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SILICA THIN FILM SYNTHETIZED BY SOL-GEL PROCESS FOR THE PROTECTION OF OUTDOOR ARTISTIC CERAMIC IN ARCHITECTURE

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Abstract

A coating treatment with a silica thin film synthesized by sol-gel process was performed during the restoration of the Gio Ponti's Church of Santa Maria Annunciata in Milan (Italy). Despite its recent construction (1964-67) the external façades made of porcelain stoneware tiles presented degradation phenomena caused either by exposure to weathering and by the fabrication characteristics of the material. A careful analysis of the deterioration of ceramic elements by optical microscopy and ESEM-EDS was performed in order to characterize the material and to project a proper intervention. A silica thin protective film was synthesized by sol-gel process, according to the method developed by the research group of Chemistry for Cultural Heritage of the University of Padua, and adapted to the specific application by the spin-off company Siltea srl. The method involves the neutral pH catalysis of the *sol* and the room temperature condensation of the film. The coating was applied by means of conventional air-spray gun. Optical microscopy and ESEM-EDS analysis before and after accelerated ageing on several samples were performed to evaluate the protective efficiency of the coating. The treatment was realized on a surface of 300 square meters with good results, encouraging the application of this new technology as an efficient alternative to the traditional methods, often based on the use of synthetic organic polymers.

INTRODUCTION

The Church of *Santa Maria Annunciata* at the *San Carlo Borromeo* Hospital in Milan was built between 1964 and 1967 on the architectural project of Gio Ponti. (Milan, 1891-1979) who was a very important Italian architect but also an industrial and furniture designer.

The church is considered a prime example of the XX century Italian architecture. Despite its recent construction, a poor state of preservation was observed. For this reason the Italian Ministry of Cultural Heritage (MiBAC Ministero per i Beni e le Attività Culturali) promoted the restoration and conservation of the building. Particular attention was paid for the external façades which were studied in depth [1]. The diagnostic analyses on the outdoor ceramic revealed a complex situation, identifying surface alteration processes caused by exposure to weathering and by the characteristics of the material itself. This kind of degradation is not usually recognized in historical buildings and it is considered a particular issue of contemporary art conservation. It is important to note that the tiles were designed by Gio Ponti and produced for this church, thus their conservation is of primary

interest. The application of a silica sol-gel synthesized coating for the protection of the ceramic tiles was considered particularly suitable for this case, where physical and chemical stress conditions on the surfaces are relevant, and where the stability to direct solar irradiation is necessary. These requirements are not satisfied by the traditional protective systems, often based on organic polymers which tend to turn yellow and to become a substrate for biological attack. Moreover, the formation of cross-linking in aged polymers, changes their solubility with the consequence of a complicate and even damaging removal [2] [3] [4]. The thin film is chemically bonded to the substrate and the compatibility is guaranteed since the coating and the substrate have the same silicates chemical nature.

A specific sol-gel synthesis for the application on surfaces of artistic value was developed by the group of Chemistry for Cultural Heritage of the University of Padua. The innovative method of silica thin film deposition allows obtaining inorganic, pure and highly compatible protective layers for silicates substrates without heating treatments and in neutral environment, in accordance with the requirements of an intervention on cultural heritage material. The method was successfully applied to the surface of ceramic tiles of the church. The treatment was performed at the end of the restoration of the façades and involved the original and the replaced tiles which were both subjected to laboratory and *in situ* tests before and after the protective coating treatment.

MATERIALS AND METHODS

A specific protocol for the definition of the more suitable treatment was elaborated. It involved the synthesis and the choice of a proper method of application.

Three different situations were identified among the material and taken into account:

1. well preserved original tiles
2. degraded original tiles
3. tiles of new production for the replacement of missing and unrecoverable elements.

The material was previously studied with particular attention to the degradation processes and their consequences on the ceramic tiles. Samples and cross-sections optical microscopy analysis, together with morphological and chemical analysis using ESEM-EDS technique (Environmental Scanning Electron Microscopy with Energy Dispersive Microprobe, FEI Quanta 200) allowed a complete characterization of the ceramic material.

On the basis of the results, a specific formulation of the sol-gel coating was adopted using Tetraethoxysilane (TEOS) as a precursor and a specific catalyst to avoid migration of lead ions and iridescence effects [5]. The sol-gel method is based on chemical reactions in liquid phase and not on the high temperature treatment of raw materials as sand and clay, thus the resulting material is characterized by high purity, being composed only by silicon and oxygen atoms. The potential application of the sol-gel coatings for the protection of surfaces in the cultural heritage field was limited by the pH of the solution and the thermal treatment for the densification of the film. The innovative formulation developed at the department of Chemical Sciences of the Padua University overcomes these problems.

In neutral environment the kinetic reactions is very slow, thus the synthesis is usually accelerated by means of acid or basic catalysts. For example, an acid catalyst is able to promote the hydrolysis, the first step of the sol-gel process in which the resolution of the molecules of the precursor is observed when reacting

with water. The modified method is characterized by the substitution of the acid catalysts with aqueous solutions with Lewis acids, thus realizing a film deposition in neutral conditions. During the next phase of condensation, the polymerization occurs with the elimination of water or alcohol molecules and the formation of $\equiv\text{Si-O-Si}\equiv$ bonds. The viscosity increases forming a network which progressively entraps the remaining solution (gelation). An ageing is required in order to allow the structural evolution by the formation of covalent bonds. After this period, the deposition can be performed. The drying starts immediately after the application, with the loss of water and the evaporation of volatile compounds. The phenomenon involves first the surface and then the evaporation of liquid from within the pores due to the capillarity. This is a critical step of the sol-gel process, since stress produced by contraction may lead to cracking. The formation of the solid material is possible by the densification which is usually realized by a thermal treatment between 500° and 600°C [6]. With the new formulation the densification occurs at room temperature avoiding substrate damaging and impurity incorporation making possible also *in situ* applications. The film is formed instantly and reaches the total stability in few days [7] [8] [9]. The thin layer formed on the surface of the treated material has a thickness between 150 and 250 nanometers (depending on the method of application) with a composition of amorphous SiO_2 (purity >99.99%). The film is transparent, colorless and isotropic.

Two methods of application were tested: paint brush and airbrush with compressed air. The treatment was tested *in situ* and in laboratory. After a period of natural ageing for *in situ* tiles and climatic chamber cycles for laboratory samples, the morphological and chemical analysis were performed again, in order to evaluate the effect of the protective treatment. Artificial ageing was performed by means of an Atlas Sunset CPS+ climatic chamber.

RESULTS

The ceramic tiles show two different shapes: rectangular with planar surface and diamond-shaped with a central cusp. The morphological analysis of the ceramic samples and their stratigraphy revealed similar features for all the tiles. The ceramic fabric is compact, white with pink hues. Ocher and black inclusions are dispersed in the white matrix. The glaze is grey and it is characterized by a diffuse porosity with bubbles.

The polished section allowed observing two well distinct cohesive strata. The glaze has a thickness of about 350 microns with inclusion of the mean diameter of 10 microns and porosity of 100 microns (Fig.1). A silicate composition of the glaze was found through ESEM-EDS semiquantitative analysis expressed in weight %: Silicon (35 %), Aluminum (3%), Sodium (2%), Calcium (3%), Magnesium (0.8%), Zinc (2%), Potassium (2%) Lead (4%).

The silica thin film synthesized by sol-gel method was considered suitable for this kind of material, since the protective layer does not contain significant amount of alkali ions which may be subjected to leaching. This is important since the persistence of basic solution formed by the weathering of the glaze could be responsible of further damage of the surfaces.

Once characterized the surface to be protected the sol-gel treatment was formulated and tested.

An essential preliminary step was the cleaning of the surfaces, which is necessary in order to ensure the formation of the chemical bonds between the thin film and the glaze. Two methods of application were tested, by means of paintbrush and by spray coating.

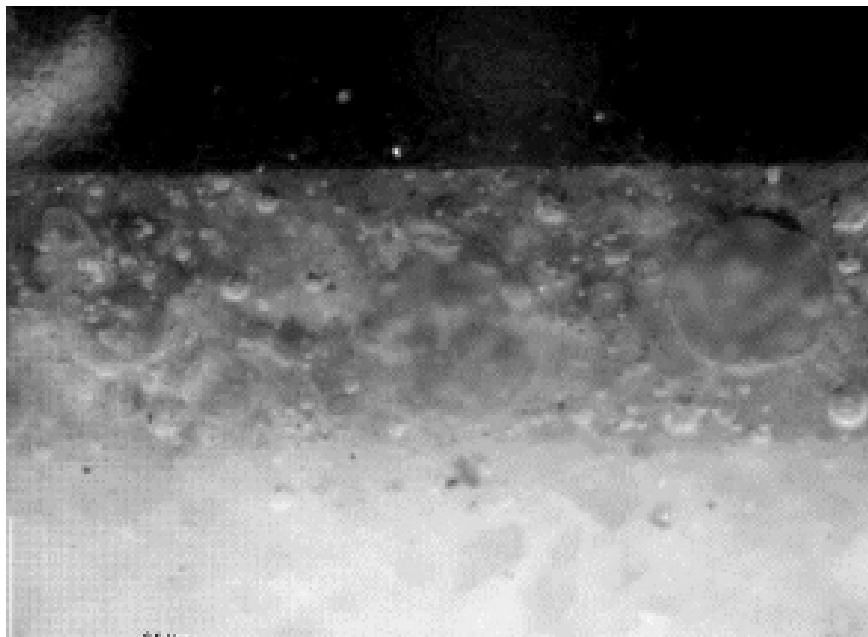


Figure 1. Digital microscope image of the polished section of the ceramic tile. The glaze (upper layer) has a thickness of about 350 microns and it is characterized by a gray color, diffuse porosity and bubbles. The ceramic body is white, compact with ocher and black inclusions.

The deposition performed with the paintbrush was not satisfactory even if the resulting film was homogeneous and covered the entire surface.

Excellent results were obtained by applying the product with an airbrush, ensuring a uniform deposition and an adequate homogeneous thickness thanks to the rapid evaporation of the solvent. The method was also thought to be suitable for the *in situ* application.

Since the two main problems of the glaze were the pitting corrosion and the cracks, particular attention was paid for these phenomena. In order to verify the capability of the protective treatment to repair and to stop this kind of degradation, two original samples affected by pitting and cracks and two samples of the replacing material were analyzed before and after the treatment. The ESEM-EDS analysis in the crack showed siliceous deposits along the discontinuity of the glaze and Cu, Fe and P presence inside of the fracture.

In the ESEM images it is also possible to distinguish different phases of the pitting formation and development. The phenomenon starts with concentric fractures whose evolution leads to the loss of the material and the formation of the alveolus reaching in some cases the diameter of 2 millimeters. Inside this area it is possible to recognize a hole. The deposition tests on the tiles of new production show an excellent compatibility of the film with the glazing. Considering the good results obtained in the laboratory tests, a deposition was performed also *in situ*.

In order to evaluate the behavior of the treated tiles in the environment and under specific stress conditions, the samples were undergone to artificial ageing. The first step was the simulation of the solar irradiation with cycles of 12 hours of lighting and dark. The aim was to test the resistance of the film to the daily temperature range and to the UV irradiation. The second and the last steps allowed verifying the interaction of the coating with water, simulating the water condensation on the surface and the effect of rainfalls.

Moreover, the *in situ* coated tiles were analyzed after 60 days, in order to check the interaction of the film with the environment in which it will be applied.

Alterations were not observed after either the natural and artificial ageing. The optical microscopy and ESEM images showed the stability of the film which is not affected by lifting or gaps.

Once tested the conservation conditions of the ceramic and the compatibility of the thin film, the treatment was considered suitable for the application on the outdoor tiles.

The protective treatment was the last phase of the restoration of the church. The surface was firstly cleaned on the basis of a specific protocol developed for the application of this sol-gel thin film. The pre-treatment removes the organic matter from the surfaces and at the same time facilitates a rapid drying. The cleaning was performed by means of a solution of deionized water and surfactant (10:1) which was then washed with deionized water. At the end isopropyl alcohol was used to eliminate organic carbon residues. The cleaning must be realized immediately before the application of the silica coating in order to avoid contamination from the environment. This is fundamental especially when working on outdoor surfaces.

The procedures of the treatment involved contiguous portions of the façade, spacing out cleaning and film deposition according to the scaffolding. In this way the overlapping of different layers of film was prevented. The spray deposition was realized for a total area of 300 mq.

DISCUSSION

The chemical composition of the glazing and its morphology are the results of the traditional production techniques based on the use of sodium chloride salts and alkali lead compounds. Similar results were obtained for the tiles of new production but no lead was recognized in the glazing layer as expected on the basis of the present production methods.

Even if the surfaces appeared in general smooth, the ESEM images show an irregular surface of the tile, due to efflorescence, deposits and alveoli. At a microscopic scale, the glaze is affected by micro cracking, pitting corrosion and scattered alveoli. The phenomena are much more diffuse in tiles from the southern façade, which is exposed to direct solar irradiation and thus to a temperature range larger than the others. The degradation occurs especially on the cusps. The discontinuities of the glaze weaken the whole material thus being more subjected to degradation with respect to the intact material. The micro cracks may evolve in fractures involving not only the glaze but also the ceramic body (Fig.2). Chemical elements not belonging to the chemical composition of the ceramic were transported by polluted meteoric water which flows preferentially along the discontinuities. In particular, Cu and Fe are attributed to the drainpipes and to the metallic roofing, while P is associated to organic residues.

The pitting corrosion was observed in the original and in the new material, supporting the hypothesis that the phenomenon is not related to weathering processes but it is an intrinsic feature of the ceramic. The shape and the distribution of the alveoli which are characterized by a central hole are related to the expulsion of air bubbles during the firing process.

Once tested the chemical compatibility of the sol-gel silica coating, the deposition method was chosen on the basis of the application tests. The paintbrush was excluded due to the thickness of the resulting film, which was considered excessive, compromising the elasticity of the film. Moreover, the traces of the bristles were perceptible, altering the aesthetic features of the glaze. The method was thus considered not suitable for the application on the ceramic tiles. Macroscopic and microscopic observations of spray coated samples confirm that the material after the deposition maintains its aesthetic and chromatic features

since the protective film is homogeneous and does not reveal iridescence effects. Moreover, this method allows a better exploitation of the product, without residues and it is also suitable for the treatment of large surfaces.



Figure 2. The degradation processes affect especially the cusps of the tiles. The cracking of the glaze evolves in the fracture of the ceramic body with loss of tiles. It is possible to recognize the efflorescences along the path of flow of water.

After natural and artificial ageing, any change in the aesthetic and chromatic aspect of the tiles was observed. The function of filler of the treatment for micro cracking is still preserved and the film is also able to contain the phenomenon of pitting corrosion. In the coated samples the degradation process does not develop even in presence of stress conditions while the untreated reference samples show the diffusion of the alveolar areas (Fig.3).

The images referred to the same areas observed after the coating treatment confirm that the film is able to flatten the surface roughness of the glaze, stopping the evolution of the underway degradation processes. As expected, the protective film is not able to restore the deep cracks, since their micrometric dimensions are not comparable with the nanometric thickness of the coating. The tiles showing this kind of damage were substituted before the treatment.

After the restoration and the protective treatment the immediate efficacy of the coating was observed as an increasing of the waterproofing, due to the considerable decrease of the surface roughness. The less specific surface exposed to the environment results in smaller amount of OH groups interacting with water. This is an important feature, since in most cases the degradation processes are linked to the presence of water on the surfaces.

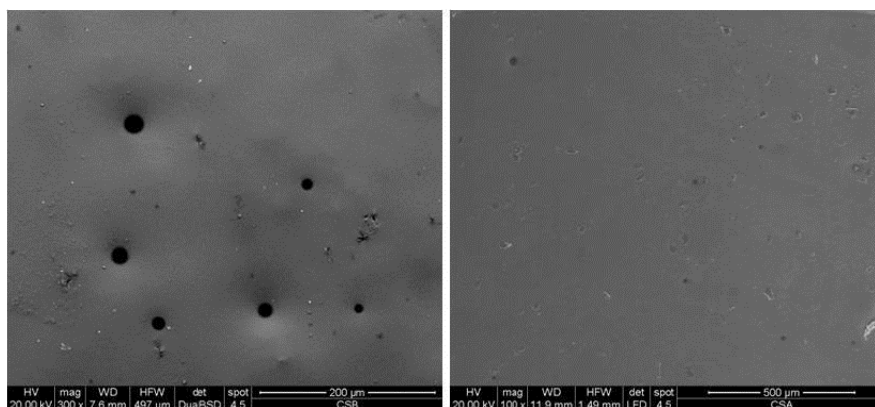


Figure 3. ESEM images of the pitting corrosion in untreated (left) and treated (right) samples after the artificial ageing. The corrosion phenomenon is diffused in the reference sample while in the coated surface the degradation process is stopped.

CONCLUSIONS

The protective silica thin films synthesized by the sol gel method have been successfully applied in the restoration of the outdoor ceramic of the S. Maria Annunciata church in Milan. The treatment was performed after a careful analysis of the material and of the degradation processes. The tests performed *in situ* and in laboratory allow the definition of the protocol of intervention. The coating has proven to be compatible, able to stop the degradation processes and to protect the substrate from further damages. It was verified an increasing of the waterproofing, which gives a better resistance to the material from the atmospheric agents and to pollution. The treatment will be also useful to extend the time between the intervention of maintenance and restoration. From the conservation point of view it was possible to preserve the original material.

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