A Comparative Study of TEOS-based Formulations for the Consolidation of Adobe

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Abstract—Archaeological site managers and conservators alike face challenges in preserving outdoor structures constructed of earthen materials. The material requires ongoing maintenance and sometimes immediate action upon excavation due to its susceptibility to erosion by water and other climatic elements. Tetraethoxysilane (TEOS) is a liquid precursor of silica (SiO_2) used as a consolidant for silicate-based stone, which has also found application for the conservation of earthen materials. Though this intervention is irreversible, the application of TEOS has the potential to preserve earthen structures at high risk of surface and structural deterioration. This study tested the effectiveness of TEOS-based formulations at consolidating bricks to prevent physical alteration and deterioration from water.

TEOS Dynasylan® 40 was evaluated alone and in combination with the water repellant, hydroxyl-terminated polydimethylsiloxane oligomer (PDMS-OH), both with and without solvent dilution. The formulations were applied by brush to lab-prepared adobe bricks. Consolidation effectiveness was estimated by measuring penetration depth, and with water immersion and capillarity tests and was complemented with Scanning electron microscopy observations. . Dynasylan® 40 provided impressive cohesion when immersed and as expected, the addition of PDMS-OH induced a reduction of water absorption through capillarity. Consolidant formulations diluted with ethanol resulted in reduced penetration, while dilution with white spirits did not have an impact on penetration depth. Our results also suggest that application via brush is imprecise and difficult to control.

1. Introduction

Earth-based materials, like adobe bricks, have been utilized for building and architectural purposes by nearly every civilization in the course of history, yet adobe poses many challenges in both its immediate and long-term preservation. The vulnerability of the material to most environmental threats, in particular those involving water, emphasizes the worldwide need for adobe conservation research.[4] When adobe is formed, the lamellar structure of the dispersed clay minerals contributes to the formation of a cohesive mass during the drying phase (Velde 2008, Minke 2006). However, the capacity of clays to interact with water makes adobe highly susceptible to liquid and vapor water damage, a duality that reflects the antinomic behavior of clay minerals.

Adobe is used as a building material with the understanding that it needs continual maintenance, repair and replacement throughout its life—demands that are mirrored in its treatment, and which

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[4] A short list of environmental threats to adobe include: rainfall, water infiltrations, flooding, earthquakes, fluctuations in relative humidity and temperature, wind erosion, salt deposition and migration, as well as biological and human factors. (Helmi 1990, 280; Ren and Kagi 1995, 433; Oliver 2008, 110; Rainer 2008; Velde 2008).

further illustrate the inherent challenges of its conservation. Conservators and archaeologists require methods to preserve adobe well beyond its intended lifespan, often without the resources for monitoring or maintenance. A number of preventive and remedial measures have been employed at archaeological and cultural heritage sites to protect adobe architecture (Oliver 2008a, Matero 2015). Among them, the use of alkoxysilane-based consolidants, such as tetraethoxysilane (TEOS), have shown to be viable and compatible treatment options to restore cohesion to adobe.

Some of the relevant sources that detail the chemistry and physics of alkoxysilanes and their gels include Brinker and Scherer (1990) and Wheeler (2005). In the case of TEOS, the ethoxy groups can react with water to form Si(OH) moieties and through a series of condensation reactions, lead to the formation of a silica gel deposit. Although mainly studied and used for stone consolidation (Wheeler 2005, Scherer and Wheeler 2009, Doehne and Price 2010), in recent decades, TEOS and other silanes have also been evaluated for the consolidation of adobe, in both laboratory and field settings (Chiari 1987, 1990; Coffman, Selwitz and Agnew 1990; Helmi 1990; Oliver 2008b; Ferron and Matero 2011). In addition to their compatibility with silicate materials, TEOS-based consolidants have low viscosities and surface tensions, low toxicity and volatility, cause little visual alteration, and allow for retreatment. However, they are not without limitations, and their effectiveness is highly dependent on a number of factors that are not yet fully understood nor controllable.

One of the recognized issues with TEOS is the propensity for cracking and embrittlement during gelation and drying, which negatively affects performance. Several approaches have been explored to minimize this effect, including the use of pre-polymerized TEOS (oligomers), dilution with solvents,[5] and the addition of nanoparticles and/or short-chain polymers such as hydroxyl-terminated polydimethylsiloxane (PDMS-OH) to impart some flexibility to the silica network (Wendler 1996; Escalante et al. 2000; Scherer and Wheeler 2009; Salazar-Hernández et al. 2010; Zárraga et al. 2010; Verganelaki et al. 2015; Liu et al. 2016). However, these 'hybrid' solutions have yet to be tested for the consolidation of adobe, and the addition of compounds with water-repellent properties, like PDMS-OH, needs further consideration as it may not be desirable for every adobe treatment (Chiari 1987, 1990).

For this preliminary research aimed at investigating the behavior of TEOS-based products for the consolidation of adobe, we have used a commercially available oligomeric TEOS (D40) and lab-made adobe brick samples. The consolidant was tested pure and in dilution with different solvents, as well as mixed with small amounts of PDMS-OH. For our adobe formulation, we have incorporated an acid-activated clay (Montmorillonite KSF) as a potential in-situ solid catalyst for the formation of the silica or modified-silica network, which could also favor interactions with the clay matrix. The consolidation treatments were evaluated based on depth of penetration, distribution and texture of the consolidant, water absorption, and hydrophobicity.

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[5] Dilution has been shown to reduce cracking in the dried gel (Scherer and Wheeler 2009, 9), as well as enhance penetration and overall strengthen the consolidation. Likewise, solvent choice can impact the condensation reaction, influencing the efficacy of the treatment (Wheeler 2005, Scherer and Wheeler 2009). Ethanol is predominantly used to dilute alkoxysilanes, however other solvents include white spirits, white spirits, toluene, xylenes and acetone (Wheeler 2005, 76).

2. Experimental

2.1 Materials

2.1.1 Adobe Bricks: Composition and Preparation

Adobe bricks were made with 62.5% of coarse sand, 11.6% of silt (fine sand), 14.7% of montmorillonite KSF (Acros Organics), 9.8% of kaolin (non-swelling clay, Kremer 58250) and 1.3% rice hulls (percent by weight). Approximately 1300mL of water was added to the dry ingredients to achieve a workable adobe mixture that was pressed into a rigid 25.4 x 38.1cm plastic container with 5cm high walls. The slab was left to dry under ambient conditions for four days before it was cut into 22 brick samples, approximately 5.0cm x 7.5cm x 6.0cm. The bricks were further desiccated in the oven for three days at 50˚C and re-equilibrated in lab conditions, which resulted in an average weight increase of 0.6 grams due to the adsorption of water from the ambient atmosphere. The porosity of the bricks was measured through capillary absorption with white spirits. After conversion of the weight increase into volume, the calculated accessible porosity was about 27%.

2.1.2 Conservation materials

Dynasylan 40 or D40 (Evonik Industries) is a colorless, low viscosity ethyl silicate, which in the presence of water and a catalyst, hydrolyzes and condenses to form a silica-based network. D40 will form approximately 40-42% (w/w) silicon dioxide upon complete hydrolysis and condensation. D40 was catalyzed with 1% (w/w) di-*n*-butyltin dilaurate (DBTL), a neutral pH organotin catalyst (Gelest Inc.). The catalyzed D40 was tested pure and mixed with 10% (in volume) of short-chain PDMS-OH (Gelest Inc.), abbreviated D40 + PDMS-OH hereafter. These formulations were tested undiluted and diluted 5:1 with either white spirits (Specturm Stodder Solvent S1957) or ethanol (J.T. Baker Anhydrous Alcohol). The overall concentration of D40 at 5:1 with solvent was 83% without the addition of PDMS-OH and 75% with PDMS-OH by volume.

2.2 Methods

2.2.1 Application

Brush application was chosen because it is most widely used in the field. The consolidant mixtures were applied using 6 passes over the top surface (Fig. 1); the number of passes was determined at half the apparent refusal.[6]

2.2.2 Testing procedure

Treated bricks were left to react in ambient conditions for 30 days prior to testing. To facilitate our tests, the bricks were cut into three equal parts: the outer sections were used for evaluation and the central sections were kept as controls. Results were compared against untreated reference bricks.

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[6] Apparent refusal is the condition when the consolidant pools on the surface, and the substrate remains wet for 1 minute after application (Graziani, Sassoni, and Franzoni 2015, 2). For both practical and economic reasons, application in the field is more closely represented by half the apparent refusal.

Brush application was evaluated by measuring depth of penetration and calculating the amount of consolidant delivered to the bricks. Each brick was weighed before application and immediately after. The penetration depth of the consolidant was measured using the differences in wetting imparted by the treatments. Exposed cross-sections of the cut portions were lightly wetted with water and the penetration of the consolidant assessed as un-treated areas visibly saturated while treated areas repelled the water (Fig. 2). Direct measurements were taken in the center of the brick at the most shallow and deepest points, excluding penetration down the sides of the brick—a bias of brush application.

To test capillarity, right brick sections were placed in petri dishes on top of a layer of glass beads, with the consolidated surface in contact with less than 2mm of tap water (below the depth of consolidant penetration). Their capillary absorption was followed over 48 hours to evaluate how liquid water permeates through the consolidated surface. The bricks were photographed using a Nikon D90 SLR camera, and the maximum height of water absorption was measured from the photos using ImageJ software. Left portions of the bricks were placed into individual plastic cups within a tub which was then filled with tap water, in order to test the consolidations against prolonged and full immersion in water. After 30 minutes, the bricks were removed and allowed to dry for 2 days, including one day in an oven at 50° C, before being re-submerged for 3 days. Photographs were taken 1.5 hours after immersion, and again after 3 days.

Scanning electron microscopy (SEM) observations were done with a NOVA NanoSEM 230 (FEI) instrument equipped with an EDS detector (UltraDry, Thermo Scientific. Gold-coated samples were imaged at different magnifications in secondary electron mode and under high vacuum with an acceleration voltage of 5 keV.

3. Results & Discussion

3.1 Application method evaluation

The total amount of consolidant applied ranged from 3.7 to 5.1 grams (Table 1), which corresponds to an average consumption of about one liter per square meter. The differences do not show any obvious correlation with variables such as composition, dilution or solvent type, and it seems that they are primarily related to the discontinuous, and somehow difficult to control, aspects of the brush application method (Franzoni et al. 2014a). Allowing for a delay between brush passes would likely result in deeper penetration, however the inherently discontinuous method of brushing will always give variable results.

D40 +PDMS-OH penetrated deeper than consolidant without PDMS-OH (Table 2). When diluted with ethanol, the consolidant formulations had the shallowest depth of penetration, averaging around 0.35cm, despite having higher than average weight of consolidant applied. The ethanol likely penetrated less deeply because of its quick evaporation time. For mixtures without ethanol, penetration averaged 0.65cm. Dilution with white spirits did not have a significant impact on depth of penetration compared to undiluted active ingredients, though perhaps greater dilution would have an effect.

3.2 Texture and structure: SEM observations

At high magnification, the main components of the untreated brick are clearly distinguishable with the coarse and fine sand particles embedded in the clay matrix, which shows the typical lamellar structure of montmorillonite and kaolinite (Fig. 3, A, B). For the sample treated with D40, the silica gel that was formed upon the TEOS reaction can be observed in the form of a compact and relatively homogeneous material, without many cracks, filling large pores (Fig. 3, C). In porosity structures of much smaller size, the silica gel can be observed within the clay aggregates or even bridging individual kaolinite platelets (Fig. 3, D). Also noticeable on the latter picture is the – partly –mesoporous texture of this gel which could be related to different reaction conditions and rates in these very small pores. For the sample treated with D40 + PDMS-OH, no major difference could be observed, in particular regarding the flexibility of the silica network, though the distribution of the consolidant in the clay matrix seems to be more homogeneous (Fig. 3, E, F).

3.3 Consolidation performance during water exposure

During water immersion, the untreated areas of adobe began to visibly disintegrate after two minutes, and fully disintegrated in less than a half hour, leaving behind only the portions of adobe treated with consolidant. Each formulation of D40 withstood immersion in water for 3 days, without visible losses or change to the consolidated portions (Figure 4).

Within the first 45 minutes of the capillarity test, the untreated brick suffered slumping and deformation as the water reached the top surface (Figure 5). Water also permeated to the top of the bricks treated with D40 without PDMS-OH, however the brick did not suffer appreciable loss of structural integrity, likely because the unconsolidated areas near the top had less prolonged contact with water. In comparison, a non-wetting meniscus was observed at the interface between water and the brick samples treated with D40 + PDMS-OH, which lasted for over 18 hours with no observed wetting (Figure 5). By 32 hours, the water permeated all treated bricks, and by 48 hours it rose by capillary action to between 12.2 mm and 12.9 mm for all bricks treated with formulations including PDMS-OH. Ethanol dilution of D40 +PDMS-OH had the lowest capillarity height. The water repellency of bricks treated with PDMS-OH may have influenced the lower capillarity height values.

It should be mentioned that exposure of both treated and untreated adobe bricks to water caused a yellowish precipitate to form (Figure 5), which contained hexahydrite ($MgSO_4(H_2O)_6$ based on X-ray diffraction, and a predominant peak for iron when analyzed with X-ray fluorescence spectroscopy. The formation of salts and the release of iron in ferric form can be related to the interaction of water with the acid-treated montmorillonite.

TEOS imparts a temporary degree of hydrophobicity, which can last for several months as ethoxy groups continue to react (Chiari 1987, 2; Ferron and Matero 2011, 58; Franzoni et al. 2014b, 349-50; Scherer and Wheeler 2009, 17). For this reason, water absorption for the sample treated with pure D40 was reevaluated after two years. Within an hour of contact between water and the treated surface, there was a 10% weight increase by the test brick, as well as a softening and progressive disaggregation of the non-consolidated areas. This indicates that the TEOS lost its initial hydrophobic character though continues to significantly improve the resistance to water-induced de-cohesion of the adobe material.

Our results suggest that dilution of D40 beyond 75% (the D40 content in formulations containing PDMS-OH and solvent) could likely occur without reducing the effectiveness of the treatment. This is supported by a study by Ahmadi (2008), which diluted other TEOS products up to 50% with ethanol. Dilution with white spirits provided results consistent with the undiluted formulations, likely because of its slow evaporation rate.

4. Conclusions

Throughout the last decades, studies have shown alkoxysilane-based consolidants to possess a unique compatibility and potential for preserving earthen materials, yet there are conflicting accounts of their success in the field. In this experimental research, preliminary results have shown that undiluted D40 was effective at providing cohesion and strength to the adobe brick samples as well as a long lasting resistance to liquid water. Moreover, the paucity of cracks in the deposited silica gel observed with SEM indicates that the pre-polymerized nature of D40 might indeed reduce the brittleness of the gel. The addition of 10% of a hydroxylated oligomeric polydimethylsiloxane (PDMS-OH) did not significantly modify the appearance and structure of the silica gel, but as expected, brought water repellent properties to the treated part of the brick samples. Our experiments also found that TEOS-based formulations diluted with solvents could offer a more economical alternative without compromising the performance of the treatment. However, the shallower penetration depth obtained for the dilutions with ethanol needs to be investigated further.

Regarding brick composition, while the amount of activated montmorillonite used in the formulation was clearly too high due to the formation of secondary salts, it is believed that the effect of a low percentage of activated clay should be further investigated. It could prove to be an interesting approach to increase the efficiency of TEOS-based consolidation treatments, in particular for the creation of protective and sacrificial earth-based veneers. More generally, the evaluation of 'hybrid' solutions based on innovative multi-component formulations, combining properties which contribute synergistically to performance, represent avenues of research that could significantly contribute to the improvement of remedial treatments of earth-based materials.

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