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EXAMINING FOUR RESINS AS STONE SURFACE CONSOLIDANTS FOR CONSTRUCTION SANDSTONE OF ARCHAEOLOGICAL SITES, KARNAK TEMPLE, UPPER EGYPT, CASE STUDY

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ABSTRACT :

The sandstone is reported to be used for construction of temples, Churches and walls every-where in the world e.g. some parts of Temple of Karnak in Upper Egypt, Qasr El-Sagha and Karanis Temples in El-Fayum City in Lower Egypt, as well as St. John Church and Chester City Walls of Roman age in UK. These archaeological sites suffer weathering from low grade represented by stone surface discoloration to severe weathering noted as intense disintegration of construction rock.

The four resins namely; Methylmethacrylate Co-polymer; polyvinyl butyral; OH (tetraethox-ysilane); and H (ethyltrimethoxy silane) have been compared together and evaluated as stone surface consolidants based on the following basis :

1.Resin penetration depth, 2.Keeping stone surface colour and breathability, 3. Progressing rock petrophysical and mechanical properties, and 4. increasing rock durability to acids and salts.

Polyvinyl butyral at 10% and 15% level of concentration verifies the best results as it penetrates within the stone up to 21 mm, keep stone surface colour and breathability, keep stone surface dry, progress rock petrophysical properties as it reduces rock porosity from 15% down to 1.1%, rock effective porosity from 13.2% down to 1.0%, rock water absorption from 7.72% down to 0.54%, and increases rock strength from 50.4 Kg/cm^2 up to 193.9 Kg / cm^2 . Also the samples consolidated with either 5%, 10% or 15% polyvinyl butyral experienced the highest durability class (to salts and acids) indicated by lowest weight loss% after 15 cycles of each test. So, this resin is highly recom-mended.

Polyvinyl butyral is easy to be applied on large scale and it is not expensive as much as reconstruction process. It can give good results if the stone under consolidation is well prepared as its cleaning from rock meal and dust as well as salt efflorescence before consolidation.

The present work aims to examine four resins to be used as stone surface consolidants in par-ticular for the slightly weathered rock to withstand with the present day environmental conditions e.g. salt weathering, acid rain, and wetting and drying. Sandstone samples have been collected from Temple of Karnak (Pharaonic episode, Upper Egypt) to run the study on them.

INTRODUCTION :

Karnak temple has been mostly built from sandstone in addition to limestone at some

parts. It suffers weathering by salts in such arid climate (Ismaiel & El-Habaak, 1995 and Selim, 2001).

Temple of Karnak require urgent remedy to survive for so long. The slightly weathered parts in this archaeological site (Fig. 1) require consolidation, so, four resins namely; Methyl-methacrylate Co-Polymer, Polyvinyl butyral; OH (tetraethoxysilane), and H (ethyltrimethoxysilane) have been examined in this current work on some sandstone samples collected from this site. This is to find out which resin verify good results on exposure to acids and salts as that dominate in Egypt.

The resins' selection has been based on previous literature in this field of study (Hawkins, 1972; Subbaraman, 1980; Gauri, 1990; Ismaiel and El-Habaak, 1995 and Marketa and Kotl, 2001). The most suitable resin is that verify the following four items, namely; (1) not to change stone surface colour, (2) not to react with stone components forming new harmful components e.g. siliconates are reported to react with iron content in ferrigenous sandstone making stone to be rusty (Bell and Coulthard, 1990), (3) increase stone surface hardness and durability to weathering processes, (4) verify greatest penetration depth within the weathered stone and keeping rock breathability.

The resins that are suggested to be examined are: Methylmenthacrylate Co-polymer, Polyvinyl butyral, OH (tetraethoxysilane), and H (ethyltrimethoxysilane). They have been prepared at limited concentrations to be examined and evaluated from four points of view, namely;

1-Examining stone surface colour change on rock exposure to ultra violet for 48 hours after its treatment with each resin.

2-Measuring penetration depth of each resin using Scanning Electron Microscope to compare between these resins based on their penetration depth.

3-Examining and comparing rock petrophysical and mechanical properties before and after treatment with each resin.

4-Examining rock durability before and after treatment with each resin and comparing between them i.e. comparing between all of the treated samples together on one hand, and between the weathered treated and weathered untreated samples on the other hand to detect which resin verify the highest rock durability (i.e. the lowest weight loss%).

METHODOLOGY :

Rock samples have been collected from temple of Karnak and treated with the resins mentioned above. Their petrophysical (represented by rock porosity, effective porosity, bulk density and water absorption) and mechanical properties (represented by unconfined compressive strength) have been examined before and after treatment to clarify the impact of each resin on rock strength and absorption to soluble materials. The penetration depth of each resin has been measured at right angle to the treated surface using Scanning Electron Microscope. The weight loss of stone samples has been computed after 15 cycles of each of soundness (salt durability) and simulation (acid durability) tests applied on rock samples before and after treatment with each resin, then rock durability class has been determined using Barry (1991) diagram (Fig. 2). This is to find out the impact of resins on rock durability to weathering processes at study area.

The following represent the limits of concentration of each resin that has been prepared for this study :

Methylmethacrylate Co-polymer: It has been diluted in solvent (mix of 50 : 50 acetone : IMS, where IMS is methylated spirits) to get the required limits of concentrtion. It is examined for rock samples code R5, R6, R7 and R8 at the following limits of concentration: Pure methylmethacrylate Co-polymer (i.e. without solvent); 5%; 10%; and 15% respectively.

Polyvinyl butyral (= Mowital B30H) : It is diluted in solvent of 50 : 50 acetone : IMS, where IMS is methylated spirits. It has been examined for samples code R2, R3 and R4 at three limits of concentration 5%, 10% and 15% respectively.

OH (tetraethoxysilane) : It is used as spray i.e. used without dilution in solvent where there is no solvent for this resin. It is used for sample code R9.

H (ethyltrimethoxysilane) : It is used without dilution. The rock sample code R10 is treated with this resin.

While samples of code R1 are of the same rock type as the other Rx (where x is any number given above e.g. R6, R7) but at dry and untreated with any resin. This is to compare all measurements (e.g. petrophysical, mechanical properties and durability) before and after treatment with the resins under investigation.

RESULTS :

The change of stone surface colour after treatment with each resin; the penetration depth of each resin at its limits of concentration; the petrophysical and mechanical results of the sandstone samples before and after treatment with each resin; and rock durability before and after treatment are given below.

Stone Surface Colour :

The first point to be examined for these resins is the change of stone surface colour after consolidation because if the surface colour is altered after consolidation, the resin is preferred to be excluded from the rest of the experiment (Abd El-Hady and Kamh, 2001). The resins under consideration don't show any change in stone surface colour after treatment. Also, the stone surface become dry quickly (during 24 hours of treatment), so, it doesn't give chance for air dust or pollutants to stick on the treated surface.

Penetration Depth :

Scanning electron microscope has been used to measure penetration depth of these resins at their levels of concentration. Table (1) illustrates the resin's name, its concentration, code of rock sample that indicate the methodology of the sample treatment as explained in Section (1.2) and the penetration depth of the resin.

The differentiation between the image of treated and untreated sample i.e. the view of resin under scanning electron microscope can be noted in Figures 3,4,5,6 and 7 that also show the view of penetration depth of some of the examined resins.

The maximum penetration depth has been recorded for samples treated with polyvinyl butyral (Fig. 4). H (ethyltrimethoxysilane) also present a noticeable penetration depth (Fig. 7). Methylmethacrylate Co-polymer and OH (tetraethoxysilane) present very negligible penetration depth (Figs. 5 and 6) that might be a result of withdraw of the resin back to stone surface and resin decomposition on drying and exposure to sub - aerial conditions (Warke and Smith, 1998).

Fig. (1) : Slightly weathered part of Karnak Temple, Upper Egypt.

Fig. (3): Scanning electron micrograph showing weathered texture of untreated sandstone sample collected from Temple of Karnak, Rock sample code R1

Fig. (2) : Barry diagram for determining rock durability class:
N.B.(a) : Rock durability class after 15 cycles of soundness test (Na_2SO_4 salt).
(b) : Rock durability class after 15 cycles of soundness test (CaSO4 salt).
(c) : Rock durability class after 15 cycles of simulation test (H_2SO4 salt).

1-10 is the number given for each of the treated and untreated samples.

Table (1) : Penetration depth of examined resins

Code of rock sample	Resin name and level of concentration	Penetration depth (microns) or (mm)
R1	Unteated sample	-----
R2	5% Polyvinyl butyral	22 mm
R3	10% Polyvinyl butyral	21 mm
R4	15% Polyvinyl butyral	20 mm
R5	Methylmethacrylate Co-polymer	3 microns
R6	5% Methylmethacrylate Co-polymer	2 microns
R7	10% Methylmethacrylate Co-polymer	traces
R8	15% Methylmethacrylate Co-polymer	traces
R9	OH (tetraethoxysilane)	traces
R10	H (ethyltrimethoxysilane)	11 mm

Petrophysical and mechanical properties:

The evaluation of a given resin also depends on the degree of reduction of rock porosity, effective porosity and water absorption, meanwhile increasing rock compressive strength and keeping rock breathability. These properties have been examined for weathered untreated and weathered treated samples taking code R1 - R10, Table (2).

The results listed in table (2) indicate that : Methylmethacrylate Co-polymer (sample code R5-R8) doesn't reflect any progress in these properties and the same for sample R9 that is treated with OH (tetraethoxysilane). This can be noted if these results are compared with that of weathered untreated samples (R1) of the same rock.

Table (2): Average petrophysical and mechanical properties of untreated and treated rock samples

Code of rock sample	Porosity %	Effective porosity %	Water absorption %	Unconfined compressive strength (kg/cm^2)
R1	15.0	13.2	7.72	50.4
R2	1.6	1.4	0.74	98.3
R3	1.1	1.0	0.54	125.1
R4	1.1	1.1	0.56	193.9
R5	14.6	11.5	6.76	48.9
R6	14.5	11.8	6.92	61.9
R7	15.1	11.4	6.69	47.2
R8	14.8	11.1	6.49	47.6
R9	15.2	11.4	6.81	58.5
R10	26.8	16.3	9.94	91.9

Sample (code R10) treated with H (ethylmethoxysilane) shows high retrogradation of rock petrophysical properties, meanwhile it presents a noticeable progress in rock strength. Samples treated with Polyvinyl butyral (code R2-R4) show a high progress in rock petrophysical and mechanical properties. The highest progress is recorded for samples treated with Polyvinyl butyral in particular R3 and R4.

Consolidants and Durability Test :

The consolidated rock samples as well as the untreated samples are exposed to durability tests using salts (Na_2SO_4 and $CaSO_4$) and acids (H_2SO_4) similar to that dominate at study areas. The percentage of weight loss has been computed after fifteen cycles of each test, then rock durability class has been determined using Barry (1991) diagram (Fig. 2). The results of soundness and simulation tests are listed in Table 3.

Table (3): Weight loss, durability class of the treated and untreated rock samples unsing the resins under investigation.

Code of rock sample	R1	R2	R3	R4	R5	R6	R7	R8	R9	R10
Weight loss % after 15 cycles $Na_2 SO_4$	100% after 9 cycles	0.0	0.0	0.0	100% after 11 cycles	100% after 9 cycles	100% after 11 cycles	100% after 11 cycles	100% after 11 cycles	2.22
Durability class	E	A	A	A	E	E	E	E	E	A

Code of rock sample	R1	R2	R3	R4	R5	R6	R7	R8	R9	R10
Weight loss % after 15 cycles $CaSO_4$	100% after 8 cycles	0.4	0.2	0.4	100% after 10 cycles	100% after 10 cycles	100% after 9 cycles	100% after 11 cycles	100% after 10 cycles	3.0
Durability class	E	A	A	A	E	E	E	E	E	B

Code of rock sample	R1	R2	R3	R4	R5	R6	R7	R8	R9	R10
Weight loss % after 15 cycles $H_2 SO_4$	100% after 9 cycles	0.9	1.0	0.82	100% after 9 cycles	100% after 10 cycles	100% after 10 cycles	100% after 11 cycles	100% after 10 cycles	2.9
Durability class	E	A	A	A	E	E	E	E	E	B

From the durability results mentioned above, it can be noted that: the percentage of weight loss of the treated rock samples is less than or sometimes equal to that of the untreated samples (i.e. the resin doesn't figure in any progress in rock durability against to the applied salts or acid as the case of samples code R5-R9) where these samples have 100% weight loss after nine to eleven cycles of any of the applied durability test. These samples (R5-R9) have low durability class "E" that is equal to durability class of the untreated samples (R1). Other resins show a noticeable progress in rock durability that is indicated by very low weight loss and high durability class "A" with all durability tests e.g. samples code R2, R3, R4 and R10 (Table, 3).

Generally, from durability point of view, it can be concluded that samples treated with Polyvinyl butyral shows the highest progress in rock durability followed by those treated with H (ethyltrimethoxysilane). But the rest of the resins indicate low or no progress in rock durability.

DISCUSSION :

The consolidation results using the four resins under investigation indicate that the resins don't experience stone surface colour change and the stone surface treated with them is getting dry within 24 hours of treatment. So, no complain on the resins from this point, but with respect to penetration depth of each of them it is found that those treated with polyvinyl butyral show the greatest penetration depth up to 21 mm depth from stone surface, and this is considered a great penetration depth (Abd El-Hady and Kamh, 2001, and Kamh, 2001), and that treated with H (ethyltrimethoxysilane) present a considerable penetration (11mm from stone surface), while the rest i.e. those treated with Methylmethacrylate Co-polymer (code R5-R8) and that treated with OH (tetraethoxysilane) show a penetration depth measured in few microns or noted as traces on the stone surface.

The sequence of progress in penetration depth of the examined resins is in a great harmony with the sequence of progress in rock petrophysical and mechanical properties and durability test. The samples treated with Polyvinyl butyral shows the highest progress in the rest of points of evaluation of the resins if compared with the same results measured for the untreated or the samples treated with other resins. The rock samples treated with H (ethyltrimethoxysilane) also experience a considerable progress in rock strength (but not in petrophysical properties) where this might result in dissolution of the rock soluble material. So, the rock petrophysical properties are not progressed but are retrograded, meanwhile, it sticks the rock grains resulting in increasing rock strength, and rock durability.

Samples treated with Methylmethacrylate Co-polymer and OH (tetraethoxysilane) don't indicate any progress in rock petrophysical and mechanical properties or rock durability or experience penetration within the treated samples if compared with that treated with other resins or the untreated samples.

CONCLUSION:

Resins' evaluation as stone surface consoldants is an important study to get the most suitable resin that verify the best results in increasing stone durability to the surrounding environmental conditions. The four resins examined in this study have been recommended in similar studies, hence, they are examined to get the most suitable one. At all conditions of evaluation of these resins, polyvinyl butyral show the best results i.e. it doesn't change rock surface colour and keep rock breathability, maximum penetration depth within the stone, highest progress in rock petrophysical and mechanical properties, and the highest rock durability. So, it is recommended for consolidation of sandstone.

To get the best consolidation results on applying this resin on stone surface, three items are recommended to be carried out as preparation of stone surface before applying the resin (Dukes, 1972; Arranitakis and Monokrousos, 1988 and Gauri, 1990), namely:

1-Cleaning stone surface to remove loose grains and salt efflorescence if present.

2-Enlargement of rock pore size (for deeper penetration of the resin) using Infrared applied to stone surface.

3-Drying stone surface as much as possible before resin spray.

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REFERENCES:

1-Abd El-Hady and Kamh, G.M.E. 2001: Examining three resins as stone surface consolidants and currently used reconstruction rock at some Greco - Roman excavations in Alexandria. Proceeding of the 11th International Conference at Alexandria (Environmental protection in a must), May 2001, V.1, PP. 58 - 70.

2-Arranitakis, M. and D. Monokrousos, 1988 : Consolidation work on the sacred rock of the Acropolis, Athens. The engineering geology of ancient work, Monuments and historical sites, Preservation and protection, Marinos and Koukis (eds.), V. 3, PP. 1833 - 1838.

3-Barry, D. F., 1991: Frost weathering and hydration as rock weathering mechanism on Schist, A laboratory study. Earth Surface Processes and Landfroms, V. 8, PP. 535 - 545.

4-Bell, F. G. and J. M. Coulthard, 1990: Stone preservation with illustrative examples from the United Kingdom. Environmental Geology and Water Science, V. 16 (1), PP. 75 - 81.

5-Dukes, W. A., 1972: Conservation of stone: Chemical treatments. The architects Journal Information Library, Part 23 AVG, PP . 433 - 438, Technical study 2.

6-Gauri, K. L., 1990: Decay and preservation of stone in modern environments. Environmental Geology and Water Sciences, V. 15 (1), PP. 45-54.

7-Hawkins, A. B., 1972: The adverse impact of acid rain on some resins used for ancient monuments in Athens. The Science of the total environment, V . 60 (1), PP. 102 - 114.

8-Ismaiel, B. M., and G. H. El-Habaak, 1995: Durability characteristics of some sandstone, diorite and granodiorite monuments. J. of South Valley Univ., Qena, Fac. of Arts, V. 5 (2), PP. 59 - 85.

9-Kamh, G. M. E., 2001: A comparative study on the impact of environmental geological conditions on some archaeological sites at Giza (Saqara region) and Alexandria governorates, and their modes of preservation. Ph.D. Thesis, Faculty of Science, Menoufiya University, Egypt, Jan. 2001.

10-Marketa K.. and P. Kotl, 2001: Modification of stone consolidants based on organosilicon compounds. SWAPNET meeting held on May 2001, UK.

11-Selim, S. A., 2001: Impact of human activities on the Nile Valley aquifer system at Karnak Temples area, Luxor City, Upper Egypt. The Second International Conference on the Geology of Africa, October 2001, Assuit Univ., V. 1(A), PP. 11 - 25.

12-Subbaraman, S., 1980 : "Conservation of Shore Temple, Mahabalipuram and Kailasanatha Temple, Kancheepuram". Studies in Conservation, V . 28 (3), PP. 1025 - 1033.

13-Warke, P.A. and B.J. Smith, 1988 : Effects of direct and indirect heating on the validity of rock weathering simulation studies and durability tests. Geomorphology, V. 22, PP. 347 - 357.

اختبار أربعة مواد تصلب لتقوية الحجر الرملى المستخدم فى بناء الآثار (معبد الكرنك) بجمهورية مصر العربية كحالة دراسية

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يعانى معبد الكرنك الفرعونى بمصر العليا المبنى من الحجر الرملى الأحمر من مظاهر التلف التى تبدأ من تآكل بسيط فى أسطح الأحجار حتى التلف الكامل لأحجار البناء ، وتحتاج الأجزاء التى تآكلت بسيط إلى إضافة مواد لزيادة صلابتها ضد الأمطار الحامضية والأملاح .

تم اختبار أربعة مواد مفضلة لهذا الغرض ، وبخاصة الحجر الرملى ، وهذه المواد هى : (ميثيل ميثاكريلات ، بوليفينيل بوتريال ، تترا إثوكزيسيلان، إيثيل ترايميثوكزيسيلان) ، ولقد دلت النتائج على أن البوليفينيل بوتريال هو أفضلهم عند تركيزات ١٠% ، ١٥% لأنه يعطى عمق تسرب ٢١ مللى داخل الصخر ، كما أنه لا يغير لون السطح ، ويحفظ نفاثيته ، ويقلل المسامية والمسامية الفعالة للصخر ، وكذلك امتصاصه للماء بدرجة كبيرة عما كان عليه قبل تصلبه ، وكذلك يزيد بدرجة كبيرة من صلابة الصخر من ٥٠,٤ كجم/سم^{٢} إلى ١٩٣,٩ كجم/سم^{٢} .

أيضاً عند تعرض العينات غير المتصلبة بهذه المواد للأملاح والأحماض اتضح أن تلك العينات المعالجة بهذه المادة هى أكثرهم مقاومة حيث أنها تفقد نسبة صغيرة جداً من الحجر الرملى بعد ١٥ دورة من تلك التجارب ؛ لذلك يوصى باستخدام هذه المادة لزيادة صلابة الحجر الرملى المكون لمعبد الكرنك ، وأى آثار مبنية من مثل هذا النوع من الحجر الرملى فى الدول أخرى حيث أن الاختبار تم لهذه المادة ضد عوامل بيئية يمكن أن يتعرض لها الأثر.