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BIFUNCTIONAL ORGANOSILICON COMPOUNDS AND THEIR POTENTIAL APPLICATION

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A B S T R A C T

Biological corrosion refers to the undesirable deposition and growth of microorganisms, such as fungi, bacteria and algae. This multistage, complex process leads to the destruction of the materials on which the deposition and growth, in the form of a biofilm, took place. This phenomenon not only causes technological problems, but can also have harmful effects on human health. This phenomenon is most often discussed in the context of construction materials, such as bricks, concrete, roof tiles, glass, and wood. This publication presents a synthesis of functionalised organosilicon compounds and their selected surface properties.

1. INTRODUCTION

Advanced materials are a kind of driving force for the development of human society, and this progress constantly requires new materials with specific properties. Based on the results of basic research, materials with specific structures and parameters are designed. Research on compounds with amphiphilic properties is a very dynamically developing field of scientific research, which combines research in the fields of chemistry, physics, and biology, and at the same time fits perfectly into the current trends (Kashapov et al., 2020; Ghosh et al., 2020).

Thanks to their unique structure, silicon polyethers undoubtedly make one of the most important groups of modified polysiloxanes (Liu & Costanzo, 2019). They are composed of a siloxane chain, which in addition to hydrophobic methyl groups, also contains hydrophilic polyether groups (Tan et al., 2018; Fu et al., 2019). The specific structure of organosilicon compounds with amphiphilic properties allows them to exhibit surface properties. Therefore, they can be used to obtain antifouling or self-cleaning protective coatings, e.g. for wood, glass or other building materials, Figure 1.

Figure 1. An example of the occurrence of biological corrosion on serviceable surfaces

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The amphiphilic properties of organosilicon compounds and their low toxicity are a friendly alternative in the fight against biological contamination, the so-called biofouling (Khan et al., 2015; Guazzelli et al., 2020; Adamopoulos et al., 2021). Silicone polyether surfactants also have a bacterial effect (Hawkins et al.. 2014; Hawkins et al., 2017). Therefore, they are used, for example, to obtain bacterial cellulose (Szymańska et al., 2022) or antifouling agent (Lagerström et al., 2022; Stafslien et al., 2015). Taking into account the above facts, it is interesting how organofunctional siloxane will affect the biocorrosion process. The work presents a completely different direction of applications of silicon-polyether polyethers. In the presented study, the effectiveness of a silicon polyether coatings of glass against microbiological colonization was assessed using the microalgae as a key component of widespread aerial biofilms. It has been proven that silicone polyethers can be used as effective protective coating to prevent biological corrosion of glass.

2. MATERIALS AND METHODS

2.1 Materials and physicochemical characterization

All commercially available chemicals were used as received without any further purification. 1,1,1,3,5,5,5-heptamethyltrisiloxane (HMTS) was purchased from Sigma-Aldrich. Allyl polyether (BIKANOL7) was purchased from ICSO Chemical Production, Kędzierzyn-Koźle. The hydrosilylation catalyst was a commercially available Karstedt catalyst purchased from Sigma-Aldrich. Magnetic Nuclear Resonance spectra: ^{1}H NMR, ^{13}C NMR and ^{29}Si NMR were taken on a Bruker Ascend 400, at room temperature, with $CDCl_3$ as a solvent. FT-IR spectra were taken on a Nicolet iS20 Mid-Infrared FT-IR Spectrometer with a Gate diamond ATR attachment. The spectra were collected in the range 500-4000 cm^{-1}, with the resolution of 2 cm^{-1}, recording always 32 scans of the background and the sample.

2.2 Synthesis of 3-[3-(hydroxy)(polyethoxyy) propyl]-1,1,1,3,5,5,5-heptamethyltrisiloxane

Siloxane containing polyether group was synthesized in the hydrosilylation reaction of 1,1,1,3,5,5,5-heptamethyltrisiloxane (HMTS) and allyl polyether containing 7 ethoxy groups with the terminal hydroxy group (BIKOANOL7). The process was carried out in the presence of Karstedt catalyst and using toluene as a solvent. The reaction substrates are not sensitive to moisture, so the hydrosilylation reaction could be performed in an open system, which significantly facilitated the synthesis. The pure product was obtained in high yields of 99% (19.71g product). The products were subjected to spectroscopic analysis to verify obtainment of the assumed structure.

2.3 Modification on glass plates

All microscope plates were cleaned by sonication in a detergent (15 min), distilled water (15 min) and acetone (15 min) and dried at 50°C for 30 min. The substrates were dipped in 3% solutions of HOL7 in ethanol solvent for 20 min and dried at 120°C for 60 min. Next, the samples were subjected to the egg white test and microalgal tests.

2.4 Egg white test

A specified amount of egg white was applied to modified glass plates and a control sample. The plates were exposed to air for 24 hours. After the protein dried, the appearance of the plate was evaluated (Shanmugavel et al., 2021; Barletta et al., 2018).

2.5 Microalgal experiment

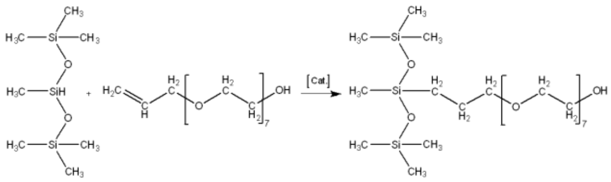
The effect of HOL7 compound in concentrations – 3% dissolved in alcohol (A) and water (W) applied on glass substrates by immersion (I) and rubbing (R) was tested.

A mixture of four strains of algal taxa isolated from building materials in the quantity was used for the experiment – Chlorodium saccharophilum PNK010, Klebsormidium flaccidum PNK013, Pseudostichococcus monallantoides PNK037 and Trebouxia aggregata PNK080. The choice of these algae followed from their wide occurrence in terrestrial environments. The glass surfaces were inoculated with 500 µl of the algal mixture by pipetting and cultivated for 14 days in laboratory conditions optimal for algal growth. The effect of HOL7 coatings on the glass substrate was evaluated based on the changes in algal biological activity which can be directly expressed by the photosynthetic activity of cells via chlorophyll fluorescence measurements. The analysis was based on the pigment fluorescence intensity (chlFI) measurements after 7 and 14 days of incubation relative to control samples – glass substrates without organosilicon compound coatings. The chlFI measurements were made using the Leica TCS SP8 confocal microscope (Lecia Microsystems, Wetzlar, Germany).

3. RESULTS

3.1 Results of synthesis

The synthesis of siloxane polyether was performed using allyl polyether containing 7 ethoxy groups with the terminal hydroxy group. In this way, a compound with amphiphilic properties was obtained. In addition to hydrophobic methyl groups, hydrophilic polyether groups were attached to silicon. All reactions were carried out in toluene as a solvent. The catalyst was the commercially available Karstedt one. The hydrosilylation reaction is shown in Scheme 1.



Scheme 1. Hydrosilylation of allyl ether with 1,1,1,3,5,5,5-heptamethyltrisiloxane

The isolated product was subjected to spectroscopic analysis (^{1}H NMR, ^{13}C NMR, ^{29}Si NMR) whose results are presented below:

^{1}H NMR ($CDCl_3$, TMS) δ (ppm): -0,13 (-Si(CH_3); -0.06 (-Si(CH_3)$_3$); 0.32 (-SiCH_2-); 1.46 (-$CH_2CH_2CH_2$-); 3.27 (-CH_2O-); 3.45-3,51 (-OCH_2CH_2-); 3.55 (-OH).
^{13}C NMR ($CDCl_3$, TMS) δ (ppm): -0.75 (-SiCH_3); 1.48 (-Si(CH_3)$_3$); 13.10 (-SiCH_2-); 22.79 (-$CH_2CH_2CH_2$-); 61.17-69,61 (-CH_2O-); 70.18 (-OCH_2CH_2-).
^{29}Si NMR ($CDCl_3$, TMS) δ (ppm): -21.74 (-Si(CH_3)CH_2); 7.00 (-Si(CH_3)$_3$).

3.2 Results of egg white test

Egg white, due to its high content of albumin and amphiphilic proteins in its structure, shows great morphological similarity to marine organisms. Studies on the protein can therefore be used as a preliminary test for further biological studies on the properties of antifouling coatings. A protective coating applied to the surface of a glass pane causes dry egg white to be repelled from its surface. When the glass is tilted, the protein slides off the surface (Figure 2, left). On an unmodified surface, the protein adheres completely to the surface after drying (Figure 2, right side).

Figure 2. Egg white after drying modified with HOL7 (left side) and unmodified plate (right side)

3.3 The effect of 3% HOL7 on algal biofilm

The photosynthetic activity of control biofilms growing on a glass substrate did not vary significantly with time ($p = 0.273$), but all tested biofilm samples coated with HOL7 showed statistically significant changes in chlFI compared to the control. However, some biofilm samples showed a high level of fluorescence after 14 days of cultivation, a high level of chlFI - over 85% of the emission intensity of the control sample. In these samples, HOL7 was applied at a concentration of 3% in a water or alcohol solution by rubbing the glass substrate. The average values of maximum and median chlFI for the samples coated with HOL7 were lowest for a 3% HOL7 solution - (avmax/ avmed gv) 3%_7: 155.0/34.6; 3%_14: 165.1/40.9. After 7 days of cultivation, the highest change in chlFI was detected in the biofilm growing on the glass coated by immersion in 3% HOL7 water solution (max. 77.3 gv; Q3 29.8 gv); however, with time, the photosynthetic activity of the algal cells increased (max. 126.1 gv; Q3 67.8 gv). For the same HOL7 concentration and application method, but in a different solvent, the best results were obtained for all samples at the end of the experiment (7 days: max. 171.2 gv; Q3 59.1; 14 days: max. 90.5; Q3: 38.9 gv). However, the best result in inhibiting the photosynthetic activity of algal biofilm was obtained using a 3% HOL7 alcohol solution applied to glass by immersion Figure 3.

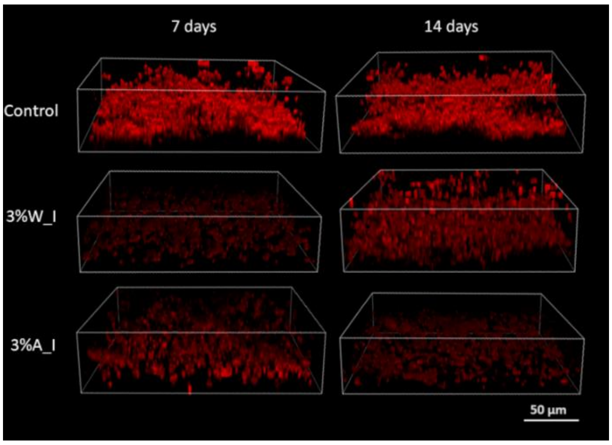


Figure 3. 3-DCLSM visualization of algal cells in biofilms growing on the uncoated glass substrate (control) and the substrates coated with 3% HOL7 solution in water (W) or alcohol (A) by immersion (I) after 7 and 14 days of cultivation

4. DISCUSION

Various hydrophilic compounds have been investigated for their efficacy against biological fouling. Mostly, the targeted bio-factors were pathogenic bacteria and fungi. Application of coatings made with the use of such compounds brings the protective effect via the reduction of adhesion abilities, especially in an early stage of biofilm formation. In the experiment described, we used a mixed cul-ture of well-formed biofilms that already had EPS matrix and contained cells in a much higher number than the ones spread by the wind in ex situ environment (Nowicka- Krawczyk et al., 2022). Such inoculation is more resistant to the stress factors and and if any positive effect occurs in response to the exposure to HOL7, it will most probably be much more intense in the case of aeroterrestrial colonization.

Application of a bifunctional organosilicon compound on the glass reduced, more or less effectively, the photosynthetic activity of algal cells. Since the structure of the compound was not based on biocide active agents, its effectiveness was associated with reduction of water content in the cells. The changes in chlFI prove the effect.

The best result in suppressing the photosynthetic activity of algal biofilm was achieved using a 3% HOL7 alcohol solution applied to glass by immersion. This moderate concentration of solution and method of application may not increase the desiccation rate as observed upon exposure to a higher percentage of HOL7, but it may highly affect cells by the loss of the repair capacity leading to the degradation of the biofilm.

5. CONCLUSIONS

The above presented analyses and interpretations permit drawing the following conclusions. An effective method was developed for the preparation of functional-ized polysiloxanes containing functional groups. The method is based on the hy-drosilylation of olefins catalyzed by a Karstedt catalyst. The isolated product was subjected to spectroscopic analysis (^{1}H NMR, ^{13}C NMR, ^{29}Si NMR). The product structure was confirmed. Next, the surfaces of glass plates were modified with obtain organosilicon derivative that contained the hydrophilic groups ena-bling the formation of bonds to the substrate (polyether group). It has been prove that a simple test with egg white allows a preliminary verification of the effectiveness of the coating produced before biological corrosion occurs.The modification was performed using two methods: (a) by immersion (I), (b) and by polished with a cotton cloth (R). The substrates were dipped in 3% solution of HOL7 in ethanol (A) or water (W) solvent. In the next step the microalgal activity investi-gation was conducted. The best result in suppressing the photosynthetic activity of algal biofilm was achieved using a 3% HOL7 alcohol solution applied to glass by immersion. This moderate concentration of solution and method of application may highly affect cells by the loss of the repair capacity leading to the degradation of the biofilm. In summary, the syn-thesis of polysiloxane functionalized with a polyether group in the hydrosilylation reaction is relatively simple. Moreover, microbiological tests showed the effective-ness of the HOL7 coating in the against biological colonization. The obtained re-search results seem to be promising from the point of view of practical application.

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