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NON-DESTRUCTIVE METHOD FOR ASSESSING THE STATE OF THE SURFACE STRUCTURE OF SHELL LIMESTONE

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Abstract. The article proposes a method for assessing the state of the surface structure of shell limestone. Shell limestone taken from the wall of a destroyed one-story building in Odessa was used as the test material for analyzing the surface characteristics of the structure. The structural characteristics of shell limestone vary significantly even within a single layer, so to ensure reliable adhesion of restoration materials, it is necessary to take into account the individual characteristics of the surfaces. The material was studied using modern methods of fractal analysis, which make it possible to determine the key parameters of the surface structure. For the study, photographs of the shell limestone surfaces were obtained using electronic macrophotography, which were then processed in the Guiddion software environment, designed for research in the field of scanning probe microscopy. Modern analysis algorithms were used, including image filtering, calculation of fractal dimension and construction of autocorrelation functions. This made it possible to estimate surface parameters such as roughness, texture and fractal properties. Several methods have been selected to determine the fractal dimension, which is an important task in analyzing structures such as reliefs, textures, and functional surfaces. Fractal analysis of shell limestone images was performed using several measurement algorithms – cube counting methods, triangular prisms, power spectrum, and separation (variational). The used method for assessing the state of the shell limestone surface structure allows for the selection of restoration materials with the required physical, chemical, rheological, and strength properties. The proposed approach can be used to adapt restoration compositions to porous substrates in various architectural and historical monuments.

Keywords: fractal analysis, shell limestone, restoration compositions, adaptation, structure, surface, porous materials, substrate.

Introduction. One of the important tasks of construction materials science, especially relevant in cities – centers of historical, cultural and architectural heritage, is to obtain restoration compositions with an optimal set of performance characteristics: adhesive strength, the ability to set and form a composite layer of sufficient strength with low shrinkage, high vapor permeability, preventing moisture accumulation and freezing in winter. In addition, such composite materials are required to have maximum compatibility in terms of basic chemical properties and physical parameters – the material must be maximally adapted to the surface of the base. A common building material that was used in architectural monuments – historical buildings in Italy, France, Greece, Ukraine, etc. is a local material – shell limestone. This porous sedimentary rock is widely used in architecture, especially in regions with abundant marine sediments. Despite its aesthetic appeal and environmental benefits, the low mechanical strength of shell limestone and its high susceptibility to weathering pose significant challenges in ensuring the durability of buildings and structures made from this stone. Therefore, over time, historical buildings need restoration, reconstruction and preservation, and the choice of composite materials used is a pressing issue in these processes.

Careful selection of compositions of restoration composite materials for shell limestone buildings requires special approaches. The main task of such composites is to strengthen structural elements while preserving their authenticity and historical value. At the same time, restoration compositions should have good adhesion to the base material (substrate) while maintaining physical and aesthetic integrity over time, that is, ensure sufficient adhesion of the composite and the substrate – shell limestone. The development of methods for selecting optimal compositions of such composite materials to ensure the main performance characteristics is an important and urgent task. A rational approach to the selection of such compositions is a multifaceted and multi-criteria task, in the solution of which an important aspect is related to the study of the surface structure of the substrate to ensure maximum adaptation of restoration composites to the base material.

Analysis of recent research and publications. The main requirement for restoration materials when restoring stone surfaces is to ensure maximum compliance and authenticity both in appearance and in chemical composition, physical and operational properties [1, 2]. Therefore, it is necessary to conduct a thorough chemical and physicochemical analysis of the material being restored, in particular, its surface, for an informed decision when determining the restoration composition.

It should be noted that when restoring the surfaces of stone materials, it is necessary to form a thin intermediate layer, which ensures maximum adaptation of the main volume of the composite material to the restored surface [3]. In this case, conditions are created for even greater adaptation of the main volume of the restoration composite. Therefore, it is important to ensure contact of high adhesive strength between the intermediate restoration layer and the stone, for example, shell limestone, taking into account the high degree of structural heterogeneity of its surface.

A prerequisite for the manifestation of high adhesive characteristics is the ability of the adhesive to fill the main elements of the surface structure – pores of all types, irregularities, surface roughness, cracks and fissures [4]. At the same time, to obtain high adhesive strength of the "adhesive-substrate" contact, it is necessary to ensure the maximum contact area of the base material and the restoration composite. It should be noted that it is necessary to take into account the individuality of the structure of each specific restored area of shell limestone [5] when solving such problems.

The study of the surface structure of restored products, in particular shell limestone, is of interest for the targeted selection of a primer intermediate composition, as well as a restoration composite, in order to ensure chemical, physical and structural compatibility with the porous irregular surface of the shell limestone and adaptation to it.

Fractal geometry as one of the approaches to the description of complex surfaces has been widely used in the study of natural and artificial materials [6-8]. Shell limestone, which is a porous sedimentary rock, exhibits a multi-level structure that is well described by fractal models [9].

It should be noted that there are several types of display and study of fractal-like objects [10]. Often, a boundary or edge fractal is studied, determined by the nature of the boundary surface of the structure being studied. Another option for studying self-similar structures is their representation by a mass fractal associated with the distribution of particles with a certain mass index in space. The micrographs of materials studied in this case are fractal surfaces, the third coordinate of which corresponds to the intensity of the corresponding pixels in the electronic image. Determining the fractal dimensions of surfaces, where intensity acts as the third coordinate, is an important task in the analysis of structures such as reliefs, textures and functional surfaces.

Let us consider some methods for determining fractal dimension in image analysis.

The Box-counting method [11] is designed to estimate the fractal dimension of images considered as three-dimensional surfaces, where the brightness of each pixel is interpreted as height. This approach is useful for analyzing structural properties of complex surfaces, such as roughness or porosity, and relates them to scaling characteristics.

The method is represented by the following algorithm. A two-dimensional image of size $M \times M$ converted to three-dimensional space (*x, y, z*), where (*x, y*) – pixel coordinates, *z* – pixel brightness level. The image base is divided into a uniform grid with a step *s*, where is the cell size $s \times s$ is selected so that M / 2 > s > 1. Above each cell, a column is constructed consisting of parallelepipeds with a height calculated from the ratio $\frac{C}{s_z} = \frac{M}{s}$, where C – number of gradations of image brightness.

For each cell the parameter is calculated $n_s(i, j) = z_{\max} - z_{\min} + 1,$ where $z_{\max}$ and $z_{\min}$ – maximum and minimum brightness in the current cell. Next, summing up the values $n_s(i, j)$ for all grid cells, get the number of filled cells for the current size *s*. For different values of *s*, calculate N_s, which allow you to build a dependency *log* (N_s) from *log(s)*. The slope of the regression curve is used to determine the fractal dimension $D = -\tan(\alpha)$, where α – slope angle of a linear section on a graph *log* (N_s) from *log(s)*. The fractal dimension is determined by the formula (1):

$$D = -\lim_{\text{т} \to 0} \frac{\log N(\text{т})}{\log \text{т}}. \tag{1}$$

The triangulation (triangular prism) method [12] is used to calculate the fractal dimension of complex surfaces by analyzing the dependence of the surface area on the grid step size. It starts with representing the surface as an image, where the brightness of each pixel is interpreted as the height at the corresponding point. The image is covered with a grid with a step size s, each cell of which includes four points forming a base. For each cell, two diagonals are drawn, the intersection of which defines the central point. The height of this point is calculated as the arithmetic mean of the heights of all four nodes of the cell. Next, four triangles are formed from the cell points, which form the upper part of a polyhedron approximately corresponding to the real topography of the surface. For each triangle, the area is calculated using the standard vector formula through two of its edges. The total area of all triangles gives an estimate of the surface area for a given cell. This process is repeated for all cells of the grid, and the final sum of the areas serves as an estimate of the surface area of the image for a given step size. To calculate the fractal dimension, the method is repeated with different values of s, and the dependence of the surface area on the step is recorded. A logarithmic dependence of the area on the step, known as the Richardson curve, is constructed based on the data obtained. The slope of this dependence on the linear section corresponds to the parameter B, from which the fractal dimension is calculated using the formula $D = 2 - B$. Thus, the triangular prism method allows for an accurate assessment of the fractal characteristics of complex surfaces, for example, to analyze the porosity or roughness of materials.

The idea of the variational method [13] for determining the fractal dimension is to estimate the degree of change of the surface depending on the sample scale by calculating the variational measure *V(r)*. For this purpose, the surface is divided into cells with a linear size *r*. For each cell, the deviation of the intensity values $z = f(x, y)$ from the average value in the cell is calculated (2):

$$V(r) = \sum_{cell} \left| f(x, y) - \bar{f} \right|. \tag{2}$$

Change the scale *r* and repeat the calculations, then plot the dependence graph *log V(r)* from *log r*. The fractal dimension is calculated using the formula (3):

$$D = \lim_{r \to 0} \left(3 - \frac{\log V(r)}{\log r} \right). \tag{3}$$

The Power Spectrum Method [14] is based on the analysis of the power spectral density *S(k)* of the surface at spatial frequency *k*. To do this, the following successive steps are carried out:

1. Calculate the two-dimensional Fourier transform of a surface *f(x, y)* (4):

$$F(k_x, k_y) = \iint f(x, y) e^{-i(k_x x + k_y y)} dxdy. \tag{4}$$

2. Determine the power spectral density (5):

$$S(k) = |F(k_x, k_y)|^2, \tag{5}$$

from $k = \sqrt{k_x^2 + k_y^2}$.

3. Build a dependency *log S(k)* from *log k*.

4. Calculate the fractal dimension using the formula (6):

$$D = \frac{7-\beta}{2}, \qquad (6)$$

where β – slope of the linear section of the graph *log S(k)* from *log k*.

The methods discussed above allow us to evaluate the fractal properties of shell limestone surfaces, which are the basis for the next stage of rational selection of the composition of restoration materials. Their use in the study provides a comprehensive approach to the analysis of the cellular structure, taking into account the multi-level nature and uniqueness of each sample. Thus, Box counting and Power Spectrum are effective for integral assessment, while Triangulation and Partitioning are better suited for local analysis. This allows us to combine average characteristics with local features, which meets the challenges of developing optimal compositions of composite materials for restoration.

The purpose of the work is to develop a non-destructive method for assessing the state of the surface structure of shell limestone.

Materials and methods of research. A sample of shell limestone taken from the wall of a one-story building being destroyed in Odessa was used as the source material. The surface of its natural chip and cut was examined.

The research methods were based on obtaining photographs by electronic macrophotography and their subsequent processing in the software environment of the Guiddion system, designed for research in the field of scanning probe microscopy. In particular, filtration modules, fractal dimension calculation and autocorrelation function were used.

Various methods of fractal analysis were used in the work: Box counting (cube counting method), Triangulation (triangular prism method), Power Spectrum (power spectrum method), Partitioning (variational).

Research results. The study is the primary stage of the materials science analysis of historical materials with a porous structure for the further application of restoration methods to products made from them, for example, structural elements of buildings of the cultural heritage of Odessa and other cities. The main property of primers and restoration compositions is effective adhesive interaction with the substrate, which requires an individual selection of the compositions of the corresponding composites for each architectural restoration project. The compositions under consideration should be distributed over the surface of the shell limestone in such a way as to ensure the maximum contact area of the "adhesive-substrate". Therefore, certain requirements are imposed on the physicochemical, rheological characteristics, the nature of the distribution of particles in the adhesive composition and its ability to effectively harden within a specified time. The considered set of properties can be characterized as the ability to geometrically and physic mechanically adapt the adhesive to the substrate.

An intermediate stage in the sequence of each restoration project is the assessment of the condition and structure of the surface being restored. Such an assessment can be carried out using several methods, one of which (fractal analysis) has an obvious physical meaning and is associated with the characteristic self-similar properties of the porous materials being restored. In terms familiar to restoration specialists, the determined fractal dimension Df describes the degree of structural heterogeneity (relief, jaggedness and roughness) of the surface: the greater the value of Df – 2, the more pronounced are the corresponding geometric properties. Fractal analysis allows for a quantitative assessment and differentiation of the considered structural characteristics of samples of visually similar surfaces. In the future, the obtained values of fractal characteristics will allow for an indirect characterization of the requirements for primers and restoration compounds.

The effectiveness of intermediate primers can be studied from a geometric point of view, namely, by assessing how effectively the surfaces under study are “smoothed” when they are used. Thus, for effective primers, the difference in fractal dimensions of the initial and processed surfaces, $D_f^{init} - D'_f$, should be maximum with the necessary consideration of additional conditions and restrictions, which can be a criterion for choosing a composition from several. It is also necessary to note the adaptive nature of the interaction during the formation of the considered types of adhesive contacts.

Thus, the adhesive composition of the required properties can be approximately selected based on knowledge of the fractal dimension of the substrate, which, together with the Richardson curves, which also characterize multifractal properties [7, 15], can be determined based on the results of processing the images of the material.

In our work, the calculation of fractal characteristics was implemented using the Guiddion program, designed for processing images obtained on nano-technological instruments – scanning probe microscopes. However, the internal functionality is also well suited for analyzing images of a conventional nature.

For the analysis, images of shell limestone chips from different areas of the same sample stone were selected (Fig. 1).

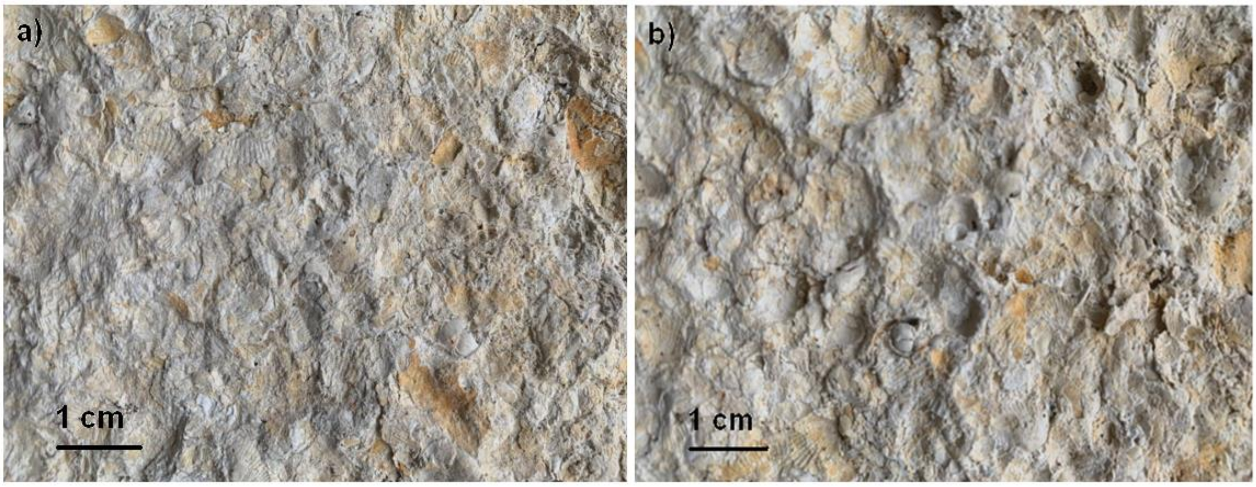


Fig. 1. Photo of the surface of shell limestone: a – section 1; b – section 2

The image was converted to grayscale and subjected to median filtering. After that, the above-described algorithms and methods of fractal analysis were applied. As a result of stepwise change of the cell scale or similar steps, mappings equivalent to Richardson curves were obtained. A linear approximation was constructed for the corresponding methods.

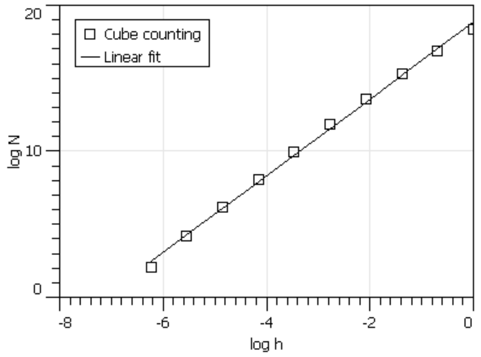
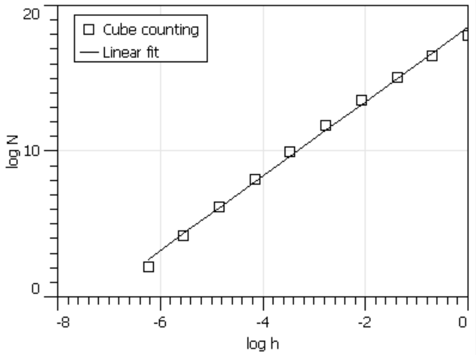
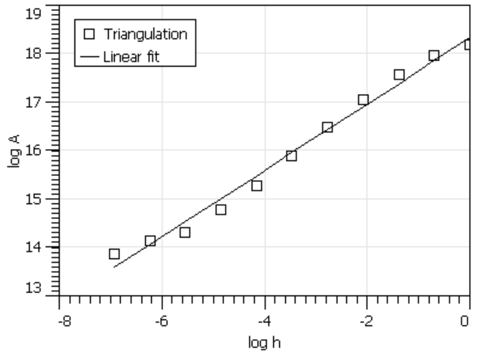
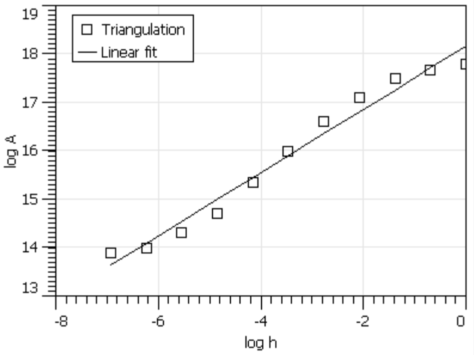
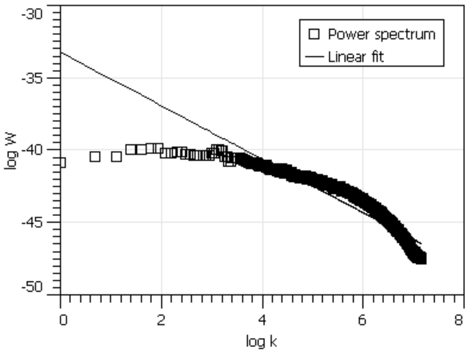
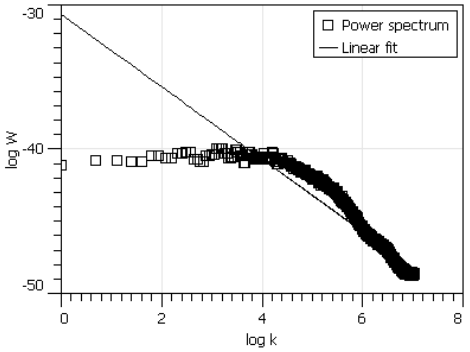
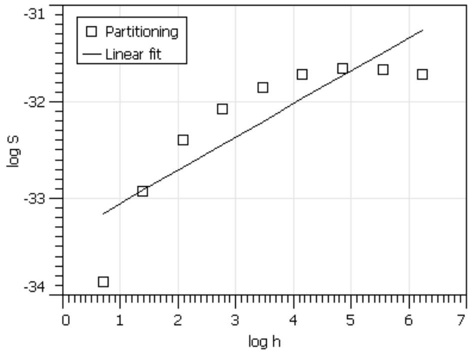
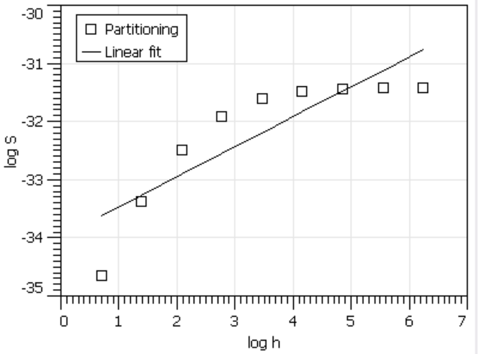
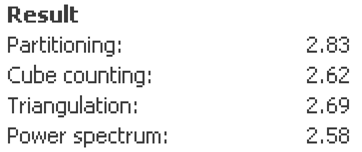
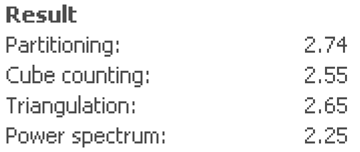
The results of the study and the corresponding values of fractal dimensions are given in Table 1.

The obtained data allow us to approximately characterize the degree of structural heterogeneity of the studied samples. Each of the values obtained as a result of fractal analysis (fractal dimension 2.69 and 2.55) indicate a significant difference between the shell limestone surface and classical mathematical surfaces, which allows us to draw a conclusion about the three-dimensional structure of the studied surfaces. Differences in fractal dimensions indicate different degrees and nature of structural heterogeneities of the surface of the analyzed material even within the same sample. Shell limestone, being a sedimentary rock, was formed and transformed under different physical conditions – significant gradients of flows and concentrations of mineral components, which could be the reason for the differentiated nature of the structural organization.

The requirement for the primer and main restoration composition to ensure maximum contact with the restored surface can be represented by the following geometric condition. Let's consider the "excess" fractal dimension $\Delta D_f^{substrat}$ equal to the fractional part of the corresponding number (for example, 0.69 for a fractal dimension of 2.69). Then the corresponding composition will geometrically adapt to the surface, filling pores and voids until its "excess" fractal dimension complements the "excess" fractal dimension of the material. $\Delta D_f^{adhesiv}$ up to 1, which corresponds to the expression (7):

$$\Delta D_f^{substrat} + \Delta D_f^{adhesiv} \approx 1. \tag{7}$$

Table 1 – Results of fractal analysis of shell limestone images

Measurement algorithm	Section 1 shell limestone (a)	Section 2 shell limestone (b)
Box counting		
Triangulation		
Power spectrum		
Partitioning (variation)		
Fractal dimension	**Result** Partitioning: 2.83 Cube counting: 2.62 Triangulation: 2.69 Power spectrum: 2.58	**Result** Partitioning: 2.74 Cube counting: 2.55 Triangulation: 2.65 Power spectrum: 2.25
Average fractal dimension	2.68	2.55

Strict equality in this case corresponds to the formation of an ideal adhesive contact, which forms a three-dimensional structure together with the substrate and the adhesive. In this case, the structural heterogeneity on the surface of the primer layer decreases. Approaching the ratio (7) is an example of the adaptation of the adhesive to the geometric structure of the surface of the porous material.

Conclusions. Thus, in the work, a non-destructive method for assessing the state of the surface structure of shell limestone was substantiated and applied based on the fractal characteristics obtained by appropriate processing of images of this surface. The obtained values of the fractal dimension for different areas of the surface indicate its significant structural heterogeneity. Quantitative results of the fractal analysis can be used to select the composition of the primer layer, and subsequently the main restoration composite, with the required structural parameters, as well as physicochemical and rheological properties.

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НЕРУЙНІВНИЙ МЕТОД ОЦІНКИ СТАНУ СТРУКТУРИ ПОВЕРХНІ КАМЕНЮ-ЧЕРЕПАШНИКА

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Анотація. У статті запропоновано метод оцінки стану структури поверхні каменю-черепашника. Як досліджуваний матеріал для аналізу поверхневих характеристик структури використано черепашник, взятий зі стіни одноповерхової будівлі, що руйнується, в місті Одеса. Структурні характеристики черепашника суттєво варіюються навіть у межах одного шару, тому для забезпечення надійної адгезії реставраційних матеріалів необхідно враховувати індивідуальні особливості поверхонь. Матеріал досліджено з використанням сучасних методів фрактального аналізу, що дозволяють визначити ключові параметри структури поверхні. Для дослідження були отримані фотографії поверхонь черепашника за допомогою електронної макрозйомки, які потім оброблялися в програмному середовищі системи Guiddion, призначеного для досліджень в області зондової скануючої мікроскопії. Були застосовані сучасні алгоритми аналізу, включаючи фільтрацію зображень, розрахунок фрактальної розмірності та побудову автокореляційних функцій. Це дозволило оцінити параметри поверхні, такі як шорсткість, текстура та фрактальні властивості. Для визначення фрактальної розмірності, що є важливим завданням при аналізі структур, таких як рельєфи, текстури та функціональні поверхні, підібрано декілька методів. Фрактальний аналіз зображень черепашника проводився з використанням кількох алгоритмів вимірів – методів рахунку кубів, трикутних призм, спектра потужності, поділу (варіаційного). Використовувана методика оцінки стану поверхні каменю-черепашника дозволяє надалі забезпечити можливість підбору матеріалів для реставрації з необхідними фізико-хімічними, реологічними та властивостями міцності. Пропонований підхід може бути використаний для адаптації реставраційних складів до пористих основ у різних архітектурних та історичних пам'ятках.

Ключові слова: фрактальний аналіз, черепашник, реставраційні склади, адаптація, структура, поверхня, пористі матеріали, субстрат.

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