As it can be presumed, successive cycles of swelling / shrinkage, favoured by humidity fluctuations may disintegrate or severely damage the stones. Moreover, during the swelling process, the cohesion of the clay particles may decrease resulting in a very low shear strength of rock materials; the clay particles slide easily over each other and cause a lot of damage. As a consequence, the plaster layer has been collapsed and fallen down.

It is well known that, evaporites (gypsum, salt and other water-soluble salts) are originally present in the Upper Eocene limestone used for monument construction. It is mainly primary, or secondary due to the drainage ⁽²⁰⁾. The studied rocks have a considerable amount of gypsum and halite, as indicated from X-ray. Presence of salts has a bad effect. Salt bearing solution migrates in the stone, and under suitable occasion water evaporates and salts deposits. They are either disfiguring the surface or when expanding during crystallization, cause damages and disruptions ⁽²²⁾. Moreover, crypto-efflorescence of these salts in-between the mother rock and the plaster layer may be caused the detachment of the plaster and painting layers.

Petrographic studies of the plaster layer reveal that anhydrite may be developed due to dehydration of gypsum. Dehydration process leads to net reduction in the bulk density and the resulted anhydrite is being to be more porous than its compact original gypsum ^{(23)&(24)}. Furthermore, rupture deformation may be originated due to volume decrease during transformation. Such process may also contribute in the detachment of the plaster layer.

Treatment

The traditional methods of conservation can not applied for the studied tomb, which has suffered from severe conditions of deterioration that affected the wall painting support (mother rock). In such severe conditions, the remaining paintings must be detached from its support ⁽¹³⁾. Consequently, for the Idut tomb restoration, a stacco method should be used, using a new support of synthetic materials with intervention layer (cf. Shoeib, 1997) ²⁵. In this case, the fallen parts of the mural painting as well as the remaining parts of paintings, which must be detached from its harmful support, collected together and fixed on a new synthetic support..

REFERENCES

- (1) Youssef, M., Cherif, O., Boukhary, M. and Mohammed, A., Geological studies on the Sakkara area, Egypt. N. Jb. Geol. Palaont. Abh., 1984, **168/1**, 125.
- (2) Firth, G. M., Annals du service des Antiquities, t. xxvii, 1927, 107.
- (3) Macramallah, R., Le Mastaba D' Idut. Imprimerie De L institute Francais D archaeologie orientale, le Caire, 1935, 3.
- (4) Dickson, J.A.D., Carbonate identification and genesis as revealed by staining. J. Sed. Petrol., 1966, **36**, 491.
- (5) Tucker, M. E., Techniques in sedimentology. Blackwell Scientific Publications, Oxford, 1988, 391 p.
- (6) Shultz, L. G., Quantitative interpretation of mineralogical composition from X-ray and chemical data for the Pierre Shale. U. S. Geol. Surv. Prof. Paper, 1964, 31 p.
- (7) Pierce, J. W. and Siegel, F. R., quantification in clay mineral studies of sediments and sedimentary rocks. J. Sed. Petrol, 1969, **39**, 187.

- (8) Ireland, H.A., Insoluble residue; in: Procedure in sedimentary petrology, R. E. Carver, ed., Wiley Intersciences, London, 653p.
- (9) Folk, R.L., Practical petrography classification of limestones. A.A.P.G. Bull. ., 1959,**43**, 1.
- (10) Dunham, R.J, Classification of carbonate rocks according to depositional texture. A.A.P.G. Mem. 1: 1962 , 108.
- (11) Flek, W. P., Jones, M. H., Kuntze, R. A. and McAdie, H. c., DTA of natural and synthetic hydrates of calcium sulphate. Cand. J. Chem. 1960, **38**, 836.
- (12) Kuntze, R. A., DTA of Ca SO₄. 2H₂O. Nature, 1962, **193**, 772.
- (13) Mora, P., Mora, L. and Philiopt, P., Conservation of wall paintings. IccRom, Rome, 1984, 494p.
- (14) Sperber, C. M., Wilkinson, B. H. and Peacer, D. R., Rock composition, dolomite stoichiometry and rock/water reaction in dolomitic carbonate rocks. J. Geol., 1984, **92**, 609.
- (15) Dawans, J. M. and Swart, P. K., Textural and geochemical alteration in Late Cenozoic Bahamian dolomites. Sedimentology, 1988, **35**, 358.
- (16) Longman, M. W., Carbonate diagenetic textures from nearshore diagenetic environments. Bull. Am. Ass. petrol. Geol. 1980, **47**, 461.
- (17) Tucker, M. E. and Wright, V. P., Carbonate sedimentology. Blackwell Scientific Publications, Oxford, 2001, 482 p.
- (18) Abu-Zeid, M. M., Petrography, clay mineralogy, and diagenesis of the subsurface Lower Tertiary rocks. Egypt. J. Geol. 1989, **33**, 47.
- (19) Espert, R. M., Ordaz, J., Alonso, F. G. and Alba, J. M., Petrographic and physical study of the building stones from Leon Cathedral (Spain): 285-298, in Rossi-Manaresi,R.(ed),The Conservation of Stone II , Proceeding of the International Symposium, Bologna, October 27-30, 1981, 520p.
- (20) Soliman, M. S., Spheroidal exfoliation in sedimentary rock monuments-Sixth approach to environmental GeoEgyptology. Sedimentology of Egypt, 1998, **6**, 1.
- (21) VanRanst, E. (1993): Clay mineralogy, Lecture note. Inter. Training Centre for Post-graduate Soil Sci., Ghent Univ., Belgium, 122 p.
- (22) Jewaka, H. J., Physico-chemical factors in the conservation of stone: 195-204 in Rossi-Manaresi,R.(ed),The Conservation of Stone II, Proceeding of the International Symposium, Bologna, October 27-30, 1981, 520p.

- (23) Goldman, M. I., Deformation, metamorphism and mineralization in gypsum- anhydrite cap-rock, Sulphur Salt Dome, Louisiana. Geol. Soc. America Mem.50, 1952, 169 p.
- (24) Akarish, A. I. M., Geological and mineralogical studies on some gypsum-anhydrite deposits of west Sinai and their assessment. M. Sc. Thesis Fac. Sci., Ain Shams Univ.1985, 195p.
- (25) Shoeib, A. S. Application of poly-urethane as an intervention layer for a new support in transferred wall painting. Annul meeting of the Association of conservators, Torun, 1997.

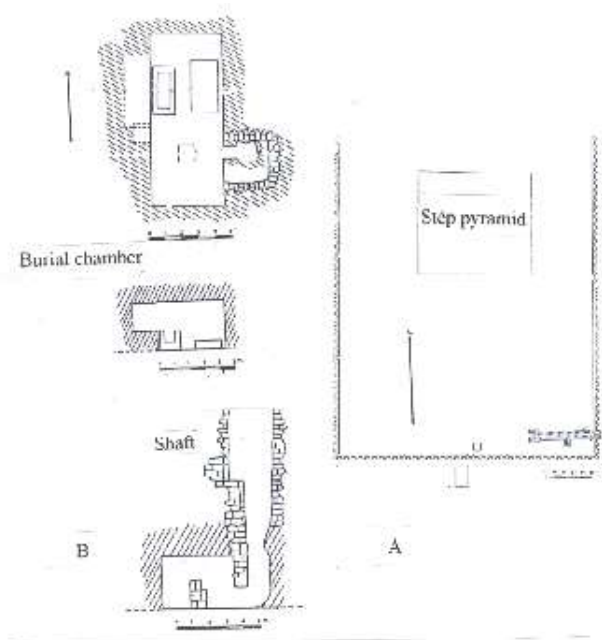


Fig.1: A, is the location of the tomb, B, show its craft and burial chamber.

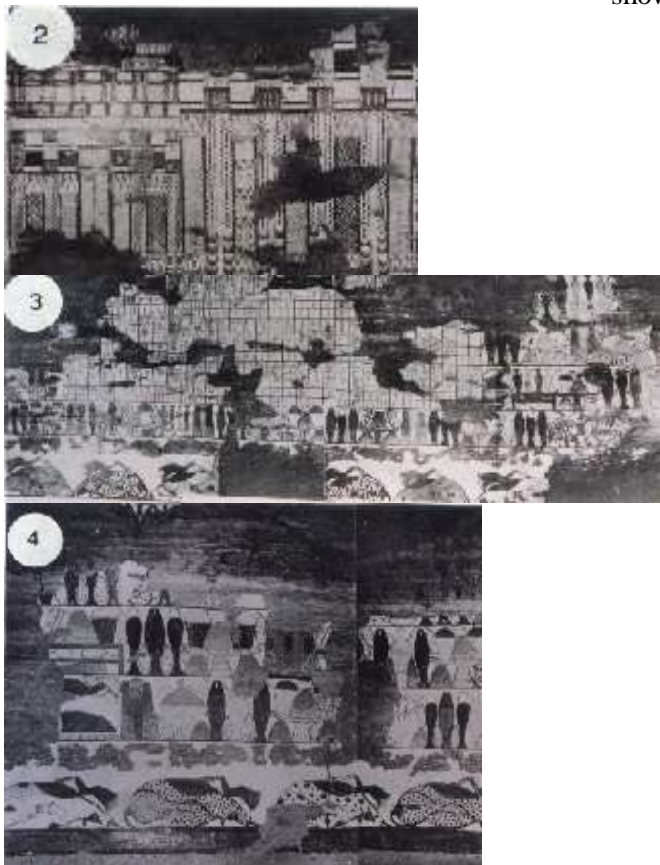


Fig.2-4: Deterioration exhibited by the studied tomb during its discovery.



Fig.5-7: Recently, more advanced deterioration, as large areas of painting have been fallen down.



Fig.8: Well stratified Upper Eocene rocks.



Fig.9: Vertical fracture cutting through well stratified beds.

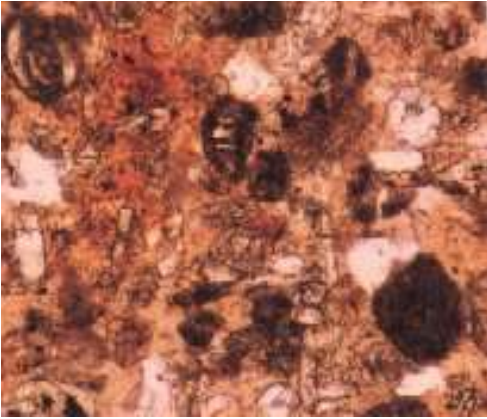


Fig.10: Biopelsparite,
argillaceous

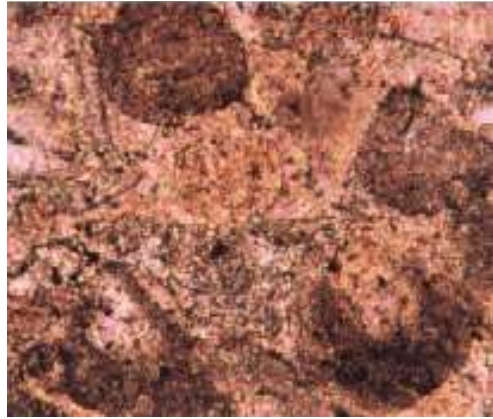


Fig.11: Rounded peloids
embedded in sparite
matrix, P.P.L. (X70).

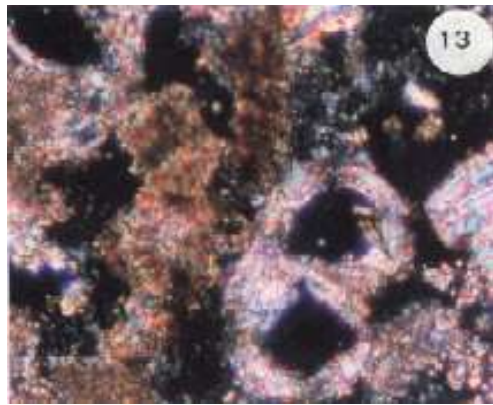
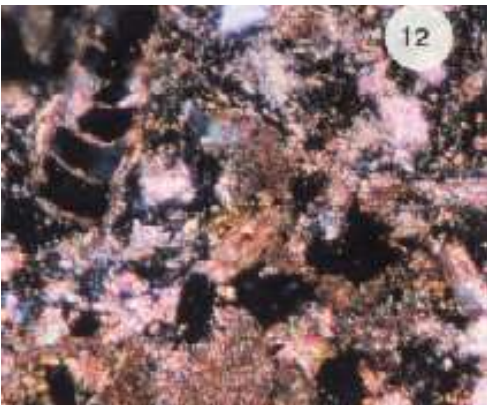


Fig.12&13: Pore spaces (both inter- and intraskeletal), having various
size and shapes, biopelsparite facies, (X70).



Fig.14: Clayey fossiliferous micrite, marlstone facies, P.P.L. (X70).

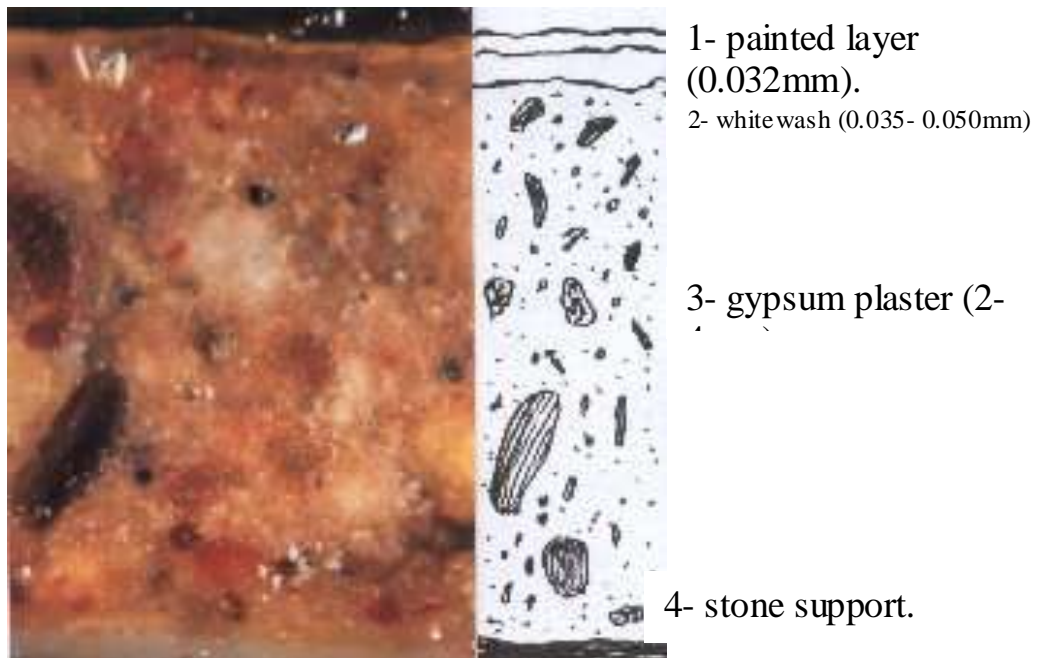


Fig.15: Cross-Section of Idut tomb mural painting.

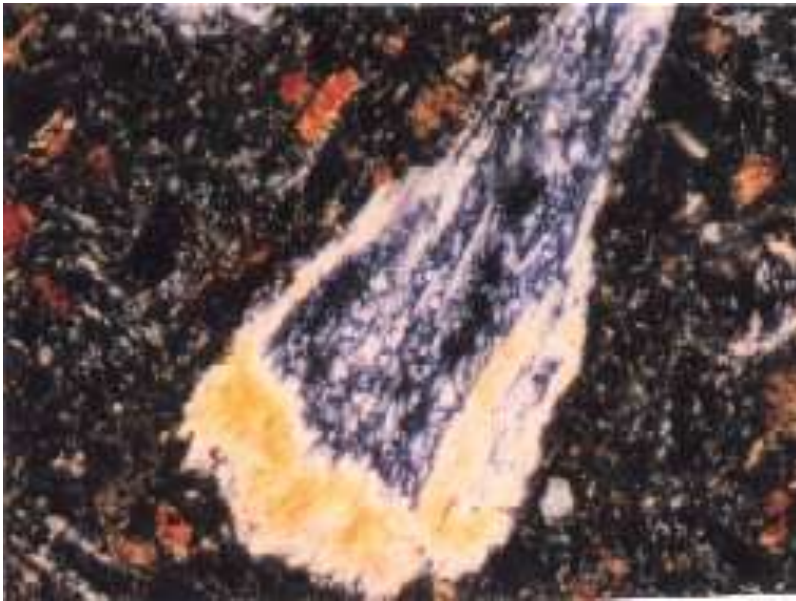


Fig.16: Coarse gypsum crystals (partly dehydrated) in fine grained gypsum groundmass, plaster layer, C.N. (X70).

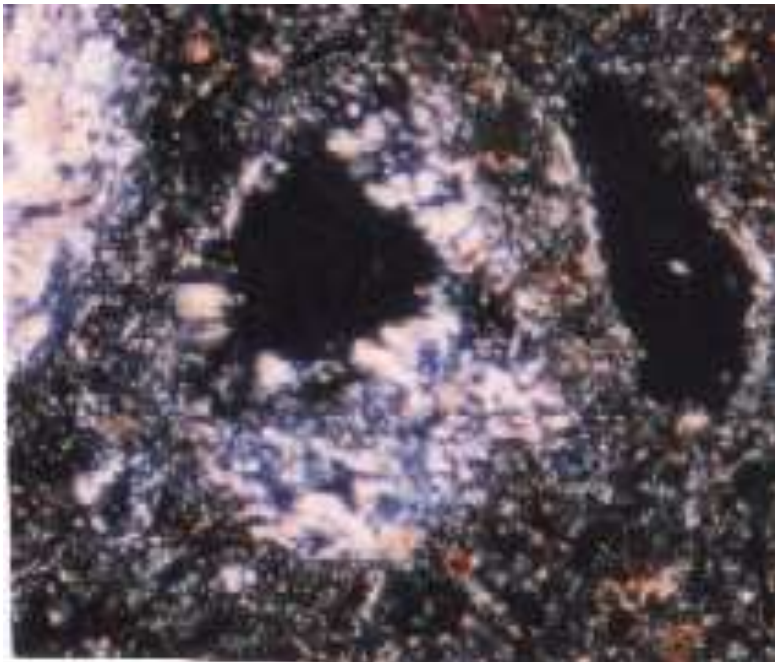


Fig.17: Well developed pores in plaster layer. C.N. (X70).

