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# The properties of experimental silicones reinforced with silica fillers for dentistry

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## ABSTRACT

**Purpose:** Silicone materials crosslinked during the hydrosilylation reaction are common in dental prosthetics and dentistry. These materials are characterized by a relatively very good properties. However, one of the fundamental problems associated with the use of such polymers is a low resistance to microbial colonization. Numerus fillers with antimicrobial potential have been used experimentally for a decade. Anyway point of concern in this type of work is the use of commercially available materials as the matrix. However, the composition of the matrix for this materials is not clearly known. Therefore, the aim of this study was to obtain a two-component silicone material, crosslinking at room temperature, with properties corresponding to the needs of dental prosthetics, in particular as denture soft linings. Such material would be a starting point to obtain more sophisticated compositions, however, of known composition in full.

**Design/methodology/approach:** As the matrix the mixtures of polymers were prepared. As the "catalyst" component a vinyl polydimethylsiloxane with platinum catalyst in xylene was prepared. As the "base" component a mixture of vinyl polydimethylsiloxane with methylhydrosiloxane - dimethylsiloxane copolymer was made. One of the two fillers differing in the functionalizing was added into prepared substrates (10 or 15% by weight) and ultrasonically homogenized. The Shore A hardness, tensile strength, bond strength to denture base material, water sorption and solubility were investigated. Measurements were made after different aging periods in distilled water at  $37\pm1^{\circ}$ C.

**Findings:** The study showed an increase in mechanical properties with increasing concentrations of fillers. The type of filler significantly affects the test results. Increasing the aging time effects on increasing the hardness of materials. Water absorption and solubility were within the desired range.

**Research limitations/implications:** Research limitations/implications The results of the presented research may be generally dependent on technology of fillers introduction into polymers matrix.

**Practical implications:** The results show promising properties of the tested materials. It can be used as starting material for further studies on soft or super soft denture lining and other materials characterized by similar requirements.

**Originality/value:** The method of receiving silicone - based composites with properties required for the selected prosthetic materials, especially denture soft linings was presented. Their properties were similar to commercially available products.

**Keywords:** Addition cure silicone; Soft lining; Dental materials; Filler; Hardness; Tensile strength; Sorption; Solubility

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PROPERTIES

# 1. Introduction

The addition of cure silicones is widely used in medicine, especially as materials for dentistry and prosthetic dentistry. They are well known as very good impression materials or transfer and impression moulding elastomers. The other destinations of those materials are long-term soft denture lining materials, relining in implantology, postoperative obturators and maxillofacial prosthesis [1-6]. During polymerization reaction the addition of silvl hydride groups to vinyl groups of vinyl polydimethylsiloxane is linked with the assistance of a platinum catalyst [5,7]. This reaction takes place without formation of by-products and allows to obtain a polymer network. The absence of by-product has an impact on stable in time properties of materials, including their dimensional stability. This is important for impression materials and transfer/moulding elastomers. However, the mechanical properties of unfilled silicone polymers are poor and unsuitable for practical applications. In order to improve these properties different types of functionalized fumed silica fillers are widely used. The applications of fillers significantly increase the strength and hardness of materials and additionally can also modulate its viscosity. Although the reaction of silvl hydride groups (-SiH) with vinyl groups (CH2=CH-) occurs at a 1:1 stoichiometry, when functionalized silica fillers are used, the ratio of vinyl to hydride is considerably higher. The optimal proportion may be determined, for example, by determining the hardness of samples achieved with diverse ratios [5]. Different applications of this materials requires the universal and specific properties. For impression and transfer/impression moulding it is required excellent detail reproduction, dimensional stability, high tear strength, consistency determined by viscosity after mixing, working and setting time, percentage of elastic recovery from deformation, strain

in compression, flow and hardness. For silicone permanent soft linings and other silicone elastomers used for prosthetic dentistry the most often considered properties are hardness, tensile and tear strength, bond strength to other materials (PMMA), sorption, solubility, colour and viscoelastic properties. However the properties must be stable in time in order to ensure provide comfortable use of these materials for longer periods. While the properties of impression materials seem to be rewarding, some difficulties are noted for different materials intended for long-term use. The most often reported problems, occurring usually after a few months of use, are low antimicrobial resistance and debonding from PMMA materials [9-11]. The bond strength to PMMA polymers is mainly dependent on bonding agents which interacts with the surface of the acrylic materials and with the silicones [8]. Antimicrobial resistance can be enhanced by using different additives, like antimicrobial fillers. Fillers influence the properties of modified materials. Effect of fillers is hard to identified in details when the commercially available prosthetic silicones are used, because of its exact composition is not known. For that reason, the aim of this study was to obtain a two-component silicone material, crosslinking at room temperature, with properties corresponding to the requirements for long-term soft denture lining materials. The material can be the base to obtain a more sophisticated compositions.

#### 2. Materials and methodology

#### 2.1. Materials

The materials used in this study and their properties were presented in Table 1. The "base" and "catalyst" components were prepared separately in glass flasks in small portion.

 Table 1.

 The list of materials used in the investigations based on manufacturers data

Code	Material	Manufacturer	Chosen properties		
	Matrix components		Viscosity, cSt	Molecular	r weight
PDMS	Vinyl terminated polydimethylsiloxane	Gelest, USA	1000	25000-30	000
PDMHS	Methylhydrosiloxane - dimethylsiloxane copolymer, trimethylsiloxy terminated	Gelest, USA	25-35	1900-200	0
CAT	Platinum - divinyltetramethyldisiloxane complex in xylene, 2.1-2.4% platinum concentration	Gelest, USA	-	-	
	Fillers		surface	Average particle size, nm	Methyl groups, 1/nm <sup>2</sup>
F1	Aerosil R974, hydrophobic fumed silica aftertreated dimethyldichlorosilane	Evonik, Germany	170±20	12	2.8
F2	Aerosil R812, silanamine, 1,1,1-trimethyl-N- (trimethylsilyl)-, hydrolysis products with silica	Evonik, Germany	260±30	7	3.5

First, 30 g of matrices of the individual components were made. The mixture of 95 % PDMS with 5 % PDHMS was prepared as the matrix for "base" component. The mixture of PDMS with 0.2 % of CAT was done as the matrix "catalyst" component. Mixtures were stirring with a magnetic stirrer at room temperature for 12 h. Next, the fillers hydrophobic fumed silica aftert reated dimethyldichlorosilane (F1) or silanamine, 1,1,1-trimethyl-N-(trimethylsilyl)-, hydrolysis products with silica (F2) were introduced into the previously prepared matrixes. It was added and initially mixed in a small portions with spatula. After introducing of the filler the compositions were ultrasonically homogenised. Particularly flasks were mounted in holder and placed in larger flask with water to facilitate cooling of the material. Homogenisation was proceeded in five cycles, each lasting circa 2 minutes, and each cycle was stopped when the temperature of the material reached 45°C. During intervals between the cycles flasks with materials were moved into the glass container with the water and cooled. With following procedure into the "base" and "catalyst" components particular fillers were introduced with the concentrations of 10 and 15 %.

#### 2.2. Methods

Samples for the scanning electron microscopy (SEM) were broken with freeze-fracturing process in liquid nitrogen. Fractured surfaces were observed using Zeiss SUPRA 35 scanning electron microscope at accelerating voltages of 10 kV.

The Shore A hardness tests after 5 s of loading was made with HDA 100-1 Shore A Digital Durometer (Zwick GmbH & Com, Ulm, Germany) and in accordance ISO standard [12] with some modifications (different storage times were used). The base and catalyst components were mixed manually, and three samples for every material, each measuring 6 mm in thickness and 40 mm in diameter, were cured in a steel mould. Hardness was measured for each sample at five different points, which there were at least 5 mm from the sample margin. Measurements after 1 h, 7 days and 28 days of storing in distilled water at  $37 \pm 1^{\circ}$ C were made. As soon as measurements were finished, the sample was immediately immersed in water.

Tensile strength tests were performed on dumbbellshaped 5B specimens [13] which were cut from cured in gypsum mold, squar plates measuring 70 × 70 mm and 1 mm thick. The samples were stored in distilled water at 37 ± 1°C. Investigations had been realized after 24 ± 1 h, 7 ± 1 days and 28 ± 2 days of aging. Prior to testing, the thickness and width of each specimen were measured using a digital caliper at the center and four other points of the test specimen, and the average values were used for crosssectional area calculations. Tensile tests were performed at 1 mm/min cross-head speed. Tensile strength (T<sub>s</sub>) was calculated according to the following equation:

$$T_s = \frac{F_T}{A} \tag{1}$$

where  $F_T$  is the maximum force (N) and A is the initial cross-sectional area (mm<sup>2</sup>).

The sorption and solubility were determined using a method based on the ISO standard [19] with modifications. Test samples measuring 50 mm in diameter and 0.5 mm in thickness were manufactured in stainless steel molds. Ten samples of each material were cured. The samples were weighed on an AS 110/C/2 analytic scale (Radwag, Radom, Poland) with accuracy of 0.0001 g and were placed into desiccators with freshly dried silica gel. The desiccators were moved into a dryer at  $37 \pm 1^{\circ}$ C. The samples were weighed every 24 h until the everyday changes in mass were not exceed 0.0002 g. Stabilized values were noted as m1 "conditioned mass". The dried samples were placed into distilled water at  $37 \pm 1^{\circ}$ C and stored 7 days and 28 days. Next, the samples were taken out from water, the visible moisture was removed and then samples were weighed again. The registered mass was noted as  $m_2$  and the samples were dried in desiccators in accordance with previously described procedure until achieve stable mass noted as m3. The sorption and solubility were calculated using the following equations [14]:

$$w_{sp} = \frac{m_2 - m_3}{m_1} \times 100\% \tag{2}$$

$$w_{sp} = \frac{m_2 - m_3}{m_1} \times 100\% \tag{3}$$

where  $w_{sp}$  is sorption,  $w_{sl}$  is solubility,  $m_l$  is the initial mass,  $m_2$  is the mass after storing in distilled water, and  $m_3$  is the mass after the final drying.

Tensile bond strength between the silicones and the PMMA denture base resin was investigated with method presented in [12] The square samples finally measured circa  $25 \times 25$  mm and the thickness  $3.1 \pm 0.2$  mm) of heatcured Vertex Rapid Simplified (Vertex-Dental B.V., Zeist, The Netherlands) were cut from larger, previously cured plates. Each sample was preliminarily wet-ground on abrasive papers (Struers A/S: Copenhagen, Denmark) in the grit size sequence 220 and 320. Next all samples thoroughly rinsed and their surfaces were ultimately wetground by 500-grit abrasive paper until removing the scratches from previous grinding. The PMMA samples were immersed in distilled water for 28 days  $\pm$  5 h and at  $37 \pm 1^{\circ}$ C. After storing PMMA plates were taken out from the bath, dried with compressed air to remove moisture and the bonding agent Ufi Gel SC Adhesive (VOCO, Cuxhaven, Germany) was applied on the both plates. The first plate was placed on the compression table mounted to the testing machine. A polyethylene ring (internal diameter of 11 mm and a thickness of 3±0.2 mm) was placed on the first plate and mixed silicone material was injected into the ring and a second acrylic plate was placed on the material. The sample was compressed with a force of 30 N at standard testing machine. Teen samples were made from

each material. After crosslinking the samples were aged in distilled water at 37  $\pm$  1°C for 23  $\pm$  1 h. After storing a handles were attached by an auto-polymer to the each sample and samples were immersed again for a next hour (so total aging time was 24  $\pm$  1 h). After completed aging tensile testing was performed. A cross-head speed was 10 mm/min. The bond strength  $\sigma_B$  was calculated according to the following equation:

$$\sigma_B = \frac{F_{max}}{4} \tag{4}$$

where  $F_{max}$  is the maximum force (N) and A is the initial area of the bond between the silicone and PMMA resin (mm<sup>2</sup>).

The results were subjected to statistical analysis with Statistica 12 software. The distributions of the residuals were tested with the Shapiro–Wilk. The equality of variances was tested with the Levene test. When the distribution of the residuals was normal and the variances were equal, the one-way ANOVA or two-way ANOVA tests with Tukey HSD post-hoc test were used ( $\alpha = 0.05$ ).

# **3. Results**

The scanning electron microscopy (SEM) images presenting morphologies of materials after polymerization were shown in Figure 1. For the matrix material (control sample) filler was not visible. For the fractured samples (investigated materials) in the matrix F1 and F2 fillers were clearly visible (Figure 1 b and c respectively).

The results of Shore A hardness measurements were presented in Figure 2. The results of statistical analyses were shown in Table 2 and Table 3. The hardness values were statistically different for investigated materials and aging times. For a given filler concentration materials with F1 show higher hardness vales than F2. The higher fillers concentrations resulted in higher hardness values. With prolonging of aging time the values of hardness increased. After the first day the increase in hardness was from 5 to 19%, and was higher for materials with lower fillers concentrations. After 27 days increase in hardness was from 16 to 18 % for all materials.

The results of tensile strength measurements were presented in Figure 3. The results of statistical analyses were shown in Table 2 and Table 4. The increasing fillers concentration resulted in significant increase of tensile strength values and it was much more effective for materials with filler F1. The highest average value was 2.61 MPa and it was noted for 15 % of F1 filler. This value was two and a half times higher than for materials with 10% of filler. The aging time generally had no impact on tensile strength during investigated period.

The results of sorption and solubility were presented in Figure 4 and Figure 5. The materials did not differ each other in a statistically significant way (Table 2). The ANOVA test results show statistically significant differences in sorption values with increasing aging time, but it was not confirmed with post-hoc test. Anyway, in Figure 4 the higher sorption for materials with 15 % of both fillers after 28 days of aging is visible.

The results of bond strength test were presented in Figure 6. The results show statistically significant differences for all materials. The best values were obtained for materials concentration of 15%.

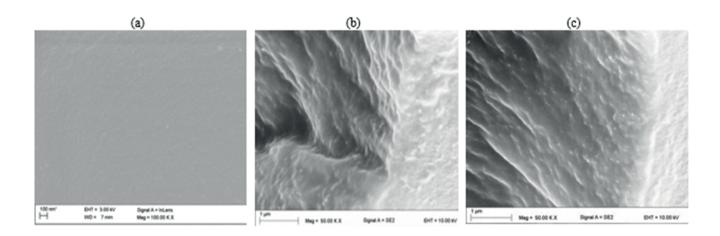


Fig. 1. Representative scanning electron microscopy (SEM) images presenting the morphologies of the fractured samples of polymerized matrix (a) and material after introducing 15 % of filler F1 (b) and F2 (c)

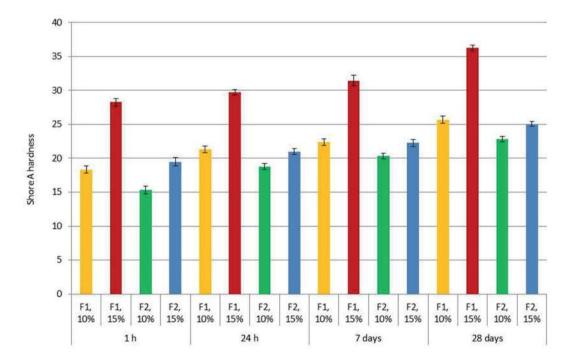


Fig. 2. Mean hardness values in Shore A units and standard deviations

# Table 2.

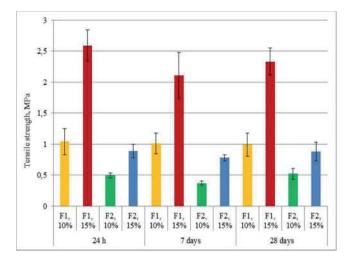
The results of statistical assessment of influence the material type and aging time on Shore A hardness, tensile strength, sorption, solubility and bond strength performed using two-way ANOVA or ANOVA tests (p < 0.05)

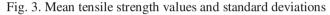
Property	- F-test value	р	
Shore A hardness	- r-test value		
Material type	261.42	< 0.01	
Aging time	4.35	0.018	
Material type $\times$ Aging time	1.52	0.19	
Tensile strength			
Material type	261.42	< 0.01	
Aging time	4.35	0.02	
Material type × Aging time	1.52	0.19	
Sorption			
Material type	1.02	0.4	
Aging time	9.24	< 0.01	
Material type $\times$ Aging time	1.37	0.27	
Solubility			
Material type	1.921	0.15	
Aging time	2.28	0.14	
Material type × Aging time	0.87	0.47	
Bond strength			
Material type	59.79	< 0.01	

Table 3.

The results of Tukey HSD post-hoc test of Shore A hardness for particular materials and aging times, the same uppercase letters; (A–D) for each column and lowercase letters; (a–d) for each row are not significantly different at the p < 0.05 level

Aging	Filler type and concentration			
time	F1, 10%	F1, 15%	F2, 10%	F2, 15%
1 h	A;a	A;b	A;c	A;d
24 h	B;a	B;b	B;c	B;a
7 days	C;a	C;b	C;c	C;a
28 days	D;a	D;b	D;c	D;d





#### Table 4.

The results of Tukey HSD post-hoc test of tensile strength for particular materials and aging times, the same uppercase letters; (A–C) for each column and lowercase letters; (a–d) for each row are not significantly different at the p < 0.05 level

Aging	Filler type and concentration				
time	F1, 10%	F1, 15%	F2, 10%	F2, 15%	
24 h	A;a	A;b	A;c	A;a,c	
7 days	A;a	B;b	A;c	A;a,c	
28 days	A;a	A,B;b	A;c	A;a,c	

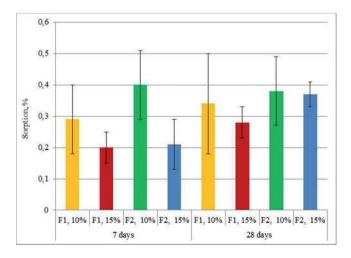


Fig. 4. Mean sorption values and standard deviations

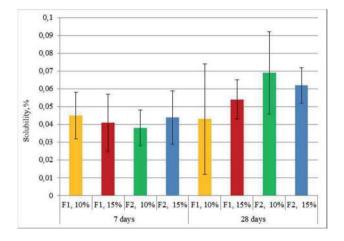


Fig. 5. Mean solubility values and standard deviations

#### Table 5.

The results of Tukey HSD post-hoc test of bond strength of different materials, the same uppercase letters (A–D) are not significantly different at the p < 0.05 level

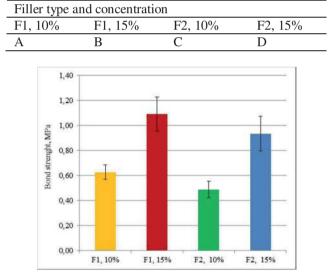


Fig. 6. Mean bond strength values and standard deviations

# 4. Discussion

One of the most important property of prosthetic silicones is their hardness measured in Shore A scale. The materials are classified as soft or super-soft and their initial hardness 24 h after crosslinking should be between 25 and 50 units or below 25 units, respectively [3]. Among the tested materials only one (F1, 15%) can by classified as soft, the others can by categorized as super soft. It should be noted, that most of materials available on the market can

be categorized as soft [15-19] and only a few as super soft [17-19]. The changes in hardness of all investigated materials were in accordance of ISO standard. The hardness stability in time was also relatively good, especially if we consider that several commercially available materials show increase in hardness higher than 50% [15,18], which is the value greater more than two and a half times than in the case of the examined experimental materials. The hardening of materials was probably related with cross-linking occurring during the experiment [20].

Tensile strength of investigated materials was too low for materials with 10 % of F2 filler because obtained values are too low to provide the appropriate value of bond strength [8]. The obtained values were not aging – time dependent, what must be find as beneficial.

The water sorption and solubility of soft lining materials are generally dependent on the hydrophilicity of the matrix and on the leachable components [5]. This properties are related with changes in the hardness, bond strength, discoloration, odour, and easier bacterial adhesion to the surface a can be different [19,21]. The silicones are well known prosthetic materials characterized by low sorption and solubility values [5] and it corresponds very well with presented results. Additionally the obtained values are similar to those for other materials available on the market [14,22,23]. The bond strength to the PMMA denture base materials is an important factor directly related with durability of soft lining materials. Debonding of silicone soft lining from denture base is one of the causes of lining replacement. Bond strength can be investigate with peel, shear and tensile tests but only last one is recommended by ISO standard. The materials containing 15% of fillers F1 and F2 meet the requirements of the ISO standard for soft and extra soft materials, respectively. Nonetheless, all failures observed during bond tensile tests for both materials were cohesive. This shows that bonding agent presents higher strength than silicone based materials. It can indicate that mechanical properties, like tensile strength and tear resistance, should be enhanced in future. It can be realized by obtaining better distribution of filler in polymeric matrix. The SEM investigations shown numerous aggregations measuring a few dozen nanometres, when average particles of fillers were below 10nm. However, mixing the fillers with matrix at laboratory scale is difficult. The elastomers at larger scale can be mixed more effectively with use of planetary mixers, sigmablade mixers, two-roll mills or twin-screw extruders. Additionally, in future some tests can be performed to optimize composition of filled systems and the ratio of Si-H to vinyl groups. The optimum cure ratio can be measuring by the Shore A hardness of cured samples at different ratios.

#### 5. Conclusions

In the work were achieved silicones with satisfying properties of two from four studied materials. The type of the filler significantly affected the investigated properties. Hardness of materials increased with aging time, which was probably related with cross-linking continuing with the aging at elevated temperature. More effective technology of filler compounding should be performed to enhance some mechanical properties.

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