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Physico-Mechanical Behavior of Room Temperature Vulcanized Maxillofacial Silicon after Addition of Glass Flakes

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Abstract

The aim of this study was to assess the effect of glass flakes addition on some physical and mechanical properties of a maxillofacial silicone material. Material and method: A room temperature vulcanized (RTV) maxillofacial silicone (VST-50, Factor II Inc., USA) and Micronized glass flake, surface pre-treated with silane coupling agent (GF002, Glass flake Ltd, Leeds, UK) were used in this study. Two hundred (200) specimens were prepared and divided into five main groups based on the tests conducted (tear strength, tensile strength, elongation percentage, surface hardness and surface roughness). Then, each group was farther divided into four subgroups according to weight percentage of glass flakes as follow; control group (0%), other groups contain 0.5%, 1% and 1.5% of glass flake micro-particles (10 specimen from each group). Results: The collected data were analyzed with a one-way ANOVA and LSD multiple comparison test were utilized to show the differences among four studied groups. The 1% glass flakes-incorporated specimens exhibited the highest mean values of tear strength, tensile strength and elongation percentage. While, the highest mean values of hardness and roughness were obtained with 1.5% specimens.

Keywords: *Glass flakes, Maxillofacial silicone material (VST-50), Tear strength, Tensile strength, elongation percentage, Hardness, Roughness.*

Introduction

Prosthetic devices have been widely used to compensate for defects either from congenital or acquired origin (such as disease or trauma), the need for prosthetic rehabilitation has proportionally increased since surgical intervention may not always be possible because of the location and size of the defect [1]. Various types of polymeric materials such as polyvinyl chloride, polyurethanes, poly (methyl methacrylate), chlorinated polyethylene and silicones (poly(dimethyl siloxane); PDMS) elastomers are well-known materials for the production of maxillofacial prosthesis [2]. However, given their low surface-free energy (hence poor wettability), they can cause adverse reactions such as tissue irritation, abrasion and ulceration [3]. The ideal mechanical and physical characteristics of the maxillofacial materials should be comparable to that of the tissue to be substituted. These materials should be non-toxic and tissue-compatible, should be colored with intrinsic and extrinsic pigments, should have easy handling and processing and should not deteriorate during clinical use [4]. Regarding the vulcanizing process using heat or not, silicones are available as heat vulcanized (HTV) or room temperature vulcanized (RTV). In comparison to other materials, both HTV and RTV have high tear resistance, because the specimens do not tear but expand, as in tensile elongation and high percent elongation ranging from 422% to 445% [2]. Medical grade silicone adhesives have been combined with RTV silicone based in various ratios to control the elastic properties [5]. Due to hydrophobic nature, these have low adhesion to non-silicone adhesive material and suffers from limited working time [6]. Surface-treated silica with small particle size fillers had increase the surface area and significantly enhance the physical and mechanical properties of

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silicone elastomers. Lately, researchers have found even stronger enhancement through the use of nano silica powder, which has an even larger surface area than micrometer-size silica powder [7]. Glass flake has been utilized as a reinforcing agent in many industrial and commercial applications, their manufacturers proved that its addition to some thermoplastics has resulted in a significant improvement in flexural properties and planar reinforcement [8]. The efficacy of the glass flakes reinforcement is strongly based on the interfacial adhesion between the glass and the surrounding polymer matrix [9].

Materials and Method

Materials

In the present study, a room temperature vulcanized (RTV) maxillofacial silicone (VST-50, Factor II Inc., USA) and Micronized glass flake, surface pre-treated with silane coupling agent (GF002, Glassflake Ltd, Leeds, UK) were used. The glass flake consists of flake particles of 1.3-2.3 μm thick with a range of diameters mostly below 50μm.

Specimen grouping

A total of two hundred (200) specimens were prepared and divided into five main groups based on the tests conducted (tear strength, tensile strength, elongation percentage, surface hardness and surface roughness) with 40 specimens for each test. Then, each group was farther divided into four subgroups according to weight percentage of glass flakes as follow; control group (0%), other groups contain 0.5%, 1% and 1.5% of glass flake micro-particles (10 specimen from each group).

Specimen preparation

Four plastic mold were fabricated using laser cutting machine (JL-1612, Jinan Link Manufacture and Trading Co., Ltd., China). Each mold was fabricated into the specified dimension for each test. The thickness of the plastic sheet used for tear, tensile and percentage elongation tests was 2 mm, while the sheet used for hardness and roughness tests was 6 mm. Around 40 mm × 40 mm square test specimen was used for hardness and roughness tests in accordance with ISO 7619-1:2010 (10). Whilst, an angle test specimen without nick with dimensions in accordance with ISO 34-1:2010 was selected for tear strength test (11). While, a dumb-bell specimen was chosen for tensile strength and elongation at break test according to ISO 37:2011 (12). For each plastic mold, two sheets one on top and other glued sheet on the bottom were fabricated with the same outer dimensions of the mold to sandwich the mold between them (Figure 1).

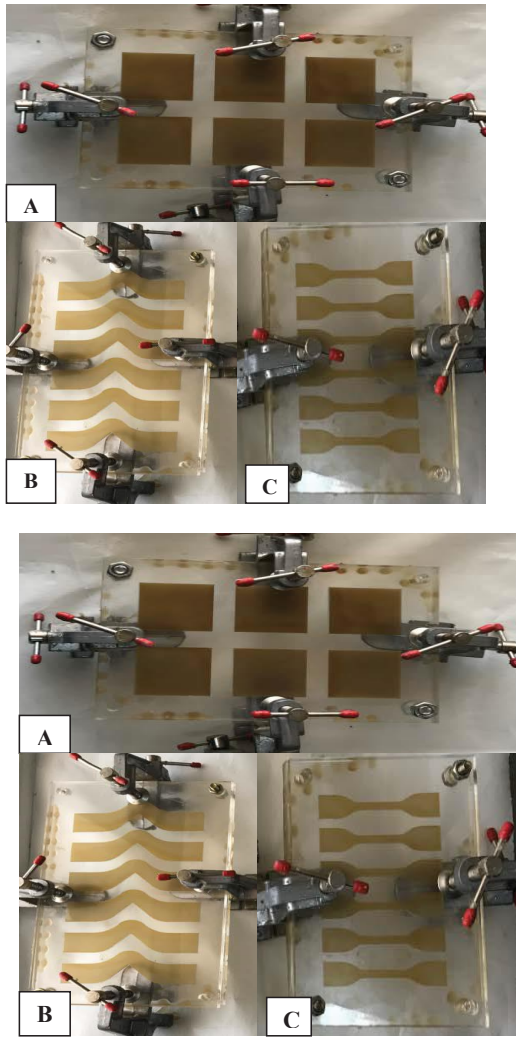

Figure 1. Metal molds for A) Hardness and roughness tests, B) Tear strength test, C) Tensile strength and elongation tests

The mixing of RTV silicone was done according to manufacturer's instructions. For control groups, part A (base) of silicone and part B (crosslinker) was weighed with a ratio of 10:1 and mixed by a vacuum mixer (Multivac 3, Degussa, Germany) for 5 minutes.

Testing method

Tear strength test was done by adjusting the test specimen on a universal testing machine (WDW-20, Laryee Technology Co. Ltd., China) and stretching

it with a speed of 500 mm/min until the break. The thickness of the specimen was measured at the area of the right angle by a digital caliper before testing. The tear strength was measured by dividing the maximum force obtained from the universal testing machine by the thickness of the specimen.

For tensile strength test, the thickness and the width of the narrow portion of the specimen were measured by a digital caliper at 3 areas; at the two ends and in the middle. The average of the 3 readings was considered as the thickness and the width of the specimen. Then the specimen was fixed on a universal testing machine and stretched at a crosshead speed of 500 mm/min until it breaks. The tensile strength was calculated using the following equation: recorded for each specimen, and the transverse strength was calculated using the following equation:

$T_S = Fm / Wt,$

Where Fm is the maximum force in Newton, W is the average width of the narrow portion of the specimen in millimeters, and t is the average thickness of the specimen over the narrow portion in millimeters.

On the other hand, elongation percentage was assessed using an extensometer for each tensile specimen and percentage of elongation was calculated using the following equation:

$$E_b\% = \frac{L_b - L_0}{L_0} \times 100$$

Where L_o is the initial test length in millimeters, L_b is the test length at the break in millimeters.

Statistical analysis

Statistical analyses were performed using SPSS (statistical package for social science – version 24) computer software. Descriptive statistics were made which include Means and Standard deviation. The homogeneity of variances was confirmed by Levene test and also inferential statistics includes; One-way analysis of variance (ANOVA) was used to compare means among all groups and LSD multiple comparisons test was utilized to show the significance among different groups.

Results

The results of this study agree with the hypothesis that the silica filler-treated will increase the surface area of silicone elastomers and is an important factor to enhance the physical and mechanical properties.

Table 1. Descriptive statistics, one-way ANOVA and multiple comparisons test of tear strength values among different concentrations of incorporated glass flakes.

Group	Descriptive statistics				ANOVA	
	N	Tear strength Mean ± S.D. (N/mm)	95% C.I. for Mean		F-test	p-value
			Lower Bound	Upper Bound		
0% of glass flakes	10	19.45±2.70	15.50	24	5.054	0.005
0.5% of glass flakes	10	21.65±1.70	19.50	25		
1% of glass flakes	10	22.80±2.12	20	27		
1.5% of glass flakes	10	20.75±1.08	19	22.5		

Cont... **Table 1. Descriptive statistics, one-way ANOVA and multiple comparisons test of tear strength values among different concentrations of incorporated glass flakes.**

Groups Comparison		Mean Difference	p-value	Sig.
0% of glass flakes	0.5%	-2.20	0.018	*
	1%	-3.35	0.001	***
	1.5%	-1.30	0.153	N.S.
0.5% of glass flakes	1%	-1.15	0.205	N.S.
	1.5%	0.90	0.319	N.S.
1% of glass flakes	1.5%	2.05	0.027	*

Table 2. Descriptive statistics, one-way ANOVA and multiple comparisons test of tensile strength values among different concentrations of incorporated glass flakes.

Group	Descriptive statistics				ANOVA	
	N	Tensile strength Mean ± S.D. (MPa)	95% C.I. for Mean		F-test	p-value
			Lower Bound	Upper Bound	10.51	0.000
0% of glass flakes	10	4.41±0.53	3.50	5		
0.5% of glass flakes	10	5.53±0.75	4.50	6.75		
1% of glass flakes	10	5.88±0.59	4.88	6.88		
1.5% of glass flakes	10	5.24±0.54	4.80	6.50		

Groups Comparison		Mean Difference	p-value	Sig.
0% of glass flakes	0.5%	-1.12	0.000	***
	1%	-1.47	0.000	***
	1.5%	-0.83	0.005	**
0.5% of glass flakes	1%	-0.35	0.211	N.S.
	1.5%	0.29	0.290	N.S.
1% of glass flakes	1.5%	0.64	0.024	*

Table 3. Descriptive statistics, one-way ANOVA and multiple comparisons test of elongation percentage among different concentrations of incorporated glass flakes.

Group	Descriptive statistics				ANOVA	
	N	Elongation % Mean ± S.D.	95% C.I. for Mean		F-test	p-value
			Lower Bound	Upper Bound	39.28	0.000
0% of glass flakes	10	361.56±50.67	325.31	397.81		
0.5% of glass flakes	10	488.63±35.03	463.56	513.69		
1% of glass flakes	10	546.26±40.03	517.63	574.90		
1.5% of glass flakes	10	446.59±27.48	426.93	466.26		

Groups Comparison		Mean Difference	p-value	Sig.
0% of glass flakes	0.5%	-127.07	0.000	***
	1%	-184.70	0.000	***
	1.5%	-85.03	0.000	***
0.5% of glass flakes	1%	-57.63	0.002	***
	1.5%	-42.03	0.022	*
1% of glass flakes	1.5%	99.67	0.000	***

Table 4. Descriptive statistics, one-way ANOVA and multiple comparisons test of shore A hardness values among different concentrations of incorporated glass flakes.

Group	Descriptive statistics				ANOVA	
	N	Hardness Mean ± S.D. (units)	95% C.I. for Mean		F-test	p-value
			Lower Bound	Upper Bound		
0% of glass flakes	10	32.46±2.53	30.65	34.27	14.31	0.000
0.5% of glass flakes	10	35.16±1.41	34.15	36.17		
1% of glass flakes	10	36.32±1.58	35.18	37.45		
1.5% of glass flakes	10	37.47±1.38	36.48	38.46		
Groups Comparison		Mean Difference	p-value		Sig.	
0% of glass flakes	0.5%	-2.704	0.002		**	
	1%	-3.856	0.000		***	
	1.5%	-5.007	0.000		***	
0.5% of glass flakes	1%	-1.152	0.159		N.S.	
	1.5%	-2.303	0.007		*	
1% of glass flakes	1.5%	-1.151	0.160		N.S.	

Concerning the results of the present in vitro study, the superior tensile strength, tear strength and elongation percentage of the micro-particles glass flakes treated groups in comparison to the non-treated control group were likely attributed to the presence of high molecular weight Polydimethylsiloxane (PDMS) chains combinated with glass micro-particles. Cross-linking of a high molecular weight polymer produces a highly elastic material, thus increasing the viscosity of the resulting polymer as well increase the length of PDMS chains and produce a very elastic cross-linked network and this may considered for the high mechanical properties, thus the resulting polymer matrix is able to withstand greater deformation without rupturing or tearing. Tear strength is the most important property of maxillofacial silicone which indicates the thin margin integrity and durability of maxillofacial prosthesis [14]. The results of tear strength test indicated that the tear strength was significantly increased ($p > 0.005$) when the glass flakes were incorporated to the silicone (Table 1). In all experimental groups, the flakes mechanically interacted with the silicone matrix and associated strongly with the polymer chains thus increasing tear strength values. Tear strength of a material depends on the ability of the polymer to scatter energy at the area of the crack as tearing propagates. Micro-sized fillers dissipate strain energy within the matrix of the polymer, thus making it more resistant to tearing and a higher applied force is needed to completely break the polymer chains. This explains the increase in tear strength, as explained by Kraus in 1978 and Sun et al. (2009) [15,16]. For the control group, the interaction between the –OH groups and PDMS chains is not strong enough to prevent the material from rupturing under an applied force. Generally, the tensile strength depends greatly on the crosslinking between the silicone chains [4]. It seems that the results of tensile strength was significantly increased after incorporation of the glass flakes into the RTV silicone specimens ($p > 0.000$) (Table 2). The silicate groups act as multifunctional crosslinks through the generation of new bonds with silicone chains. These multifunctional crosslinks increase the overall crosslinking density of the cured silicone and make it stiffer and stronger. When the polymer is subjected to tensional forces, these crosslinks inhibit the chains from sliding over each another as well prevent the breakage, thus increasing the tensile strength [17]. The elongation of cured silicone elastomer depend greatly on the cross-linking system [4]. One of the reinforcement mechanisms of silica is that glass micro-particles act as multifunctional cross-links via formation of strong hydrogen bond between its surface hydroxyl group and PDMS chains; these multifunctional cross-links increase the overall cross-linking density of the polymer and make it more stiff and strong [18]. In this study, the experimental groups revealed reduction in tear strength, tensile strength and percentage of elongation when the concentration of glass flakes was increased to 1.5% might be due to the agglomeration of flakes particles within the matrix. These agglomerates are formed when two or more filler particle aggregates bind together by weak electrostatic Van der Waals forces. These agglomerates act as stress concentration

areas within the polymer matrix. When external forces are applied to the polymer, the agglomerates break and weaken the matrix leading to crack propagation, as explained by [13].

Conclusion

The addition of glass micro-particle is necessary for achieving certain degree of reinforcement that leads to significant improvement of the mechanical properties. Within the limitations of this in vitro study, the following conclusions can be obtained that the addition of various concentration of glass flake to RTV silicone material considerably improved the mechanical and physical properties of the material. The 1% glass flakes-incorporated specimens revealed the highest mean values of tear strength, tensile strength and elongation percentage. While, the highest mean values of hardness and roughness were obtained with 1.5% specimens.

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Conflict of Interest: None to declare.

Ethical Clearance: All experimental protocols were approved under the College of Dentistry/ University of Baghdad, Iraq and all experiments were carried out in accordance with approved guidelines.

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