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Investigation of the change in the characteristic properties of epoxy and silane coated natural stone surfaces

Z. Ebru SAYIN^{a[*](https://orcid.org/0000-0003-0543-5233)}[®],Hakan ÇİFTÇİ^a and Mustafa GÜRSOY^a

a *Afyon Kocatepe University, Engineering Faculty, Mining Engineering Department, Afyonkarahisar, Türkiye*

Research Article

ABSTRACT

Travertine, Silane, Epoxy, Surface Protection, Porous Structure. *Received Date: 25.05.2023 Accepted Date: 25.03.2024* Travertine is a natural stone with macro and micropores; these pores are connected by capillary means. Their visual appearance and porous structure add a different style and diversify their usage areas. However, the large pore structure of travertine brings some limitations in determining the usage areas. In the study, protective solutions called silane and epoxy, which provide protection against water, were used to prevent and/or minimize decomposition in areas of use, depending on the porosity of the travertine, and their effects on travertine were compared. Treatment of both chemicals separately on the travertine surface was carried out by spraying, applying with a brush, and dipping into chemicals. With the findings obtained from the physical examinations after curing, the work continued using the applying and dipping method. The dipping method was evaluated with the chemical's 10, 20, and 40-minutes waiting times. The study found that the best application condition was chemical immersion for 10 minutes. However, in SO_2 wear experiments, it was understood that surfaces treated with epoxy were more durable due to the high viscosity of the chemical. As a result, it has been determined that silane application provides more effective surface protection results than epoxy.

1. Introduction

Travertine is carbonate sedimentary rocks containing calcium bicarbonate $Ca(HCO₃)$ that precipitate after the volatilization of carbon dioxide (CO_2) in contact with the air of spring waters containing calcium carbonate $(CaCO₃)$ and especially hot water sources. Its precipitation generally contains calcite and aragonite crystals (Chafetz and Folk, 1984; Polat, 2011). Travertine has a fossiliferous and porous morphology due to its structure resulting from its formation, and it creates more problems, especially during processing, compared to the other natural stones (marble, granite etc.). It is thought

that the controlled use of tools and equipment used in the process of making sized products in factories can help increase production quality (Kamacı, 2013). While the fossiliferous and porous (porous-porous) structure of travertine is considered a disadvantage in factories (Kamacı, 2013), it should not be forgotten that this structure will be an advantage for thermal insulation. Altay et al. (2001) examined some of the natural stones found in Türkiye in terms of thermal conductivity coefficient. They determined that the thermal conductivity coefficients of travertines, which they chose to represent rocks with high porosity, were lower than other rocks.

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^{}Corresponding author: Z. Ebru SAYIN, zerkan@aku.edu.tr*

In recent years, the increase in buildings in the construction and tourism sector and the demands for using natural stone in the environments have created great activity in the use of natural stone. With the developments in surface processing technologies of natural stones and the construction industry's tendency towards raw materials as buildings surface covering materials, natural stones have begun to be used more than other covering materials. However, the widespread use of building stones, especially porous stones such as travertine, causes limitations according to the region where they will be applied (Erdem, 2016; Hasbay and Hattap, 2017; Tayşi, 2021).

Building materials deteriorate with external factors such as time, environment, and climatic conditions, so they lose their physical and mechanical properties (MEB, 2013). The primary deficiencies in the building blocks used in the construction sector are the changes that may occur in the material characteristics under the influence of moisture and water (Dal and Yılmaz, 2015). It is influential in determining the building material's porosity value. The water absorption value by weight, where this value is intense, increases (Öztank and Bacakoğlu, 2001; Öcal and Dal, 2012) due to wear and tear on non-protective surfaces. However, the capillary channels in the natural stone continue the water movement with the effect of capillary pressure, which affects the water permeability of the travertines and occurs in the veins. Possible temperature changes in the environment and humidity rate also change, transforming from liquid to solid state at water temperature, leading to dew and freezing house (Dal and Yılmaz, 2015). In case of freezing, it is essential to examine the capillary water permeability properties and fill it with a protective material that prevents the water from entering the body to protect the discharge against freezing-thawing since the water exposed to the volume causes internal stresses in the natural stone structure. It is known that the use of travertine, as mentioned earlier as a flooring, facade, or decoration addition, reveals its phenomenon even though it is slow during atmospheric environments. It accelerates this situation in its chemical and physical effects. For example, the impact of sulfate (wearing in SO_2) not only reduces the strength of the natural stone by creating cracks on the surface but also causes the

internal cohesion loss of the hydrated structure and a decrease in the adherence between the building fasteners (Erdoğdu, 1998). With the increase in sulfate concentration, natural stone caused high levels of corrosion. As a result, swelling and disintegration will occur on the surface. The effect will manifest with the spread of fragments from its components and breakage at the edges and corners. (Uğurlu, 2003; Öcal and Dal, 2012; Dal and Yılmaz, 2015). It will cause significant damage to the internal structure by keeping it inside the earth during exposure (Uğurlu, 2003).

Application examples of repairing porous and cracked natural stones and the additional gains these applications bring to businesses are examined in this study. Since crack repair and pore filling is the last stage of production, production losses experienced at this stage increase the cost. All the expenses spent so far on the stone that has been cut and prepared for polishing will be completely wasted if the necessary repairs are not carried out. On the other hand, when we look at the cost of lost materials and labor, it is seen that repair and filling operations are not as costly as expected. On the contrary, it has been stated that they are important tools for generating profits by providing additional added value (Çetin, 2001; Acar, 2003). Making natural and artificial materials resistant to environmental effects has become one of the most important research and application issues, especially in the construction sector and subsequently in various production lines. Among these research products, epoxy and silane were used within the scope of the study.

Epoxy prevents the adverse effects that may occur in the surface coating works of the materials to be used on wet and dry floors, in exposure to atmospheric conditions, eliminates the negativities that arise during production under various mechanical, physical, and chemical effects, and protects the produced material surfaces (internal and external) against all kinds of results by covering them with aesthetic materials. It is a technological building material group developed for Materials coated with epoxy-based products that are used for years, without undergoing any structural changes, to create surfaces that are resistant to chemicals, oil, friction, and abrasion and are used with

advantages such as being easy to clean, hygienic and aesthetic. It has a longer protective feature than most alternative floor coverings, and its most important benefit is that surface renovation can be done at meager costs. Due to these features, it is used in a wide area with increasing demand daily.

Some of the disadvantages of epoxy resins include the need to heat the material before processing and the curing rate being slower than that of unsaturated polyesters. Moreover, it requires special expertise in the epoxy resin system and base epoxies and hardeners may need to be sourced from different manufacturers (Gibson, 2017).

Conversely, silane is a siloxane-added, lowviscosity, colorless, transparent, ready-to-use protective material. As it is an impregnation material with high penetrating properties, it is used in building materials used in exterior cladding, on waterabsorbing surfaces such as natural and artificial stone surfaces, on historical artifacts to protect them from the effects of atmospheric gases, on concrete and cement-based surfaces caused by water, salt, chlorine, and alkalis and used for protection. Adding a solid water-repellent (hydrophobic) feature to the surfaces of the construction elements ensures that they remain dry, reduces heat losses and heating costs, and has the advantage of protecting against atmospheric gases by making the surfaces impermeable and preventing the spread of cracks (Tekno Construction Chemicals, 2023).

Travertines present patterned structures depending on their formation environments. This textural feature can be preferred in the applications of travertine in the construction sector. For this reason, protecting and revealing the structure during production may be desired. In this context, in the study in which the effect of textural properties on capillary water absorption and uniaxial compressive strength parameters of the stone is discussed, it is stated that textural properties affect both capillary water absorption and uniaxial compressive strength. The results have revealed that the technical features depending on the cutting shape should be considered when using travertine (Çobanoğlu, 2020). Natural stones, which have a porous structure and therefore hydrophilic character, should be transformed into products that do not produce bacteria utilizing additives to obtain a waterrepellent feature by making their surfaces hydrophobic, to obtain smooth surfaces and to increase their usage areas and to use them healthily (Çetin, 2001). In this context, the construction chemicals named epoxy and silane were permeated separately on the surface of the travertine selected from porous natural stones in specific proportions to be protective. Their suitability for the usage areas was evaluated by examining the changes in their characteristic features.

Natural stones used in various sectors, particularly travertines, absorb water through capillarity (voids, cracks, cracks, etc.). Over time, natural stones also cause deterioration depending on the amount of water and the penetration time. These deteriorations not only reduce the life of the stone but also cause visual disturbances. The amount of capillary water absorption varies depending on the type of natural stone. The study investigated how much silane and epoxy-coated travertine samples were affected by this situation. Samples treated with silane and epoxy; Water absorption at atmospheric pressure, capillary water absorption, determination of resistance to salt crystallization, and wear tests in SO_2 were performed to investigate the protective solution that is more effective in surface protection against deformation.

2. Material and Method

The travertine sample used in the study belongs to Konya Karaman location. Six hundred specimens of 10x10x2 cm plate and 10x10x10 cm cube samples were obtained from a company operating in İscehisar in Afyon. The specimens brought to the laboratory was subjected to drying at 70°C, then prepared, and XRD (X-Ray Diffraction Analysis) and XRF (X-Ray Fluorescence) analyses were made. Among the protective solutions used on the travertine surface, silane was obtained from Tekno Construction Chemicals (Teknosil product), and epoxy was obtained from Tenax. The guidance and methods to be followed in processing subjects vary according to the characteristics of the subjects mentioned.

Chemical analysis of the sample (in Rigaku/ZSX Primus II brand XRF device) and determination

of resistance to salt crystallization (performed according to TS EN 12370 standard, the prepared solution contains 14% sodium sulfate decahydrate by weight) experiments were carried out in Afyon Kocatepe University, Mining Engineering Department Accredited Natural Stone Analysis Laboratory. Whole rock mineralogical analysis was performed in the Technology Application Research Center (TUAM) laboratory $(2\theta=0^\circ-80^\circ)$. X-ray analyzes were made using Shimadzu 6000 model and Bruker D8 Advance X-ray diffractometers, and mineral identifications were made using JCPDS (Joint Committee on Powder Diffraction Standarts) (1993) cards. The mercury porosimetry analysis of natural travertine, epoxy, and silane-treated samples was performed in a MICROMERITICS brand mercury porosimetry device in the same laboratory.

The contact angle values of the travertine samples was measured using the One Attension Theta Optical Goniometer with the drop diffusion method. Before the measurements, the surfaces of the raw travertine samples were sanded, washed with distilled water, and dried at 105°C for 24 hours. The curing of the silane-coated samples was carried out in two different ways drying at room temperature for 12 hours and at 105°C for 4 hours. In the epoxy coating process, the travertine samples were preheated to 50°C, then coated with epoxy and kept at room temperature for 24 hours to cure. Preheating the samples at 50°C was performed to accelerate epoxy curing.

In the study, epoxy, and silane were mixed separately in a mechanical mixer for not less than 3 minutes and impregnated with a brush on the surface of the washed and dried samples. The impregnation process was applied in two different ways. The first group of work was completed by applying just one coat to the surfaces with the help of a brush. In the second group study, the samples immersed in silane and epoxy were removed and dried after being kept in protective solutions for 10 minutes. In addition to the studies with silane, it was held for 20 and 40 minutes. Since the impregnation made by spraying does not give the appropriate image, the analyses related to this method have been abandoned. The samples were used in the investigation after drying for 48 hours at 24°C to ensure the polymerization process. The grouping of the samples prepared for the studies and the treatment times with epoxy and silane are given in Table 1.

3. Discussion

The result of the X-ray diffractometry analysis of the untreated travertine sample is given in Figure 1. It was determined that the content of the sample was calcite in XRD analysis.

As a result of the chemical analysis of the sample, it was determined that the heat loss was 43.8%, and the CaO content was 55% (Table 2).

In the contact angle measurements of raw and epoxy and silane-coated travertine samples, the contact angles of the samples were measured with distilled water from several different points on the surface, and the average values were recorded. The results are given in Table 3.

As can be seen from Table 3, the contact angle of the raw travertine increased significantly after the silane coating process and gained hydrophobic properties. Coating with epoxy did not cause a severe change in contact angle. Epoxy contains two carbons and oxygen, forming a three-membered ring structure. Epoxy resins have pendant hydroxyl (-OH) groups along their chains that can create strong polar attractions and, therefore, have high surface tension. Due to this characteristic structure, when water is dropped on the epoxy surface, water molecules establish relationships with the functional groups on the surface and spread across the surface, thus giving a low contact angle. The curing method after coating with silane is also effective on the contact angle.

It is known that travertine has very high porosity values among natural stones. This situation brings many limitations in its usage areas. Porosity is most

Figure 1- XRD analysis result.

evident in the water absorption value. For this reason, the change in water absorption values by using silane and epoxy chemicals on the sample was determined according to the TS EN 13755 standard. The raw sample was studied on travertines treated with epoxy and silane for the water absorption test. The results obtained in the water absorption test studies carried out in various parameters are given in Figures 2 and 3.

According to the water absorption test results, the water absorption value of the raw sample was calculated as 0.773. The water absorption value of the epoxy was obtained as 0.582 with a standard deviation of 0.36, and the water absorption value of the silane was obtained as 0.296 with a standard deviation of 0.20 with the same method as a result of the application of epoxy to the surface. When the values were examined, the water absorption value of the raw sample was higher than those treated with epoxy and silane. Silane, on the other hand, has lower water absorption than epoxy. This shows that silane

Table 3- Contact angles of raw and surface coated travertine sample.

Sample	Raw Travertine	Epoxy coated travertine		Silane coated travertine Silane coated travertine (curing: 12 hours, 25°C) (curing: 4 hours, 105°C)
Average contact angle	64.9°	61.62°	109.3°	130.9°

Figure 2- Water Absorption Test Results with Epoxy Applications.

Figure 3- Water absorption test results with silane applications.

and epoxy effectively reduce the water absorption value. Under normal conditions, epoxy surface coating provides complete sealing and zero water absorption. However, as a result of this study, observing some water absorption in epoxy-coated samples is entirely related to the coating process. In other words, since the air bubbles in the epoxy resin were not removed in a vacuum environment before the coating process, they formed partial porosity during curing, which caused the sample to absorb a small amount of water.

The effectiveness of the dipping method was investigated after the application of impregnation with the application. As a result of immersing the samples in epoxy and silane for 10 minutes, water absorption values of 0.291 with a standard deviation of 0.05 in epoxy and 0.278 with a standard deviation of 0.20 in silane were obtained. In this study group, it was determined that the water absorption value of silane gave more effective results than epoxy. In the continuation of the study, the silane immersion time was changed to 20 and 40 minutes, and the results were examined. It was observed that the water absorption values of 0.240 with a standard deviation of 0.18 and 0.231 with a standard deviation of 0.22 respectively, were slightly better against immersion for 10 minutes, but these time changes were not very effective for the study.

The capillary water absorption potentials of the travertine sample were examined, and the relationships between the determined capillary water absorption coefficients and other index properties of the materials were evaluated according to the TS EN 1925 standard. The changes in the capillary water absorption value due to the treatment of travertine used in the research

with silane and epoxy with different methods were examined. The results are given in Figures 4 and 5.

When the capillary water absorption test results were examined, the capillary water absorption value of the raw sample was calculated as 0.841. While the capillary water absorption value of the epoxy was 0.648 with a standard deviation of 0.31 due to the application of rubbing on the surface, the capillary water absorption value of the silane obtained by the same method was 0.383 with a standard deviation of 0.22. When the values were examined, the capillary water absorption value of the raw sample was higher than those treated with epoxy and silane. On the other hand, the capillary water absorption value of silane was lower than epoxy. This shows that epoxy and silane effectively reduce the capillary water absorption value.

The effectiveness of the dipping method was investigated after the application of impregnation with the application. As a result of immersing the sample in epoxy and silane for 10 minutes, the capillary water absorption value was obtained as 0.414 with a standard deviation of 0.10 in epoxy and 0.295 with a standard

Figure 4- Capillary water absorption test results with epoxy applications.

Figure 5- Capillary water absorption test results with silane applications.

deviation of 0.04 in silane. The study determined that the capillary water absorption values of silane were more effective than epoxy. Then, the silane immersion time was changed to 20 and 40 minutes, and the results were examined. However, although the results were slightly better than 10 minutes, it was determined that it was not very effective, with 0.268 with a standard deviation of 0.09 and 0.239 with a standard deviation of 0.17 for the study.

After examining the water absorption and capillary water absorption test results, the ongoing studies on applying epoxy and silane to the surface and immersing the sample in silane for 20 and 40 minutes were canceled. Since the water absorption and capillary water absorption test results made by engaging the samples in epoxy and silane for 10 minutes and then drying them at room conditions gave the best values, another set of these samples was prepared.

To define the resistance of the travertine samples to be used in the study against salt crystallization and to observe the effect of epoxy and silane on salt crystallization, Salt Crystallization Resistance Tests were carried out according to the TS EN 12370 standard. Figures 6, 7, and 8 show the samples immersed in epoxy and silane for 10 minutes before, during, and after the study and the study results.

By the determination of Resistance to Salt Crystallization of Epoxy and Silane, and it was determined that the Average Mass Change (%) of the sample kept in epoxy for 10 minutes was 0.090, the standard deviation was 0.119, the Average Mass Change (%) of the sample kept in silane for 10 minutes was 0.018 and the standard deviation value was 0.021. The results show that the silane-treated samples had higher resistance to salt crystallization. In addition, in the observations made on the samples after the experiment, no decomposition and crack formation were observed due to the exposure of the travertine samples to salt crystallization.

Samples taken from the sample group, immersed in epoxy and silane for 10 minutes and then dried at room conditions, were used to determine the resistance to ${SO}_2$ attrition in a humid environment. The study was carried out according to the TS EN 13919 standard in 2022. A visual of the experiment process is given in Figure 9. In Figure 10, the results obtained from the experiments are given.

Figure 6- Pre-run image of a few of the samples dipped in epoxy and silane.

Figure 7- Visuals of samples dipped in epoxy and silane during and after the experiments.

Figure 8- Results of determination of resistance to salt crystallization of epoxy and silane immersed samples.

Figure 9- Image of the test for the determination of resistance to ageing by SO_2 in a humid environment.

Figure 10- Results of the determination of resistance to ageing by SO_2 in a humid environment of raw, epoxy and silane immersed samples.

On the raw sample in the resistance to ageing by $SO₂$ in a humid environment test, the Average Mass Change (%) in the mixture formed with 150 ml distilled water $+$ 500 ml H_2SO_3 was -0.608, and the standard deviation value was 0.101. In the mixture, it was determined that the Average Mass Change (%) was -0.337, and the standard deviation value was 0.014.

In the study carried out with epoxy and silane for the determination of the resistance to SO_2 attrition in a humid environment, the sample, which was kept in epoxy for 10 minutes, was found in a mixture of 150 ml distilled water + 500 ml H_2SO_3 and the Average Mass Change (%) was -0.293. The standard deviation value was 0.049 in 500 ml of distilled water. In the mixture formed with water $+150$ ml H_2SO_3 , the Average Mass Change (%) was found to be -0.144, and the standard deviation value was 0.182. The Average Mass Change $(%)$ of the sample, which was kept in silane for 10 minutes, was -0.391, and the standard deviation value was 0.138 in the mixture formed with 150 ml distilled water $+$ 500 ml H_2SO_3 , and the Average Mass Change (%) in the mixture formed with 500 ml distilled water $+$ 150 ml H_2SO_3 was found to be -0.391. It was determined that the standard deviation value was -0.158 and 0.159.

It has been seen by the Determination of Resistance to $\rm SO_2$ Attrition in Humid Environment that the epoxytreated samples gave more durable results than the silane-treated samples. This shows that epoxy-treated

surfaces have higher SO_2 resistance. In Figure 11, the image of the raw sample is given before and after the test. As can be seen from the figure, the color of the processed samples is quite dark, while the color of the raw sample is light. This showed that the sample caused a color change by absorbing SO_2 due to its porous structure. However, no effect was observed to disrupt the integrity of any part.

Mercury porosimetry is used for mass density determination by measuring pore size, pore size distribution, and surface area in powder or bulk samples. As a result of the mercury porosimetry analysis, the porosity value of the raw travertine sample was 11.99%, the porosity value of the epoxytreated sample was 6.11%, and the porosity value of the silane-treated travertine was determined as 10.92%. According to the results obtained, it was determined that silane formed a thin layer in the pores of travertine, while epoxy formed a thicker layer, causing the pores to be closed. It has been determined that silane changes the surface area of the

Figure 11- Samples before and after.

raw travertine at a low value. Analysis results of the samples are given in Tables 4, 5, and 6.

Table 4- Mercury porosimetry analysis results of raw travertine sample.

Parameters	Results	
Infiltration into the total volume	0.0520 ml/g	
The total pore area	$0.767 \text{ m}^2\text{/g}$	
Average pore diameter (volume)	$40.3418 \mu m$	
Average pore diameter (area)	$0.0248 \mu m$	
Average pore diameter $(4V/A)$	$0.2711 \mu m$	
Bulk density 0.52 psia	2.3072 g/ml	
Apparent (skeletal) density	2.6216 g/ml	
Porosity	%11.99	
The root volume used	$\%39$	

Table 5- Mercury porosimetry analysis results of the sample kept in silane for 10 min.

Parameters	Results	
Infiltration into the total volume	0.0474 ml/g	
The total pore area	$0.478 \text{ m}^2\text{/g}$	
Average pore diameter (volume)	$47.2065 \mu m$	
Average pore diameter (area)	$0.0382 \mu m$	
Average pore diameter (4V/A)	$0.3966 \mu m$	
Bulk density 0.52 psia	2.3065 g/ml	
Apparent (skeletal) density	2.5893 g/ml	
Porosity	%10.92	
The root volume used	$\%34$	

Table 6- Mercury porosimetry analysis results of the sample kept in epoxy for 10 min.

4. Results

The more porous the natural stone is, the greater the exposure to external factors and the deformation effect. The increase in porosity shortens the natural stone's life due to its tendency to absorb water. It causes the integrity of the stone to deteriorate where it is used (Erdem, 2016; Hasbay and Hattap, 2017; Tayşi, 2021). The water-soluble salts in the natural stone dissolve with the effect of moisture and heat and are transported in capillary cracks, they are recrystallized by the evaporation of water in the new place they are transported, and while these events take place, various deformations such as exfoliation, cavity formation, flowering, crusting occur (Öcal and Dal, 2012; Akbay et al., 2017). For these reasons, the effects of travertines, which are in the natural stone class, were compared by using protective solutions called silane and epoxy, which protect against water, in the study, which was carried out in order to prevent and/or minimize segregation in the places of use depending on the porosity and to eliminate their limitations.

The water absorption value and porous structure of natural stones determine the condition of the stone against external factors that cause deterioration and decomposition. Therefore, the porosity structure of natural stone is a factor that needs to be paid more attention in to geographical regions where the temperature is very low, especially in humid environments. Because water expands in volume during frost, it can cause internal pressures to increase in the cavities and decomposition that begins with the expansion of the void volume. In the study of water absorption at atmospheric pressure, the value of 0.268 obtained in the study performed by impregnating the silane with travertine for 10 minutes gave more effective results than epoxy. In the capillary water absorption study, the value of 0.295 obtained by immersing silane in travertine for 10 minutes gave a more effective result than epoxy.

Salt crystallization is the most damaging event to natural stones. Soluble salts are the main factors that cause disintegration in structures and peeling of surface decorations. Therefore, it needs to be examined carefully. In the study to determine the resistance to salt crystallization, it was determined that the Average Mass Change (%) obtained by impregnating the silane with travertine for 10 minutes was 0.018, and the standard deviation value was 0.021. The results show that the silane-treated samples had higher resistance to salt crystallization. In addition, no decomposition or crack formation was observed when the treated samples were physically examined.

It has been seen by the Determination of Resistance to aging ${SO_2}$ in a Humid Environment that the epoxytreated samples gave more durable results than the silane-treated samples. This shows that epoxy-treated surfaces have higher SO_2 resistance.

In the studies on contact angle measurement, it has been determined that the contact angle of raw travertine gains hydrophobic character by exhibiting a significant increase after the silane coating process.

When the mercury porosimetry analyzes were examined, it was determined that the porosity value of the sample treated with silane was 10.92%. In comparison, the porosity value of the sample treated with epoxy decreased to 6.11%. It has been determined that since the epoxy adheres as a thicker layer on the used surface, it also covers the delicate pores and reduces the porosity value compared to the silane treatment.

When the changes in the characteristic properties of epoxy and silane impregnated with different methods on the travertine surface are examined after the applications, the effects of water absorption in the atmosphere, capillary water absorption, and resistance to salt crystallization showed that silane is more effective in surface protection. On the other hand, in SO_2 wear tests, it has been determined that the protective feature is more effective because epoxy has a denser consistency than silane.

Although the porous structure of travertine is advantageous as a thermal insulation material and decorative product, it is quickly affected by atmospheric conditions due to its porous state, which limits its usage areas. With the study, it was seen that the disadvantageous situations in using natural stone raw materials of sedimentary origin could be turned into an advantageous niche product with high added value, and the value of the study was demonstrated once again.

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