Доведено ефективність попереднього модифікування каолінів 3-амінопропілтриетоксисиланом з метою покращення властивостей наповнених водно-дисперсійних фарб, таких як водопоглинання, міцність на розрив, стійкість до дії агресивних середовищ. Досліджено зміни фізико-хімічних та адсорбційних властивостей каолінів в результаті модифікування. Встановлено особливості процесів взаємодії між немодифікованими та модифікованими каолінами зі стирол-акриловим плівкоутворювачем

Ключові слова: водно-дисперсійна фарба, каолін, модифікування, стирол-акриловий плівкоутворювач, 3-амінопропілтриетоксисилан

Доказана эффективность предварительного модифицирования каолинов 3-аминопропилтриэтоксисиланом с целью повышения свойств наполненных водно-дисперсионных красок, таких как водопоглощение, прочность на разрыв, устойчивость к агрессивным средам. Исследованы изменения физико-химических и адсорбционных свойств каолинов в результате модифицирования. Установлены особенности процессов взаимодействия между немодифицированными и модифицированными каолинами со стирол-акриловым пленкообразователем

Ключевые слова: водно-дисперсионная краска, каолин, модифицирование, стирол-акриловый пленкообразователь, 3-аминопропилтриэтоксисилан

UDC 667.621.2:620.2
DOI: 10.15587/1729-4061.2016.79358

ANALYSIS OF PROPERTIES OF MODIFIED KAOLINS AND WATER-DISPERSION PAINTS DEVELOPED ON THEIR BASE

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1. Introduction

In economically developed countries of the world (EU, the USA, Canada, etc.), scientists and specialists in different areas operate by such concepts as "sustainable development" and "quality of life", focusing on the need to move to a qualitatively new level of consumption, which is impossible without development and implementation of eco-friendly and safe goods. Due to the need to adapt legislation of Ukraine on goods quality and safety to the EU requirements, the priority direction of development is to minimise the negative effect on health of consumers and the environment. Particularly acute this issue is for the goods of paints and coatings industry [1, 2]. For many years international community has paid considerable attention to the environmental aspects of manufacturing and the use of paints and coatings, which has led to a steady growth in the volumes of production and application of water-dispersion paints in the world. In Ukraine, the process of transition from the production of solvent-based to water-dispersion paints has also been noticeably intensified over recent years. Every year sees the growing interest towards development of water-dispersion paints using local raw materials that would ensure high quality and competitiveness of paints in the domestic and foreign markets. Considerable attention is paid to white mineral fillers, the use of which in paints formulation significantly reduces the cost of products [3]. It is known that the degree of interaction between a filler and a film former plays important role in the formation of properties of water-dispersion paints [4, 5]. Therefore, the studies aimed at searching for new and improving of the known solutions to increase compatibility of fillers with film formers that will promote provision of high level of their chemical interaction and improve the properties of paints and coatings, are relevant.

2. Literature review and problem statement

The most promising fillers for water-dispersion paints in Ukraine are considered to be kaolins, because by the volumes of production they occupy a leading position among the mineral fillers. However, the volumes of their use in the manufacture of water-dispersion paints and coatings remain low, which is caused by insignificant activity of interaction between kaolins and polymers on the border of phases' separation [6].

Scientists in many countries of the world solve the issue of activating the mineral fillers by modification [7, 8]. The results of such research are mostly used in the production of plastic masses [9], rubber [10, 11], ceramic products [12], the pulp and paper industry [13]. There are known methods of the fillers treatment by fatty acids [14], quatermary ammonium salts [15], silans [16–18], cationic [19], anionic [20, 21] and nonionic genic SAS [22]. Papers [21, 23–25] are devoted to research into the problems of the use of kaolins in the composition of paints, in which it is proposed to implement the addition of modifiers directly to a paint and coating composition. In this case, however, they do not take into account a possible competing role of other components of the dispersion medium of the paint in the process of fixing the modifier on the surface of the filler. With regard to such a possibility, one should consider as more appropriate and promising the method of pre-modification of the kaolins surface by SAS in order to maximize the implementation of their potential opportunities as a part of water-dispersion paints, in particular due to the change in physical and chemical properties of the fillers. As a result of addition of pre-modified kaolins to the composition, it is possible to achieve the increase in polymerphilicity of their surface and, consequently, to regulate the processes of interaction in the system of kaolin – film former – water, which impact directly the formation of properties of water-dispersion paints.

3. The aim and objectives of the study

The aim of this study is the research into properties of kaolins modified by 3-aminopropyltriethoxysilane and water-dispersion paints based on them.

To achieve this goal, it is necessary to solve the following tasks:

– to assess the effect of modifying on the physical and chemical properties as well as adsorbing properties of kaolins;

– to explore the processes of interaction in the systems of kaolin – modifier – film former and describe their patterns;

– to conduct a comparative analysis of the properties of water-dispersion paints filled with modified and non-modified kaolins.

4. Materials and methods of study of the effect of modified kaolins on the properties of facade water-dispersion paints and coatings

4. 1. The studied materials and equipment used in the experiment

Water-dispersion paints were received by traditional technology [4, 26, 27], except the pre-modification of kaolins. That is, technological process of paints manufacturing included the following stages.

Pre-modification of kaolins was carried out; with this aim, kaolins and 3-aminopropyltriethoxysilane were introduced to the bead mill by the calculation of 0.25 % of the mass of fillers and were dispersed for one hour.

For the manufacture of water-dispersion paints, water, thickener and dispersant were added gradually to the dissolver, which were mixed for 10 min at rotations of 450 r/min to a homogeneous state. After that, they added biocide and a defoamer and mixed for 1 min at the speed of 300 r/min. Then the speed was increased up to 700–800 r/min with consequent adding titanium dioxide and filler. Volume concentration of pigments and fillers (VCP) was varied in the range between 40 and 55 mass % (with the increment of 5 mass %). The received mixture was rubbed for 20 min. After that, the speed was decreased to 300 r/min and a film former and a coalescent were added and homogenized for 5–7 minutes up to the formation of homogeneous mass.

The water dispersion of the styrene-acrylic co-polymer *Osakryl OSA S20* (Poland, *Synthos S. A.*) was chosen as the film former. Kaolins from the largest deposits of Ukraine (Glukhovetskiy and Prosianivskiy) of the KC-1 mark, modified by 3-aminopropyltriethoxysilane, were used as the filler. We also used the white pigment – titan dioxide of the *Crimea TiOx-230* mark, and certain other functional additives such as a dispersant (ester of fatty acid *Tanemul DA 130),* the share of which is 1 mass % of mineral phase; the coalescent (dipropylene glycol mono n-butyl ester *Dowanol DPnB)* in the amount of 10 mass % of the polymer content; the defoamer (fatty acids of derivatives of esters and hydrophobic components WS 938), the thickener (hydropropylmethylcellulose *Joincel MK50MS)* and biocide (*Vincoside CMIF*).

4. 2. Method of determining indicators of properties of kaolins and water-dispersion paints

The properties of kaolins, water-dispersion paints and coatings were determined using the complementary methods of physical and chemical analysis of fillers.

The microstructure of kaolins was studied by the electronic microscopy method, using the raster electronic microscope JSM-6700F with the energy dispersion system for microanalysis JED-2300 (Jeol, Japan). Specific effective surface and wetting by polar (water) and non-polar (benzene) fluids were determined by the method of capillary impregnation. The adsorption of polymer and water was investigated by the method of liquid chromatography.

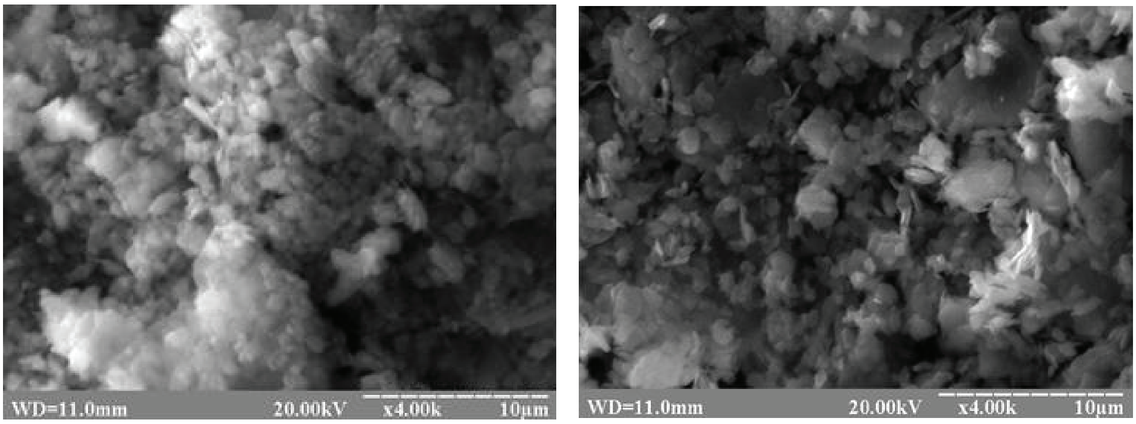
The study of chemical interaction in the systems of kaolin-modifier, kaolin-film former, modified kaolin-film former was carried out using the method of IR spectroscopy. Analysis of the processes of interaction in these systems was made using the spectrometer Fourier Avatar 370 PT-TR (USA, Termo Nicolet) in the range from 400 to 4000 cm^{-1}.

Tensile strength was determined on free films by the device TT-1100. Bending elasticity was determined using sets of metal rods of different diameters. The wetting edge angle of coatings was defined using the complex OCA 15EC with the help of video measuring system and computer program for processing of the received data SCA 20 (Germany, DataPhysics Instruments GmbH). Test for water absorption of coatings was carried out by determining the amount of water vapor, which passed through the free paint and coating film, resistance to static effect of water and aggressive media (the 3 % HCl and NaOH solutions) was defined by the method of immersion.

5. Results of research into properties of kaolins and water-dispersion paints filled with modified kaolins

Kaolins were given advantage for improving the barrier properties of coatings of facade water-dispersion paints because, due to the lamellar shape (Fig. 1), their particles are able to provide passive corrosive protection, prolonging the path of diffusion of corrosion agents.

The wetting of kaolin from Glukhovetskiy deposit by the water flow is 0.024, from Prosianivskiy deposit is 0.058. The wetting by the benzene flow is larger and equals 0.194 and 0.236, respectively. Specific effective surface by water for the kaolin from Gluhovetskiy deposit is 36.0 m^2/g, by benzene is 17.0 m^2/g. For the kaolin from Prosianivskiy deposit, these indicators equal 49.6 and 18.7 m^2/g, respectively. The modification of fillers by 3-aminopropyltriethoxysilane leads to the increase in wetting and effective surface by benzene, whereas these values by water are somewhat decreased (Table 1).



a *b*

Fig. 1. Microstructure of kaolins of the KC-1 mark: *a* — from Glukhovetskiy deposit; *b* — from Prosianivskiy deposit (Ukraine)

Table 1

Physical and chemical adsorption properties of non-modified and modified kaolins

Deposit	Wetting by flow		Specific effective surface, m^2/g		Specific mass adsorption, mg/g	
	Water	Benzene	Water	Benzene	Polymer	Water
Non-modified kaolins						
Glukhovetskiy	0,024	0,194	36,0	17,0	162	147
Prosianivskiy	0,058	0,236	49,6	18,7	193	154
Kaolins modified by 3-aminopropyltriethoxysilane						
Glukhovetskiy	0,014	0,219	26,6	21,5	214	107
Prosianivskiy	0,035	0,259	47,9	19,8	239	123

Specific effective surface by benzene of the kaolin from Glukhovetskiy deposit is increased to 21.5 m^2/g, from Prosianivskiy deposit – up to 19.4 m^2/g, as a result of which adsorption properties of kaolins undergo changes as well. The adsorption of styrene-acrylic polymer by the kaolin from Glukhovetskiy deposit grows by 32 % and by the kaolin from Prosianivskiy deposit – by 23.8 %. Specific effective surface of kaolins by water is decreased, so adsorption of water by the kaolins is decreased as well by 37.4 % (Glukhovetskiy deposit) and by 25.2 % (Prosianivskiy deposit). That is, as a result of kaolins modification, their polymer philicity is increased.

Analysis of IR spectra confirms that the most tangible changes in the process of modifying and forming the paint and coatings systems occur in parameters of the bands responsible for valence fluctuations of the OH-groups, adsorbed water and Si–O–Al bonds in kaolins composition.

It is found that when modifying the kaolin from Prosianivskiy deposit, the decrease in the intensity of absorption bands is observed in the range of 3657–3652 cm^{-1}, which are responsible for valence fluctuations of the OH-groups and adsorbed water (Fig. 2, *a, c*).

The amount of adsorbed water in the kaolin composition from Prosianivskiy deposit when modifying by 3-aminopropyltriethoxysilane decreases to 35 %, and when using styrene-acrylic film former, this indicator reaches 55 %.

In the case of Glukhovetskiy kaolin (Fig. 2, *b, g*), the intensity of the main spectral characteristic bands of absorption is 25–30 % less in comparison with Prosianivskiy kaolin.

Hydroxyl groups of the kaolins surface also play a crucial role in the processes of interaction in the systems kaolin – modifier – film former as well. In the case of kaolin from Prosianivskiy deposit, the intensity of the respective bands is decreased to 25 %, in the case of kaolin from Glukhovetskiy deposit, the magnitude in decrease of the absorption bands in the systems of kaolin – 3-aminopropyltriethoxysilane and of kaolin – 3-aminopropyltriethoxysilane – film former is in the range to 10 cm^{-1}.

A special feature of the application of 3-aminopropyltriethoxysilane and styrene-acrylic film former is the occurrence of a number of new bands (2360–2361, 2925–2928 and 1731 cm^{-1}) of different intensity on the IR spectra, caused by the presence in their composition of different functional and C–H groups (Fig. 2, *d, e*). The interaction of kaolin with the modifier and the polymer involving the Si–Al–O bonds (absorption bands 779–793 and 740–755 cm^{-1}) is not excluded either. The confirmation of this is the decrease in their intensity and shift relative to the base position (Fig. 2, *d, e*).

Increase in the interaction of kaolins with the film former due to the modification allowed decreasing the values of water absorption by paint and varnish coatings, which at the VCP of 40–55 mass % are in the range of 8.1–9.7 mass % (Fig. 3). This will ensure reliable protection of walls of the buildings because the coatings do not let the water in at condensed phase.

One of the most important problems in building sector is the increase in the durability of structures, reducing the costs for their operation and repair. In general, these problems are mainly related to the negative effect of water. The increased resistance of coatings to the effects of water is also confirmed by high edge angles of wetting, which are in the range of 88–95 degrees. The developed water-dispersion paints, due to the addition of modified kaolins to their composition, provide for the formation of coatings with high resistance to the effects of external factors and retain their protective properties under static action of water, the 3 % NaOH and HCl solutions up to 72 hours. The obtained results of the study of resistance of coatings to the static effect of these liquids are correlated with the data on their water absorption. For the coatings that show larger resistance to active aggressive environments, the lower water absorption is characteristic.

It was found that the coatings of paints filled with modified kaolins have larger tensile strength. The value of this indicator when using modified fillers is increased by 18–50 % and reaches maximum at VCP of 55 mass % (Fig. 4).

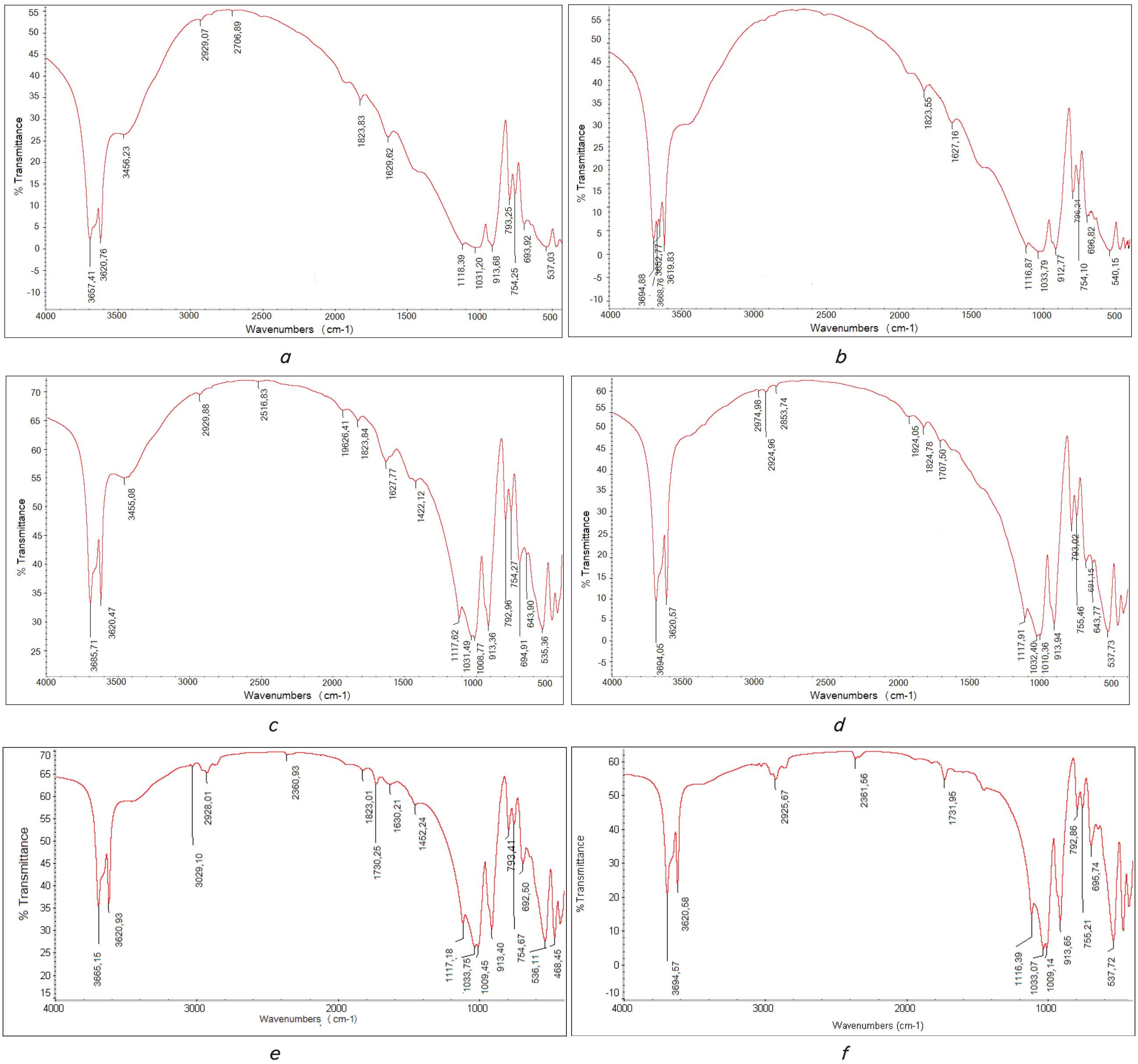


Fig. 2. IR spectra of KC-1 kaolins from Prosianivskiy and Glukhovetskiy deposits: *a, b* — non-modified; *c, g* — modified by 3-aminopropyltriethoxysilane; *d, e* — modified by 3-aminopropyltriethoxysilane with styrene-acrylic film former

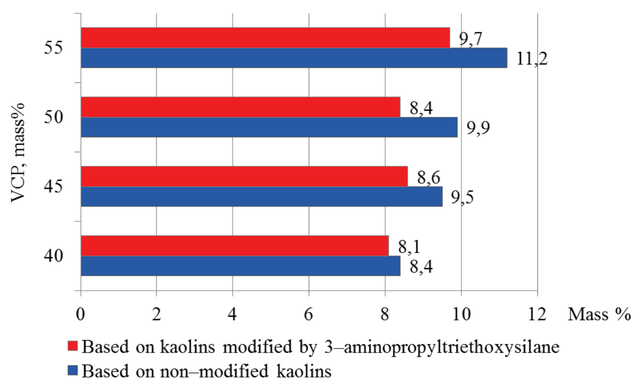


Fig. 3. Moisture absorption by coatings of water-dispersed paints at different VCP

Paints with modified kaolins have increased elasticity of coatings that equals 2 mm at VCP 40–50 mass % and 4 mm at VCP 55 mass %, whereas for paints with non-modified kaolins 4 and 6 mm.

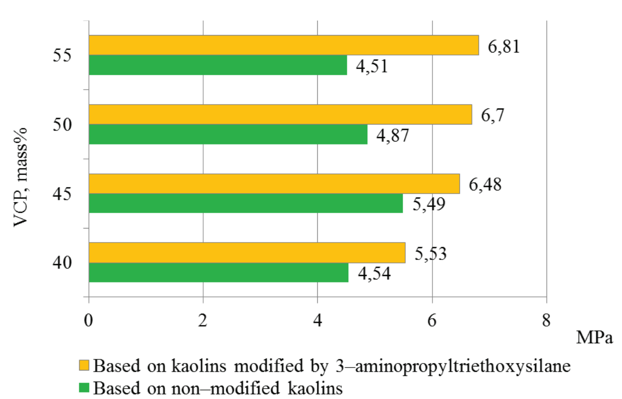


Fig. 4. Tensile strength of coatings of water-dispersed paints at different VCP

6. Discussion of results of the study of effect of modified kaolins on enhancing the properties of water-dispersion paints and coatings

Conducted studies confirmed that a significant role in the interaction of the film former with the filler is played by such factors as wetting the surface of the filler by the polymer and formation of chemical bonds between them. It was found that the process of modification of kaolin proceeds with the change in specific effective surface and wettability. The change in these physical and chemical properties improves the interaction of kaolins with the film former. It is determined that the processes of interaction between the modified kaolins and a styrene – acrylic film former proceed with the participation of hydroxyl groups, adsorbed water and the Si–O–Al bonds of fillers. Application of 3-aminopropyltriethoxysilane helps to reduce the intensity of absorption bands, which are responsible for valence fluctuations of the OH–groups and adsorbed water, which indicates the improvement of kaolins compatibility with the film former.

It is found that the increase in polymeric philicity of kaolins due to modification of their surface by 3-aminopropyltriethoxysilane allowed reducing the water absorption of paint and varnish coatings to 8.1–9.7 %. Paints and coatings based on modified kaolins also show higher resistance to static corrosive environments, increasing their hydrophobic properties. Modified kaolins reinforce polymer matrix, increase the film tensile film by 18–51 %, depending on the degree of filling.

The obtained scientific results may be used and implemented at the enterprises of paints and coatings industry, in companies of building sector, as well as in the scientific and research organizations and educational process at universities that provide training for specialists in the field of commodity research of non-food products as well as in chemical and building sectors of industry.

The drawbacks of the study include a quite narrow selection of fillers (only kaolins of the KC-1 mark were examined) and the use of only type of the film former that might be considered in future studies, devoted to the development of new facade water-dispersion paints for exterior.

7. Conclusions

1. It is determined that modifying the kaolins surface leads to changes in their physical and chemical properties: wettability and specific effective surface is increased by non-polar liquid (benzene) and is somewhat reduced by the polar one (water). The pre-modification improves compatibility of kaolins with non-polar styrene-acrylic film former, which manifests itself in the increased adsorption of polymer by fillers.

2. It is found that the processes of interaction between the modified kaolins and styrene-acrylic film former proceed with the participation of hydroxyl groups, adsorbed water and the Si–O–Al bonds of kaolins.

3. It is proved that the addition of modified kaolins to the composition of water-dispersion paints increases tensile strength of the coatings by 18–51 %, improves their elasticity to 2–4 mm, edge angle of wetting by water to 89–95 degrees and resistance to aggressive media up to 72 hours, as well as reduces water absorption by 10.4–17.9 %.

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