EVALUATION OVER TIME OF AN ETHYL SILICATE CONSOLIDANT APPLIED TO ANCIENT LIME PLASTER

Carol A. Grissom, A. Elena Charola, Ann Boulton and Marion F. Mecklenburg

Summary—*Conservare OH, an ethyl silicate solution, has been used for consolidation of ancient lime plaster fragments to enable reassembly of five large statues. The success of treatment was verified by modulus of rupture testing which demonstrated over 300% increase in strength. When viewed with a scanning electron microscope (SEM), the consolidant closely conformed to coccoliths (fossils) in the plaster, and it was uncracked. The SEM showed no detectable changes in the consolidant over the 11-year period in which samples were examined.*

Introduction

A cache of neolithic lime plaster statues from the mid-seventh millennium BC was discovered at the site of 'Ain Ghazal, Jordan, in 1984 [1]. Because the statues were fragmented and fragile, the entire cache was lifted en bloc, and the crate containing this rare material was shipped to the Smithsonian Institution for laboratory excavation and conservation treatment. After extensive analysis of the plaster and testing of potential consolidants [2, 3], the plaster was consolidated with a commercial ethyl silicate solution, ProSoCo's Conservare® OH (the US equivalent to Wacker Chemie's Stone Strengthener OH). Reassembly of the five reconstructable statues in the cache [4, 5] culminated in 1996 with their exhibition at the Arthur M. Sackler Gallery of the Smithsonian Institution followed by exhibition at the Louvre Museum and Institut du Monde Arabe in Paris and finally the Jordanian Archaeological Museum in Amman (Figure 1).

One aim of this paper is to focus on the apparent success of consolidation, verified by modulus of rupture (MOR) testing and illustrated by scanning electron microscope (SEM) examination. Publications on consolidation of deteriorated lime plaster are limited, and published SEM photographs of plaster consolidated with alkoxysilanes are, to the authors' knowledge, non-existent. However, application of Wacker OH to limestone, chemically the same as lime plaster, has been reported, and SEM photographs show the consolidant as a sponge-like mass deposited interstitially between calcite grains in the limestone [6]. This has been interpreted as amorphous silica, bonding only with itself and clumping around molecules of catalyst. Although good results have been reported when ethyl silicates are applied to calcareous stone [7, 8], recent laboratory studies suggest that ethyl silicates are not ideal. For example, it has been reported in this journal that Wacker OH generates an unstable gel because of its catalyst [9]. Brittle, highly strained, and cracked films have also been observed when Conservare OH was applied to slices of a single calcite crystal in the laboratory [10]. Finally, the consolidation of limestone with this product has often been said to be inferior to that of siliceous substrates for which it is believed to have an affinity; modulus of rupture test results for limestone and sandstone of similar porosity have been performed to demonstrate this thesis [11].

A second aim is to evaluate aging properties of Conservare OH on lime plaster, mainly through SEM examination. SEM has been used to examine mudbrick samples consolidated with partially prepolymerized ethyl silicates, and the consolidant has been reported to increase gradually in porosity over relatively short time periods [12]. Aging is also an important conservation issue for consolidated stone but, although alkoxysilanes have been widely used for stone consolidation since about 1970 [13], long-term studies of consolidated stone are rare [14, 15]. The lengthy treatment of the 'Ain Ghazal statues in a well-equipped scientific laboratory presented an unusual opportunity for SEM examination over an 11-year period.

Materials

The plaster

X-ray diffraction analysis (XRD) identified the principal constituent of the water-resistant statue plaster as calcite. The acid-insoluble fraction of the plaster ranged from 9 to 15wt%. Most of the fine material of the acid-insoluble fraction consisted of

Received July 1998

Figure 1 *Statue no. 1, 'Ain Ghazal, 6500* BC, *lime plaster; Jordanian Archaeological Museum, Amman, Jordan (photo: Diane Nordeck, Smithsonian Institution).*

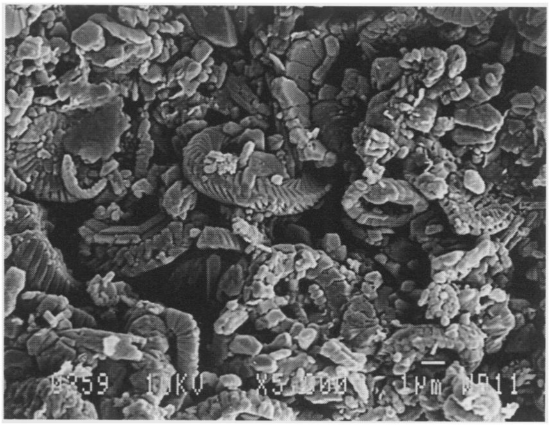


Figure 2 *Unconsolidated statue plaster (no. 3.3A) at 5000× magnification.*

clay-sized particles identified by XRD as a smectite, probably montmorillonite; and a smaller portion, particles measuring >2μm in size, was identified mainly as quartz and feldspar. Sand-sized particles were negligible (<0·2wt%). Marl, a clay-containing limestone, is found throughout the archaeological site, and XRD strongly suggests that it was the raw material for the statues, the marl having nearly identical minor constituents. Coccoliths, fossils of green algae which have been identified as middle to late Santonian in age (85–84 million years ago), are visible in the plaster as well as in marl from the site [16]. They are by far the largest structures in the fine-grained plaster, measuring up to 8μm in diameter (Figure 2). Their abundance indicates that a substantial portion of the plaster was not fully calcined because coccolith morphology is destroyed when the marl is thoroughly calcined, confirmed by tests made on marl from the site. After considerable experimentation, it has been concluded that powdered marl was mixed with a small amount of calcined marl to make the plaster [17]. Even 10% calcined marl conferred water-resistant properties on experimental plaster, and it is apparently the recarbonation of such a small percentage of lime which resulted in the survival of the statues over eight millennia.

When excavated, the plaster varied in strength from powdery and very fragile to relatively strong, but it had to be strengthened if the statues were to be reassembled. Reassembly required considerable handling, and the sculptures themselves are large (about 1m in height) and heavy (the heaviest weighed 18kg before auxiliary support materials were added).

The consolidant

To the best of our knowledge, Conservare OH contains 37·5% tetraethyoxysilane monomer, 37·5%

tetraethoxysilane dimer, 16% methylethylketone (butanone), 8% acetone (propanone), and 1% dibutyltindilaurate catalyst [18]. Its selection followed experimentation with a range of consolidants, including Acryloid B-72, other alkoxysilanes, and mixtures of those two materials. Reasons for its selection were good penetration, even deposition, ease of application, and improvement in mechanical strength.

It is noteworthy that a different method has been used for consolidation of similar plaster statues from the same site in another laboratory [19]: repeated application over many days of a mixture of uncatalyzed methyltrimethoxysilane and Raccanello E55.050 (composed principally of an acrylic resin, with some silicone resin). The condition of this cache of statues presented an altogether different consolidation problem, however, because many of the statues had become cemented to each other during burial. The consolidant mixture made the plaster strong enough that fragments of cemented-together statues could withstand separation, without preventing them from being detached.

Experimental methods

Modulus of rupture (MOR) testing

Four-point bend bar tests with an Instron TTCM at the National Institute for Standards and Testing were performed on eight samples of relatively strong original plaster, and the indicated tensile strength ranged from 1·57 to 2·65MPa, averaging 2·06MPa. These figures are probably somewhat high because the test bars (7 × 7 × 25mm) were shorter than would have been most desirable, but they provide useful comparative data in the absence of more abundant test material.

An apparatus was assembled by one of the authors (MFM) for four-point bend bar tests of plaster to which a range of consolidants had been applied. Because expendable original plaster was inconsistent and limited, replica test bars imitating original plaster in composition were made from a mixture of 85% ground chalk, 10% lime and 5% clay. They were cast in Plexiglas moulds measuring 9 × 12 × 76mm and allowed to dry in the laboratory for one week after unmoulding. Consolidants were applied by pouring small amounts into the bottom of Petri dishes containing test bars and allowing them to wick up to the top of the bars. The consolidated test bars were allowed to cure for five weeks before testing. Initial testing showed the replica plaster to be valid test material: measurements were consistent among control samples (Figure 4), and their mean tensile strength (2·98MPa) was reasonably close to that of the original aged plaster. Furthermore, the consolidation of the replica test bars imitated that of original plaster in quantity of consolidant: the average percentage increase in weight for the Conservare OH-impregnated replica plaster (7%) was the same as for fragments consolidated during treatment.

Scanning electron microscope (SEM) examination

Fragments were selected for SEM examination from those which were large enough to allow deposition to be examined at least 1·5cm from the application surface and which could be divided into at least four pieces to allow examination of comparable samples over time. In total, 17 consolidated samples from eight different fragments were examined with the SEM.

Fragments which had the oldest consolidant were consolidated during initial testing, e.g., fragment no. 19. Others were consolidated in the normal course of treatment, e.g., fragments nos. 37.15A, 35.13C and 12.11F. In both cases, the consolidant was applied by dripping from a pipette until the plaster no longer accepted liquid. Application was repeated several times at intervals over the course of one hour with the intention of achieving full penetration of the plaster. The nature of application and the material suggested that the quantity of consolidant in the plaster might vary substantially between fragments. Weight gain for five fragments consolidated in the normal course of treatment, however, ranged only from 6 to 7%. Over the years, samples were stored in the climate-controlled laboratory at about 45% RH and 25°C.

In order not to introduce toolmarks, fragments were fractured to produce surfaces to be examined. Samples were mounted for SEM examination so

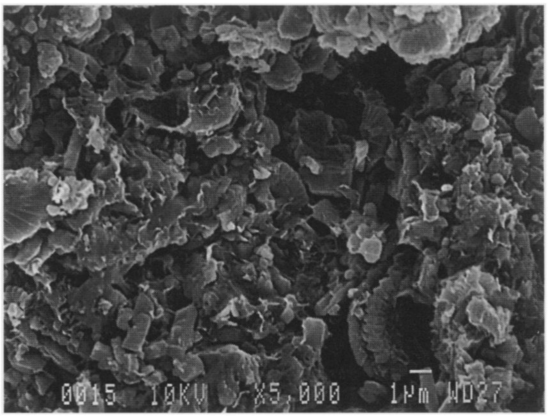


Figure 3 Un-etched statue plaster (no. 37.15A), six months after consolidation (5000 ×).

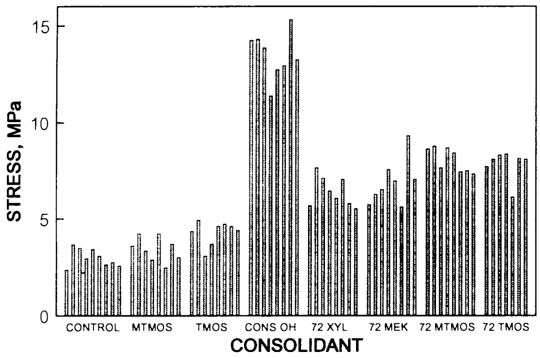


Figure 4 Results of four-point bend bar testing of replica samples made of 85% chalk, 10% lime and 5% clay, consolidated with the following materials: none (CONTROL); methyltrimethoxysilane (MTMOS); tetramethoxysilane (TMOS); Conservare OH (CONS OH); 5wt/vol% Acryloid B-72 in xylene (72 XYL); 5wt/vol% Acryloid B-72 in a solvent mixture of 40% methylethylketone, 50% xylene and 10% cellosolve acetate (72 MEK); 5wt/vol% Acryloid B-72 in methyltrimethoxysilane (72 MTMOS); and Acryloid B-72 in tetramethoxysilane (72 TMOS).

that they could be examined both near the outer surfaces of the fragments and in depth. Relatively flat portions were selected for mounting, and the surfaces were not polished. Initially, mirror-image pieces of the same fragment were examined, one etched with acid and the other not. Acid etching was found to be essential for clear examination of the consolidant, however, and subsequently all samples were acid-etched prior to examination. This was done according to a procedure used for examination of consolidated limestone [6]. 1M hydrochloric acid was dripped onto the sample and, when fizzing stopped, the sample was thoroughly rinsed with de-ionized water and air-dried; however, the acid was applied only once, this proving sufficient for dissolution of the plaster. Holes in the consolidant, most clearly visible in Figure 7, were likely made during acid etching. Samples were sputter-coated with multiple layers of carbon and then gold/palladium, and they were examined shortly after coating. Nevertheless, the porosity of the samples produced charging effects which made photography difficult.

A JEOL 840A scanning electron microscope equipped with energy dispersive X-ray analyzer (EDXA) was used for examination. Magnification of 2000 × proved best for assessment of quantity of consolidant because it is sufficiently high to see individual particles yet the area examined is large enough to be fairly representative. Samples were examined throughout to ascertain if deposition was uniform, especially between outer surfaces of the fragments and the interior. Qualitative examination proved better at 5000 × because fine detail of the consolidant, especially impressions of coccoliths, became clear. EDXA was used to confirm identification of calcite and clay particles, as well as the consolidant.

Test results and discussion

Improvement in the tensile strength of the plaster after consolidation was verified by MOR testing (Figure 4). Compared to the mean strength of replicate plaster controls (2·98MPa), that treated with Conservare OH increased in excess of 300%, measuring 13·5MPa. In practice, consolidated fragments proved quite tough when handled, and the statues were reassembled without any evidence that the plaster was insufficiently cohesive.

The SEM showed that the consolidant fills pores in the plaster and bonds clusters of small particles with larger ones, illustrated by comparing a sample of unconsolidated plaster (Figure 2) with a sample of consolidated plaster six months after treatment (Figure 3). Cracking of the consolidant, characteristic when Conservare OH is applied to sandstone or allowed to cure in a large quantity in a Petri dish, is notably absent [9, 20]. This may be because of the small particle size of the fine-grained plaster.

With calcareous material etched away, details of consolidant coating can be more easily compared between samples using the SEM, and impressions of coccoliths prove particularly useful reference features for comparisons. The consolidant conforms closely to the coccoliths, reproducing their struc-

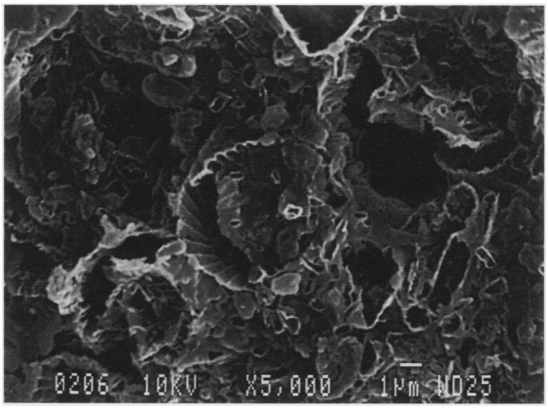


Figure 5 Acid-etched statue plaster (no. 35.13C), four months after consolidation (5000 ×).

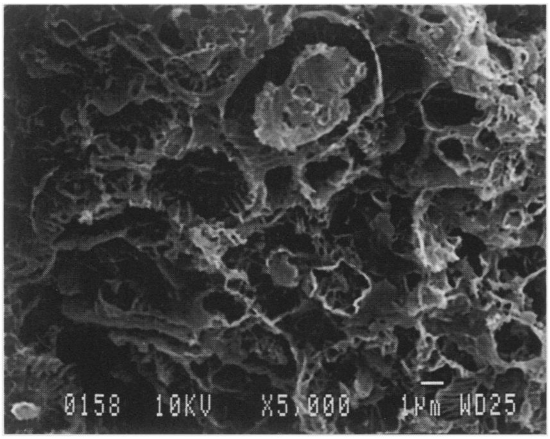


Figure 6 Acid-etched statue plaster (no. 12.11F), one year after consolidation (5000 ×).

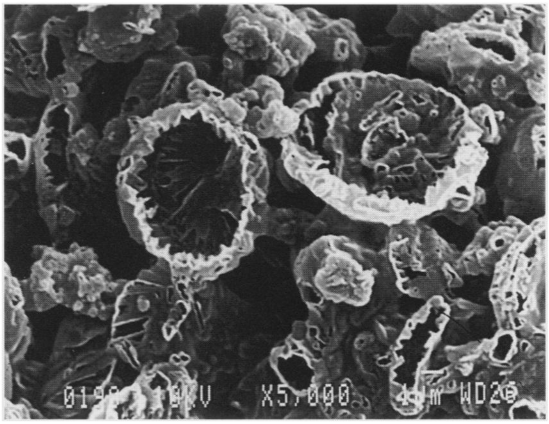


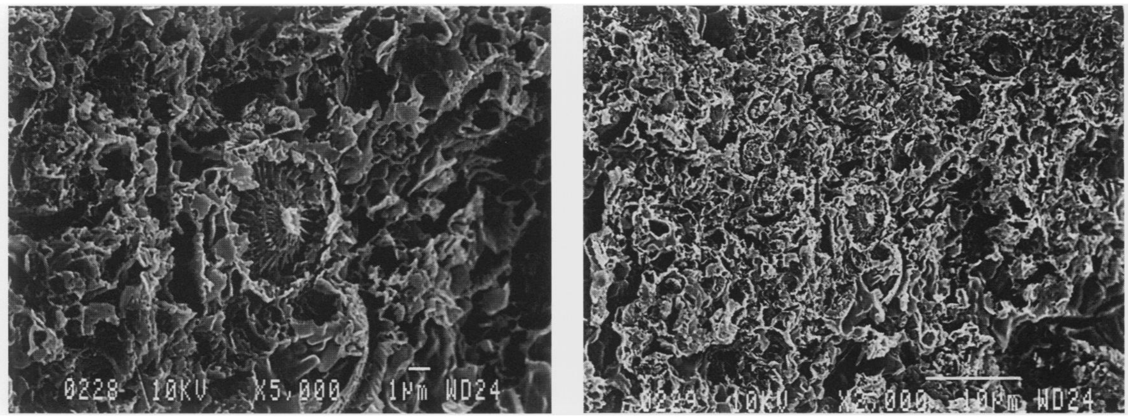
Figure 7 Acid-etched statue plaster (no. 19), six years after consolidation (5000 ×).

tures with precision: for example, the impression of the distal shield of a coccolith at the centre of Figure 5. It is also interesting to note that coccoliths appear so closely coated by the siliceous consolidant, since fossil coccoliths are found only as calcite. Particle morphology may be important in film formation, as the coccoliths are made up of crystallites of varied shapes and sizes in which the basic rhombohedral shape of the calcite has been modified by the algal cell to fit into specialized morphologies [21]. As mentioned above, particle size may also play a role, given that the plaster is much more finely divided than calcite grains found in building stone, where consolidant tends to deposit between grains.

Comparison of samples with the SEM provides information on variation in the amount of consolidant which was deposited. For example, Figures 5 and 7 show slightly more consolidant than others. Distribution of the consolidant is reasonably uniform, as shown in the lower-magnification view of the seven-year-old sample (Figure 8b). Examination across each sample confirmed that the consolidant was not deposited more heavily near the outer surfaces of fragments. This distribution may be partly attributed to the presence of the naturally occurring clays, since they may improve bonding of the consolidant on account of their siliceous nature and hydroxylated surfaces [22]. It contrasts sharply with solvent-deposited consolidants like Acryloid B-72 and Acryloid B-72/alkoxysilane mixtures which deposited more heavily at evaporation surfaces, confirmed during the experimental testing phase with iodine staining.

When samples were compared using the SEM, no significant changes were found in the micromor-

a



b

Figure 8 Acid-etched statue plaster (no. 19), seven years after consolidation. (a) 5000 ×, (b) 2000 ×.

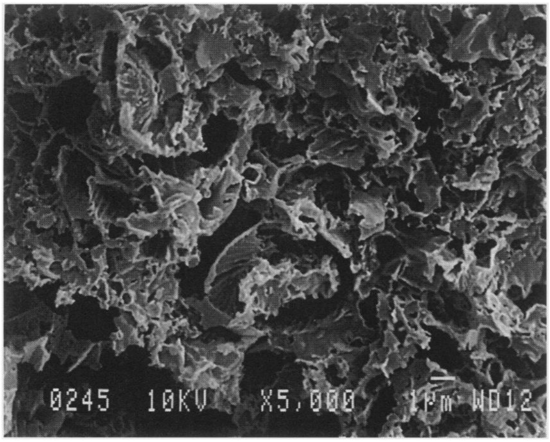


Figure 9 Acid-etched statue plaster (no. 19), 11 years after consolidation (5000 ×).

phology of the consolidant from the time it can be considered 'cured' and nearly 11 years later. This is illustrated by photographs taken of the consolidant after four months (Figure 5), one year (Figure 6), six years (Figure 7), seven years (Figure 8) and 11 years (Figure 9). Apparently no further reactions occurred after four months, and consolidation of the lime plaster appears to be stable under the environmental conditions in which the samples were kept, equivalent to museum conditions. Because the consolidant did not form gel-like masses as it does on sandstone, it is unlikely to be unstable over time on account of cracking. Our SEM results are in keeping with the fact that the handling strength of fragments did not seem to diminish from the time fragments were consolidated during treatment seven–eight years ago to the present time, based on assessment of unattached fragments remaining in the laboratory. They are also consistent with one of the few studies on the aging of silicate esters applied to stone: biaxial flexural tests on drill core slices of sandstone consolidated with two different commercial ethyl silicates showed no significant decrease in consolidation efficiency during 10 years of outdoor weathering [14].

Conclusions

Despite less optimistic predictions for consolidation of calcareous materials with ethyl silicates [11], successful consolidation of the 'Ain Ghazal lime plaster was demonstrated by improvement in strength which was sufficient for the task at hand, the reassembly of the statues. Improvement in strength was confirmed by MOR tests on replica plaster which showed an average increase in strength of over 300% compared to that of control samples. The SEM showed uniform formation of a network of consolidant throughout the samples and fulfilled criteria recently proposed for consolidants under SEM examination: 'formation of grain–grain bridges, adhesion, and grain surface filming' [23].

No changes in the Conservare OH are apparent over the 11 years it has been studied with the SEM. The absence of cracking in the consolidant is particularly noteworthy because cracking has been used as a measure of instability in laboratory studies and is considered a potential problem for the long-term durability of consolidated sandstone. Our results are consistent with aging of silicate esters applied to outdoor stone. While there is no climate control in the museum in which the statues are displayed in Jordan at present, conditions are far less severe than exterior exposure, and it may be anticipated that the consolidated statues will remain stable.

Acknowledgements

The authors wish to thank Richard Fields at the National Institute for Standards and Testing (NIST) for Instron testing; Jean M. Self-Trail and Laura Bybell of the United States Geological Survey for identification of the coccoliths; Kathy Tubb at the Institute of Archaeology, University College London, for discussions of plaster consolidation; and Melanie Feather and Camie S. Thompson of SCMRE for SEM photography of difficult samples.

Suppliers

Acryloid B-72: Rohm and Haas, Philadelphia, PA 19105, USA.

Conservare® OH: ProSoCo, PO Box 1578, Kansas City, KS 66117, USA.

Methyltrimethoxysilane (MTMOS), T4-0149: Dow Corning Corporation, South Saginaw Road, Midland, MI 48686, USA.

Tetramethoxysilane (TMOS), T1980: Petrarch Systems Inc., 2731 Bartram Road, Bristol, PA 19007, USA.

Consolidante Acrilsiliconico E55.050: Raccanello S.p.a., Industria Vernice e Smalti, Padova, Italy.

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Résumé—*Une solution de silicate d'éthyle, Conservare OH, a été utilisé pour la consolidation d'anciens fragments d'enduit de chaux en vue de reconstituer cinq grandes statues. Le succès de l'opération a été vérifié par des tests de rupture qui montrent un accroissement de la résistance de plus de 300%. Par examen au microscope électronique à balayage (MEB), on voit que le consolidant imprègne correctement les oolithes (fossiles) dans l'enduit, et n'est pas craquelé. Le MEB n'a pas montré de changements appréciables dans le consolidant au cours de la période de 11 ans durant laquelle les échantillons ont été suivis.*

Zusammenfassung—*Conservare OH, eine Ethylsilikat-Lösung, diente zur Konsolidierung alter Kalkgipsfragmente mit dem Ziel, die Wiederherstellung fünf großer Statuen zu ermöglichen. Der Erfolg dieser Behandlung konnte mit Tests zur Biegefestigkeit nachgewiesen werden, die eine 300%ige Steigerung der Festigkeit ergaben. Bei der Untersuchung mittels Rasterelektronenmikroskopie (REM) zeigte sich, daß sich das Festigungsmittel eng an Coccolithe (fossile Einschlüsse) im Gips angelagert hatte und keine Riße sichtbar waren. In dem 11-jährigen Untersuchungszeitraum konnten mittels Rasterelektronenmikroskopie keine sichtbaren Veränderungen des Festigungsmittels festgestellt werden.*

Resumen—*Conservare OH, una solución de etil silicato, ha sido usada para la consolidación de fragmentos antiguos de argamasa de cal, con el fin de facilitar el reensamblaje de cinco grandes estatuas. El éxito del tratamiento se verificó probando los módulos de ruptura, lo que demostró aportar un aumento de más del 300% de resistencia. Al ser observado usando microscopía electrónica de barrido (SEM), el consolidante claramente se adaptaba a los cocolitos (fósiles) presentes en la argamasa, y se mostraba, así mismo, íntegro y sin grietas. La microscopía SEM no mostró tampoco cambios detectables en el consolidante durante el período de 11 años que duró el examen de las muestras.*