THERMAL AND PHYSICOCHEMICAL PROPERTIES OF SILICONE-BASED COMPOSITES REINFORCED WITH SILANIZED MAGNETIC POWDER

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Abstract

Silicone-based materials are of great interest in medicine and cosmetic applications because of their biocompatibility and elasticity. Recently, there has been a significant focus on the development of functional materials that integrate multiple desirable characteristics. Elastic composites reinforced with magnetic filler are active in a magnetic field. These materials can be an interesting alternative to the currently used materials, after appropriate modification of the NdFeB powder. From the point of view of the use of materials in biomedical engineering, they require a lot of research and analysis to determine whether they are useful and will not cause potentially negative effects on a living organism. The aim of the work was to verify the influence of the powder silanization method on the thermal and physicochemical properties of silicone-based composites reinforced with NdFeB powder. The appropriate selection of the silanization parameters used in the process allows control of the properties of the composite. The powder surface silanization execution affects the physicochemical and thermal stability of the prepared composites. It has been established that, depending on the method of silanization, the composite properties were changed. The obtained experimental results may lead to further research on the functionalization of elastic composites reinforced with magnetic powder.

***Keywords**: silanization, elastic magnetic composite, thermal properties, physicochemical properties*

Introduction

Silicone materials are commonly used in a variety of medical and cosmetic applications. Nowadays, they are applied mainly outside the body, as materials for contact lenses or artificial skin. However, they can also be used in other medical applications, such as drug delivery systems, cochlear implants, and cardiology. Their biocompatibility with the human body was confirmed in many experimental tests [1,2].

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High elasticity and ability of silicone-based magnetic composites to move and act in a magnetic field make them of interest in many branches of medicine and biomedical engineering. In the group of elastic polymers, one of the most commonly used is polydimethylsiloxane (PDMS). This polymer is most often combined with highly magnetic particles, e.g. NdFeB [3]. However, the use of NdFeB-based composites in medicine is not widespread, especially due to the risk of adverse reactions in contact with the human body [4-6]. In our previous work [7] the chemical stability of PDMS-based composites was assessed. It was stated that the incubation affects the samples and liquids, changing their physicochemical properties. It is because the organic matrix is lipophilic and the NdFeB powder is hydrophilic, so the compatibility between these two phases is not enough. It can be improved, for example, through modification of the powder surface using the silanization process. The surface modification of the magnetic powder using a biofunctional silane coupling agent enhances the adhesion between the NdFeB particles and the polymer matrix. It was also stated [8] that the addition of a silane coupling agent has almost no effect on the magnetic properties of bonded magnets based on silanized NdFeB magnetic powder and organic binder.

During the silanization process, the magnetic powder surface is coated with alkoxysilane molecules of organofunctional character, as it was described elsewhere [9,10]. This method is commonly used in materials science applications, especially during composites and smart materials preparation. Silanization is successfully implemented, for example, in carbon nanotubes to change the behavior of their surface [11]. This method is also used for the modification of silica wafers to activate their surfaces [12].

Silanization is carried out using chemical compounds called silanes. An example of such a chemical is APTES, which is 3-aminopropyltriethoxysilane with an amino group attached to the silicone chain. APTES is used to modify iron oxide nanoparticles, nanosilica particles, or carbon nanotubes [11,13,14].

The aim of the work was the evaluation of the influence of magnetic powder silanization parameters on the properties of silicone composites, especially their interaction with incubation fluids. This is particularly relevant in biomedical applications.

Materials and Methods

In this study, elastic silicone-based composites reinforced with magnetic powder subjected to the silanization process were examined. The organic matrix used for composite synthesis was Ecoflex 00-30 (Smooth-On, USA). The material belongs to the Ecoflex polymers group, which includes commercially available silicones with varying Shore-A hardness [15]. The silicone used in this study is characterized by a hardness of 30 Shore A. This polymer is prepared from two components, supplied as A and B liquid parts. To obtain the correct mixture, the ingredients must be mixed in a 1A:1B ratio.

Magnetic NdFeB powder (MQFP-14-12, Magnequench, Singapore) with a particle size of d50 = 25 μm (at least 50% of the powder particles have a particle size of 25 μm or less) was the object of the silanization. The chemical composition of the NdFeB powder is presented in TABLE 1.

TABLE 1. Composition of MQFP-14-12 micro-powder.

Element	Fe	Nd	B	Nb
Concentration [%w/w]	71.7	26.0	1.0	1.9

Firstly, micropowder was subjected to the silanization process. The silane used in the experiment was (3-Aminopropyl) triethoxysilane (APTES, Sigma Aldrich, USA). Twelve different combinations of silanization parameters (TABLE 2) were used to modify the surface of the magnetic powder: a) various silane concentrations, b) silanization time, and c) the type of solution used to dilute the silane. Each silanized micropowder was mixed with liquid silicone in a mass ratio of 7:3. Finally, twelve composites (symbols B-M, TABLE 2) based on silanized micropowders were obtained. The sample marked with the symbol C was prepared in two steps. At first, the powder was phosphorated using 10% orthophosphoric acid to activate the surface, and in the second step, silanization was carried out in the ethanol/water mixture. Composites with non-silanized metal powder (symbol A) and pure PDMS (symbol 0) were used as references.

TABLE 2. Silanization parameters used in experiments.

Designation	Silanization parameters		
	Silane concentration [% w/v]	Time, t [h]	Solution type
0	-	-	-
A	-	-	-
B	4.75	>24	ethanol 95%v/v/ water 5%v/v
C	4.75	>24	ethanol 95%v/v/ water 5%v/v (preceded by soaking the powder in 10% orthophosphoric acid)
D	2	4	water
E	2.5	1	toluene
F	2.5	2	
G	2.5	4	
H	5	1	
I	5	2	
J	5	4	
K	7.5	1	
L	7.5	2	
M	7.5	4	

The prepared samples were left for the curing process, and then the samples were cut into smaller pieces, weighed, and divided into two groups: incubated and non-incubated. The samples from the first group were immersed in a 0.9% w/v sodium chloride aqueous solution (NaCl, Sigma Aldrich, USA) prepared with ultrapure Milli-Q water (Merck Millipore, Germany). The second set of samples was stored in a dry place at room temperature.

According to the ISO 10933-13 standard [16], the mass ratio of the composite to the incubation fluid was 1g:10ml. Conditioning studies were carried out in an incubator with an internal temperature range of 37 ± 0.5°C. The materials were incubated for 28 days. After this time, the samples were taken out and dried at room temperature (21 ± 1°C) and 60% humidity for 24 hours.

Thermal properties

The thermal properties of the obtained composites were evaluated using a Q500 thermogravimetric analyzer (TA Instruments, USA). Thermogravimetric tests (TGA) were performed according to the ISO 11358-1:2022 standard [17]. Each measurement was carried out in a nitrogen atmosphere, with a temperature range of 25 to 950°C and a heating rate of 10°C/min. Samples with an average weight of 5 mg were used for testing. Measurements were performed three times for each sample.

Physicochemical properties

The physicochemical properties of the obtained composites were also determined in this study. A water contact angle was measured and analyzed using a contact angle goniometer (Ossila, UK). The contact angle was defined between the surface and the water droplet [18]. The test included applying a 5 µl deionized water droplet on the surface and the drop image on the surface was recorded for 5 s. The obtained images were analyzed with the included software, where a tangent method was used to calculate the water contact angle [19]. Measurements were performed five times for each sample.

The density of the composite was determined using the hydrostatic method. A balance with special equipment and software (Mettler Toledo, USA) enables the calculation of density from the mass of the sample in two density-known environments. The mass of the sample in air and then in water is registered by the device. The density calculation using Archimedes' principle is conducted automatically by the balance. Measurements were made five times for each sample.

Fluid absorption was measured using an analytical balance (Mettler Toledo, USA) with readability up to 0.01 mg. The mass of each sample was recorded before incubation and 24 hours after removal from the containers and drying at room temperature (temperature of 21 ± 1°C and humidity of 60%). The percent change in mass was calculated from equation (1):

$$W(\%) = \frac{w_w - w_d}{w_d} \cdot 100\% \quad (1)$$

where:
w_w - mass of the sample after incubation [g];
w_d - mass of the dry sample before incubation [g].

The measurements were performed five times for each sample.

Surface roughness

The Confocal Laser Scanning Microscopy (CLSM) technique was used for microscopic observations and roughness measurements of the surface. The microscope used in this study was LEXT OLS 4000 (Olympus, Japan). Two linear roughness parameters: Ra - the arithmetic mean value of deviation from the mean surface profile line and Rz - the highest roughness height according to the 10 highest measured profiles, were evaluated [20]. From the 3D images, it was possible to measure three surface roughness parameters: Sa - the arithmetic mean height value of the absolute deviations of the surface, Sq - the mean squared deviation of the surface from the reference surface (standard deviation of the height of surface irregularities), Sp - height parameter of the highest peak of the surface [21]. The parameters were determined using microscope software. It enables an automatic calculation of the roughness on a specific line or surface. Linear measurements were made horizontally. Area measurements were made over an area of 300x300 pixels. Measurements were performed five times for each sample.

Statistical analysis

The results of the measurements are presented as mean values with standard deviations. The r-Pearson correlation test was performed to measure the linear correlation between tested parameters.

Results and Discussion

In this work, the influence of magnetic powder silanization on the thermal and physicochemical properties of silicone-based composites for biomedical applications was determined. The results of thermogravimetric analysis (FIG. 1) allow for the determination of the decomposition temperature at 1 wt% and 5 wt% for all samples before and after incubation.

For most tested samples, 1 wt% weight loss (FIG. 1a) was observed in a temperature range of 200-250°C, both for incubated and non-incubated ones. The only exception was for samples after the silanization and phosphorylation process, marked as C, where the temperature of thermal degradation was ~112°C before and ~105°C after incubation. It was even 100°C lower than for most samples. It may be concluded that the decomposition temperature of the material is lower after silanization combined with phosphorylation. The highest temperatures, although almost at the same level for non-incubated and incubated samples, were observed for the composite with non-silanized micropowder (accordingly ~255°C and ~254°C). However, for the rest of the tested samples, incubation changed the thermal stability of the composites, as the temperatures for 1 wt% weight loss for samples after incubation were lower (from 6°C to 45°C) compared to the non-incubated samples.

In the temperature range of 300-350°C, a 5 wt% weight loss (FIG. 1b) was observed for most of the examined samples, both before and after the incubation process. A similar observation, as for 1 wt% weight loss was for sample C, based on powder after silanization and phosphorylation, where the temperature reached the lowest value (~263°C before and ~252°C after incubation). Generally, the temperature of thermal decomposition after incubation was lower by about 2-28°C compared to the samples before incubation.

In the FIG. 2a the results of density before and after 28 days incubation are presented. The lowest density is obtained for pure silicone (1.04 g/cm^3 before and 1.03 g/cm^3 after incubation). The highest density was measured for the composite (A) with non-silanized NdFeB micropowder (1.70 g/cm^3 before and 1.79 g/cm^3 after incubation) and for sample (I) based on micropowder silanized for 2 h with 5% w/v APTES solution (1.81 g/cm^3 before incubation). The samples prepared with the micropowders silanized in toluene have a density in the range of 1.47-1.62 g/cm^3. The methods using ethanol and water as APTES solvents (B-D) resulted in the lowest density values. In general, it was observed that after incubation of the samples, the density of the tested composites decreased. This may be due to the dissolution of the composite components in sodium chloride solution, which was also observed in the literature [22].

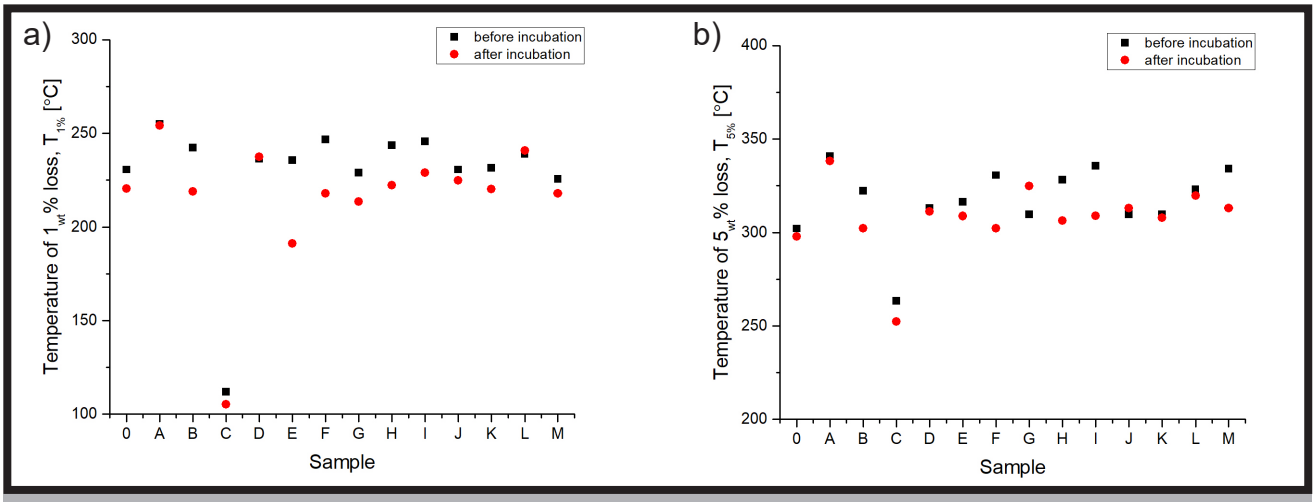

FIG. 1. Results of TGA analysis, (a) 1 wt% and (b) 5 wt% weight loss for samples before and after materials incubation.

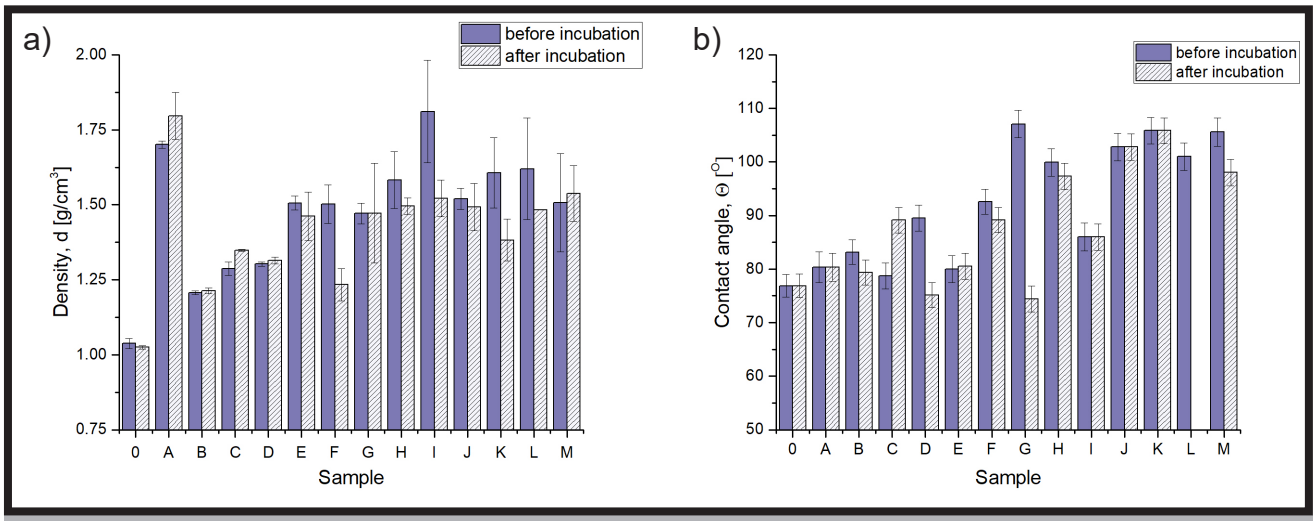

FIG. 2. (a) Density and (b) the water contact angle of the examined composites before and after incubation.

The water contact angle values on the composites before and after incubation are presented in FIG. 2b. The pure silicone (0) tends to be hydrophilic, showing a contact angle value below 90° (Θ = 77° before and Θ = 72° after the incubation process). The addition of micropowder increases the value of the contact angle. For samples, where the powder was silanized with a 5 % w/v (H, J) or 7.5 % w/v (K-M) APTES solution, the water contact angle was higher than 100°. After incubation, this parameter was more stable for samples prepared using ethanol and water as solvents for APTES, where the change in the water contact angle was in the range of 3-12°. A much greater decrease was observed for samples with powder silanized in toluene (E-M). The difference in the contact angle of the sample before and after incubation was, for example, ~32° for sample G and ~46° for sample K. It can be seen that in several cases the material retained its hydrophobic character after incubation. It is reported in the literature that the presence of silane in the composite increases the wettability of a given surface [23,24].

The results of the water absorption of incubated samples are presented in FIG. 3. The lowest percentage of mass change is measured for pure silicone (-0.003%). It was noticed that the longer the silanization time of the micropowders, the higher the water absorption. For sample (E), after 1 h of silanization with 2.5 % w/v of APTES (E), a mass increase of 0.033% was observed. For sample (G) silanized with the same amount of APTES, but for 4 h, a mass increase of 0.050% was observed. A relatively high absorption value was observed for samples with micropowder silanized in ethanol and phosphorated (~0.081%). All examined composites showed a very low level of water absorption after 28 days of the incubation process.

In FIG. 4 results of Ra and Rz linear roughness of composite surface before and after silanization are presented. The highest Ra value was obtained for sample (A) with non-silanized powder (0.25 μm before and 0.17 μm after incubation) and the composite silanized and phosphorated (C) in one process (0.19 μm before incubation). For the rest of the samples, the Ra value did not exceed 0.1 μm. However, it was observed that the silanization method influenced the surface roughness of the incubated samples. Composites prepared with powder silanized in ethanol/water solutions (samples B-D) showed lower Ra value after incubation. For the samples with micropowder silanized in toluene (samples E-M) Ra roughness was lower after incubation.

Similar observations were noted for Rz roughness. The highest Rz value was observed for sample (A) with non-silanized powder (1.58 μm before and 0.85 μm after incubation) and sample (C) silanized and phosphorated in one process (1.25 μm before incubation). For most samples Rz roughness did not exceed 0.3 μm. Similarly to the Ra roughness, for the same composites, incubation causes an increase in the Rz roughness of the materials, which is also observed in other studies in the literature [25,26]. However, for the composite with non-silanized reinforcement (A) and composites with a silanized filler using ethanol or water as solvents (samples B-D), a decrease in surface roughness Rz was observed after incubation [27].

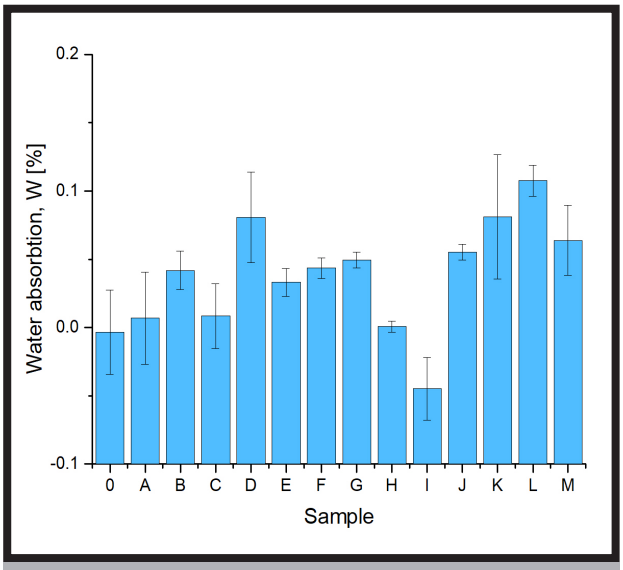

FIG. 3. Water absorption of examined composites.

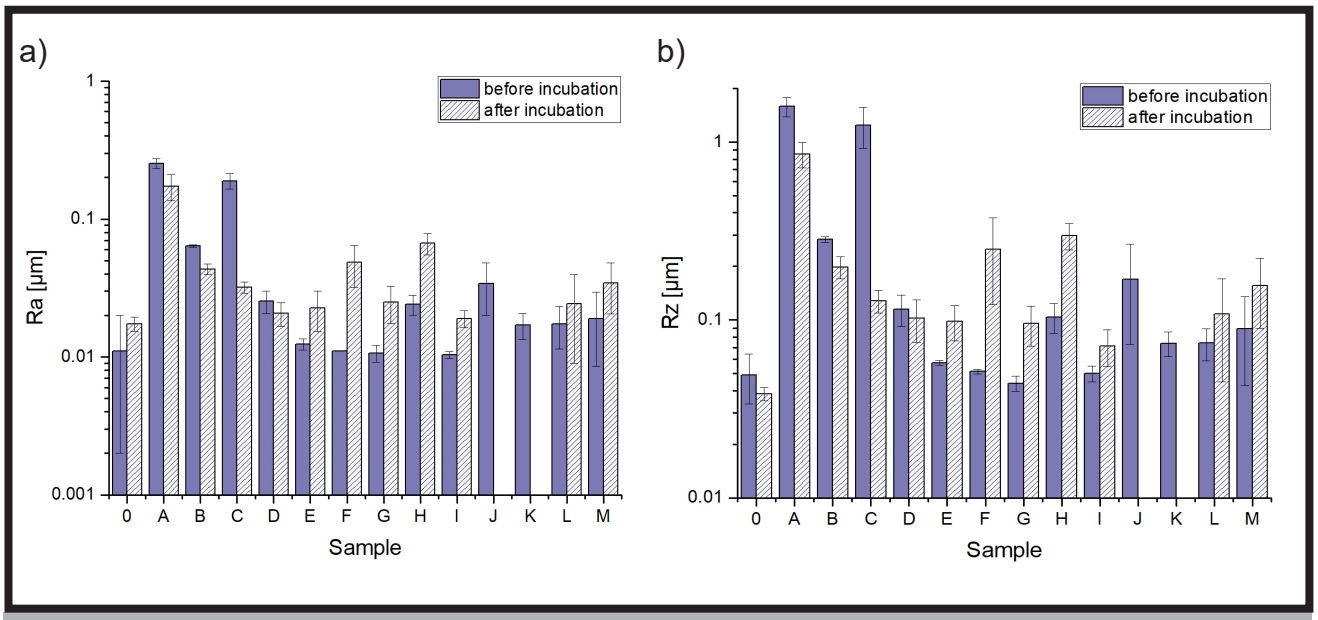

FIG. 4. The linear roughness values of the examined composites before and after incubation: a) Ra – arithmetic mean roughness, b) Rz – maximum height.

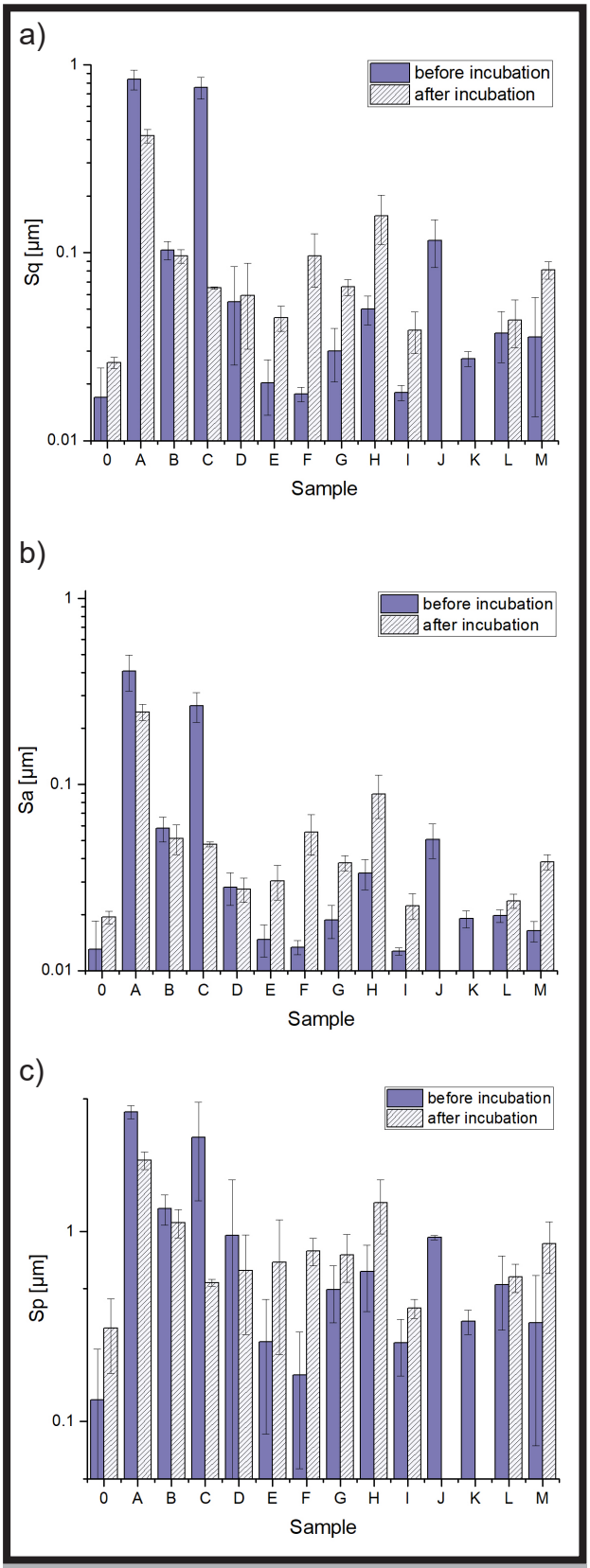

FIG. 5. Surface roughness parameters of the examined composites before and after incubation: a) Sq – root mean square height, b) Sa – arithmetical mean height, c) Sp – maximum peak height.

In FIG. 5 results for Sq, Sa, and Sp surface roughness parameters are presented. Generally, the Sq value was lower than 0.2 µm for the measured samples and tends to increase after the incubation process. The surface roughness was the highest for the non-silanized sample (A) (0.85 µm before and 0.42 µm after silanization) and the sample (C) after combined silanization and phosphorylation process (0.76 µm before incubation).

The Sa surface roughness increased for incubated composites in most of the cases. The difference between non-incubated and incubated samples was in the range of 0.01-0.02 µm. The exceptions were observed for the non-silanized sample (0.41 µm before and 0.25 µm after silanization for sample A) and the sample after the combined silanization and phosphorylation process (0.26 µm before incubation for sample C), where the roughness decreased.

Similarly, as in previous cases, the Sp roughness was the highest for the sample (A) with non-silanized powder (4.26 µm before and 2.37 µm after silanization) and the sample (C) after the combined silanization and phosphorylation process (3.13 µm before incubation). The Sp roughness for pure silicone (0) is 0.13 µm before silanization and increases to 0.31 µm after incubation.

The roughness of the examined surfaces was found to be lower for composites based on silanized powder. However, it should be noted that the roughness increased after the incubation process. In summary, it can be stated that the use of the silanization process reduces surface roughness [28].

The CLSM observations were also used to calculate the surface and volume of structures protruding above the average profile line of the material on the samples' surface (FIG. 6). They are called peaks and pits because they are higher or lower than other points in their respective neighbourhoods. Both the area and the volume occupied by the structures protruding on the surface of the sample allow us to assess how far all peaks and pits protrude from the base surface. The total surface of the peaks and pits is significantly higher for sample (A) with non-silanized microparticles (32107 μm^2) than for samples with modified powder (B-M). In most cases, the peaks and pits surface were more than six times lower for silanized powder-based composites. For all composites, the surface of peaks and pits decreased after incubation.

The volume of structures protruding above the average profile line of the material surface is of the highest value for 0 sample (pure silicone) and composite A with non-silanized powder (before incubation), for which the values obtained were 16129 μm^3 and 18027 μm^3, respectively. The most significant difference was measured for samples F and G (7653 μm^3 and 2342 μm^3, respectively). This may be due to the deepening of already existing peaks and pits.

There is a strong correlation between the surface area and the volume of the peaks and pits. Due to the r-Pearson correlation of 0.871 for $p < 0.001$, the measured values are interconnected. However, for a better description of the surface, these two parameters should be analyzed to fully characterize the tested surface.

In FIGs. 7 and 8 representative CLSM images of two samples, with non-silanized micropowder (A) and with silanized micropowder (composite M), before and after incubation were presented. Some particle agglomerations in both materials were observed. However, it can be noticed that fewer agglomerates are present in sample M, based on the silanized magnetic powder, as compared to the composite with non-silanized powder.

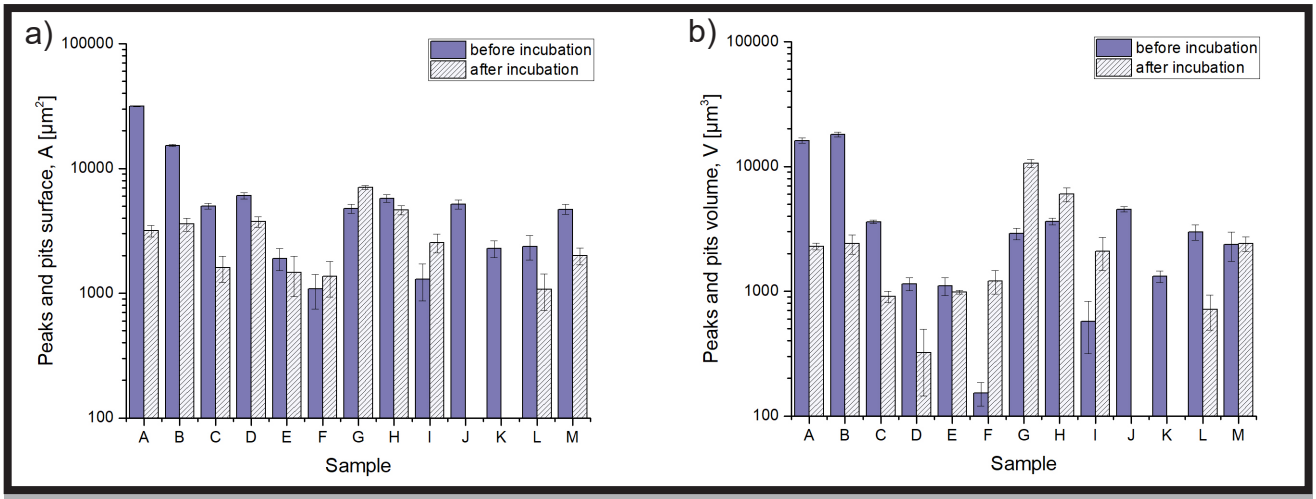

FIG. 6. a) Surface and b) volume of structures protruding above the average profile line of the surface in the examined composites before and after incubation.

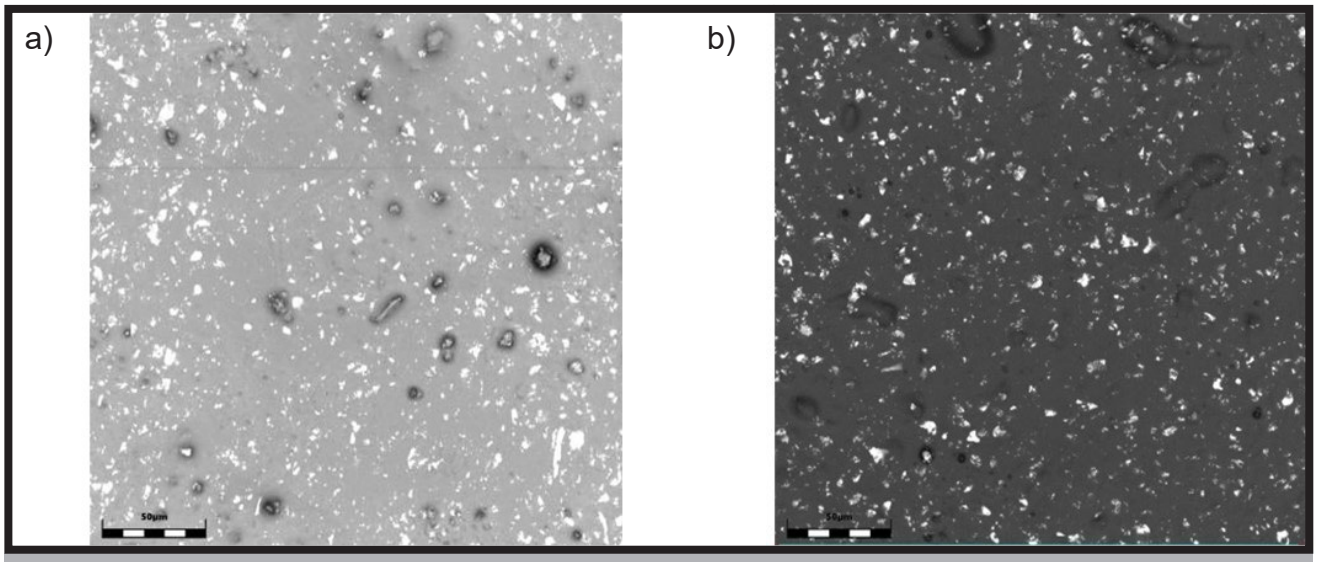

FIG. 7. The surface of composite with non-silanized micropowder a) before and b) after incubation. Scale bar 50 µm.

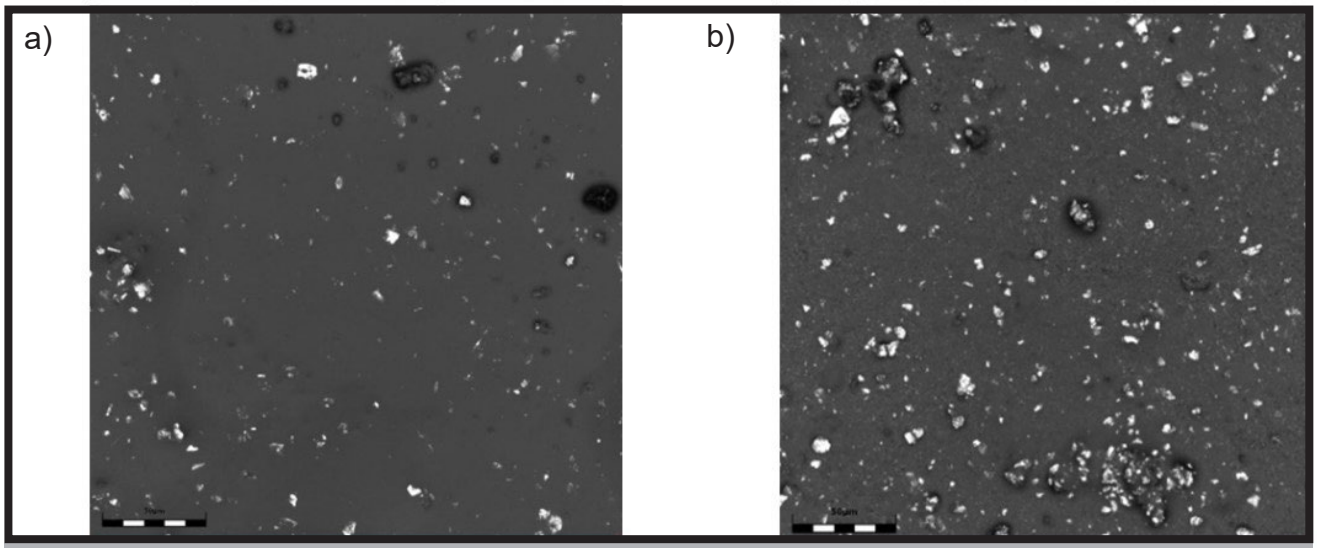

FIG. 8. The surface of composite with micropowder silanized with 7.5% APTES solution for 4 h, a) before and b) after incubation. Scale bar - 50 µm.

Conclusions

To enhance the bond between the magnetic powder and the organic matrix, the powder was silanized under different conditions. The appropriate selection of the silanization parameters allows for control of the properties of the composite. This is extremely important from the point of view of specific composite applications in biomedical engineering. Thus, in this study, variable parameters such as silane concentration, silanization time, and type of solution were examined. The obtained results show that the powder surface silanization affects the physicochemical and thermal stability of the prepared composites. Thermogravimetric analysis shows that the silanization process reduces the temperature of material's decomposition. It was also observed that the values of tested physicochemical properties were slightly higher for silanized powder-based composites. Surface roughness analysis results show that the composites with silanized powder (B-M, except for C) had significantly lower values of Ra, Rz, as well as Sq, Sa, and Sp parameters. Additionally, the silanization combined with phosphorylation presented in the literature as a promising method of modification, caused a worsening of tested composites properties, which are relevant for biomedical applications of the material. The obtained experimental results may lead to further research on the functionalization of composites based on magnetic powder.

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