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Effect of Addition of Composite Polyamide Micro Particles and Silicone Dioxide NanoParticle on Some Mechanical Properties of Room Temperature Vulcanized Maxillofacial Silicone Elastomer Before and after Artificial Aging

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Abstract

Background: The most common reason for re-making a maxillofacial prosthesis is the degradation of the mechanical properties of the silicone.

Aim of this study: To assess some mechanical properties of VST-50F maxillofacial silicone reinforced with a composite of silicon dioxide nanoparticle and polyamide-6 microparticle before and after artificial aging.

Material and Method: Preparing 240 samples tested for tear strength, tensile strength and elongation percentage, hardness, and roughness before and after aging. The Silicon dioxide was added in concentrations of 1% by weight and Polyamide-6 in the concentration of 0.25% and 0.5% by weight to the VST-50F RTV maxillofacial silicone. The one-way ANOVA and post hoc tests were used for inferential statistics.

Results: The one-way ANOVA showed a highly significant difference between all tested groups. The effect of the addition of composite fillers showed an increase in tear strength, hardness, and surface roughness but a decrease in tensile and percentage of elongation. However, the effect of artificial aging showed increased in tear strength, Percentage of elongation, and surface roughness, but a decrease in tensile strength and hardness.

Conclusion: Addition of composite of fillers into the silicone elastomer allowed enhancement of some mechanical properties. The composite of different types of filler reinforcement improves the anti-aging properties of silicone and maintain some of the mechanical properties to enhance the service life.

Keywords*: Composite, silicone dioxide, polyamide, Maxillofacial silicone, Mechanical properties, artificial aging.*

Introduction

The distortion in appearance and function could be restored with a maxillofacial prosthesis that reproduces the natural features of the lost tissues from acquired, developmental, and congenital head and neck defects [1]. The first treatment of choice is plastic surgery. However, when a medical procedure is ill-advised, because of undesirable conditions, restoration with maxillofacial prosthesis could be an alternative method for improving patient's appearance and confidence and encouraging their resumption to society [2]. Rahman et al. [3] after systematic review for various researches concluded that there was no ideal maxillofacial material that could resist different aging conditions. Liu et al. [4] concluded that merging two types of micro-particles in specific percentages could give a maxillofacial prosthesis closer to ideal properties. On the other hand, Nano- and micro-filler mixture is a practical approach to improving the strength and the features of material [5].

This study assessed some mechanical properties of VST-50F maxillofacial silicone reinforced by a composite of silicon dioxide nanoparticle and

polyamide-6 microparticle before and after artificial aging.

Materials and Method

The materials used in this study were the VST 50F RTV silicone (Factor II Inc., USA), SiO2 (Sky Spring, Inc. USA) and PA-6 (Changfeng Chemical Co., Ltd. China).

The SiO2 was added in 1% by weight and PA-6 in the 0.25% and 0.5% by weight to the VST-50F silicone.

A total of 240 samples had been prepared and separated into four groups according to the performed tests, so each test included 60 samples and each test group further subdivided into six sub-groups (Control, Control/aged,0.5PA+1SiO2,0.5PA+1SiO2/aged group,0.25PA+1SiO2,0.25PA+1SiO2/aged) with ten samples for each sub-division.

The shapes and dimensions of the molds for the test samples were designed utilizing AutoCAD 2013 and fabricated with CNC machine into which the silicone poured. The two templates had dimensions of 30 cm length x 20 cm width x 2.2 ± 0.05 mm thickness for tear and tensile strength test and 6± 0.05 mm thickness for hardness and roughness tests [2] .

The fabrication of the samples for each of the experimental groups started by weighing the fillers in the mixing bowl then the silicone base added and mixed for 10 min. The mixing was conducted under vacuum to eliminate air entrapment in the mix for the first three minutes. The vacuum was switched off to prevent the suction of the nanoparticles and then it was switch on for the last 7 min. The vacuum pressure was -28inch Hg, and the speed of the mechanical mixer was 140±10 RPM. Before adding the catalyst, the mixture was left for 2 minutes to cool — the catalyst mixed with the base mixture for 5 minutes ± 5 seconds [1] .

Pouring of the material was carried out under a temperature of 23 ±2°C, and the humidity around 50±10% [6] . The homogeneous mixture was dispensed carefully into a plastic-syringe of 60 mL (Figure 1). The acrylic mold placed over a dental vibrator working at a low vibration and the mixture poured in an excess amount from the disposable syringe. The cover was placed over the poured material in the matrix, starting from one end part of the cover by resting the margin and lifting the margin of the other end part. Then the lid was gently lowered slowly and carefully onto the matrix to allow for the escape of the excess material out of the mold. After that, a load of one kilogram placed over the middle part of the mold's cover, and the cover secured in place with nuts and G-clamps, then the weight was removed (Figure 2). The mold was fixed firmly until the samples hardened [1] .

All the samples organized, and the tests performed according to ISO 23529 [6] specifications.

The following four tests were performed to assess the various mechanical properties of the VST-50F maxillofacial silicone:

1. Tear strength test: According to ISO 34-1 [7] specifications, the samples had been prepared and tested by the universal testing machine clamps (Laryee Technology Co., Ltd., China). The tear strength sample was the type C tear sample.

Tear strength=f/d; f: The maximum force required for sample breaking in kilonewton. d:The mean thickness of each sample in the meter.

2. Tensile strength and elongation percentage tests: The testing procedure accomplished according to ISO 37 [8] specifications with dumbbell-shaped.

Ultimate tensile strength=Fm/Wt; Fm: The maximum force recorded at break(N), Wt: The original cross-sectional area of the sample(mm2).

The percentage of elongation was measured before sample fracture with a tensile strength test, according to ISO 37 [8] .

Eb (Elongation percentage at break)=100×(Lb-L°)/L°

L°: is the original length in mm, Lb: is an extension at the break in mm.

3. Hardness Test: According to ISO 48-4 [9] specification, the testing procedure accomplished. The samples marked with five points, one at the center and the others 6mm away at each direction around the center point. Shore A hardness durometer mounted on the mechanical stand used for measurement. The device was forced firmly by hand clamp on a mechanical stand over the surface of the sample for 1 second with a 1Kg load according to specification, and the mean of 5 reading recorded [9] .

4. Surface Roughness Test: All samples prepared according to ISO 48-4 [9] specifications. Portable digital roughness tester with (0.001µm) accuracy was used to conduct a surface roughness test. The device mounted on a stand and adjusted in a way so that the stylus just contacted the sample surface in 3 various places so that three measures obtained for each sample and their mean value represented the roughness value [10].

SEM (AIS2300C, Angstrom Advanced Inc, USA) scanning the dispersion of the composite fillers within the silicone matrix.

SPSS software v23.0 used for data analyzes for the descriptive and inferential statistics with a confidence interval of 95%.

Figure 1: The mixture was poured by plastic syringe into the mold placed over a dental vibrator.

Figure 2: Mold tightened with nuts and G-clamps.

Results

ANOVA test showed that a highly significant difference between all tested groups.

The results showed no significant increase in the tear strength after fillers reinforcement.

The effect of artificial aging for 150 hours showed that the tear strength of the control group decreased significantly, but for the reinforced silicone increased. It was a highly significant difference for 0.5PA+1SiO2 and a non-significant difference for the 0.25PA+1SiO2 group in comparison to the control group.

The results showed a non-significant decrease in the tensile strength after fillers reinforcement.

The effect of artificial aging for 150 hours showed that the tensile strength decreased highly significantly for the control group and the reinforced silicone. It was a highly significant difference for 0.5PA+1SiO2 and 0.25PA+1SiO2 groups in comparison to control group before aging and the non-significant difference in contrast to the control group after aging.

The results showed a significant decrease in the percentage of elongation after 0.5PA+1SiO2 fillers reinforcement and a highly significant decrease after the 0.25PA+1SiO2 fillers reinforcement.

The effect of artificial aging for 150 hours showed that the percentage of elongation of the control group decreased non-significantly, but for the reinforced silicone increased highly significantly. It was a non-significant difference for both percentages in comparison to the control group before and after aging.

The results showed a highly significant increase in the hardness after filler reinforcement.

The effect of artificial aging for 150 hours showed that the hardness of the control group increased non-significantly, but the reinforced silicone decreased highly significantly. It was a non-significant difference for both percentages in comparison to the control group before and after aging.

The results showed a highly significant increase in the surface roughness after 0.5PA+1SiO2 fillers reinforcement and non-significantly increased after the 0.25PA+1SiO2 fillers reinforcement.

The effect of artificial aging for 150 hours showed that the surface roughness increased non-significantly for the control and the reinforced silicone groups. It was highly significantly different for 0.5PA+1SiO2 group in comparison to the control group before and after aging

and also highly significantly different for the 0.25PA+1SiO2 group, in contrast, to control before aging, but it was only significantly different in comparison to control after aging (Table 1 and 2).

The SEM results showed a good dispersion of the Nano-fillers within the silicone matrix (Figure 3),

Table (1) Mean values of conducted tests before and after artificial aging

Tests Groups	Tear strength (N/mm)	Tensile strength (MPa)	Elongation percentage (%)	Shore A hardness (IU)	Surface roughness(µm)
Control	27.0056	6.2284	596.6517	27.9160	0.3607
Control /aged	24.7655	5.0542	594.6524	28.5500	0.4347
0.5PA+1SiO2	28.1010	5.6591	571.9662	30.8200	0.5195
0.5PA+1SiO2 /aged	29.8892	4.9269	595.8612	28.2400	0.5871
0.25PA+1SiO2	27.2575	5.7993	569.4999	31.0500	0.4575
0.25PA+1SiO2 /aged	28.5915	4.9768	595.6062	28.3900	0.5491

(Table 2) Post-hoc Tukey HSD for all tests except Games-Howell for elongation test

Group 1	Group 2	Tear	Tensile	Elongation	hardness	roughness
control	Control /aged	0.050	0.000	1.000	0.752	0.368
	0.5PA+1SiO2	0.699	0.066	0.016	0.000	0.001
	0.5PA+1SiO2 /aged	0.005	0.000	1.000	0.982	0.000
	0.25PA+1SiO2	0.999	0.283	0.008	0.000	0.118
	0.25PA+1SiO2 /aged	0.306	0.000	1.000	0.911	0.000
Control /aged	0.5PA+1SiO2	.001	0.043	0.026	0.000	0.226
	0.5PA+1SiO2 /aged	.000	0.988	1.000	0.985	0.002
	0.25PA+1SiO2	0.021	0.006	0.013	0.000	0.990
	0.25PA+1SiO2 /aged	0.000	0.999	1.000	0.999	0.038
0.5PA+1SiO2	0.5PA+1SiO2 /aged	0.189	0.007	0.004	0.000	0.470
	0.25PA+1SiO2	0.874	0.981	0.977	0.996	0.565
	0.25PA+1SiO2 /aged	0.987	0.015	0.008	0.000	0.968
0.5PA+1SiO2 /aged	0.25PA+1SiO2	.012	0.001	0.002	0.000	0.013
	0.25PA+1SiO2 /aged	0.530	1.000	1.000	1.000	0.911
0.25PA+1SiO2	0.25PA+1SiO2 /aged	0.499	0.002	0.004	0.000	0.158

*P-value≤0.05=significant,P-value≤0.01=highly significant.

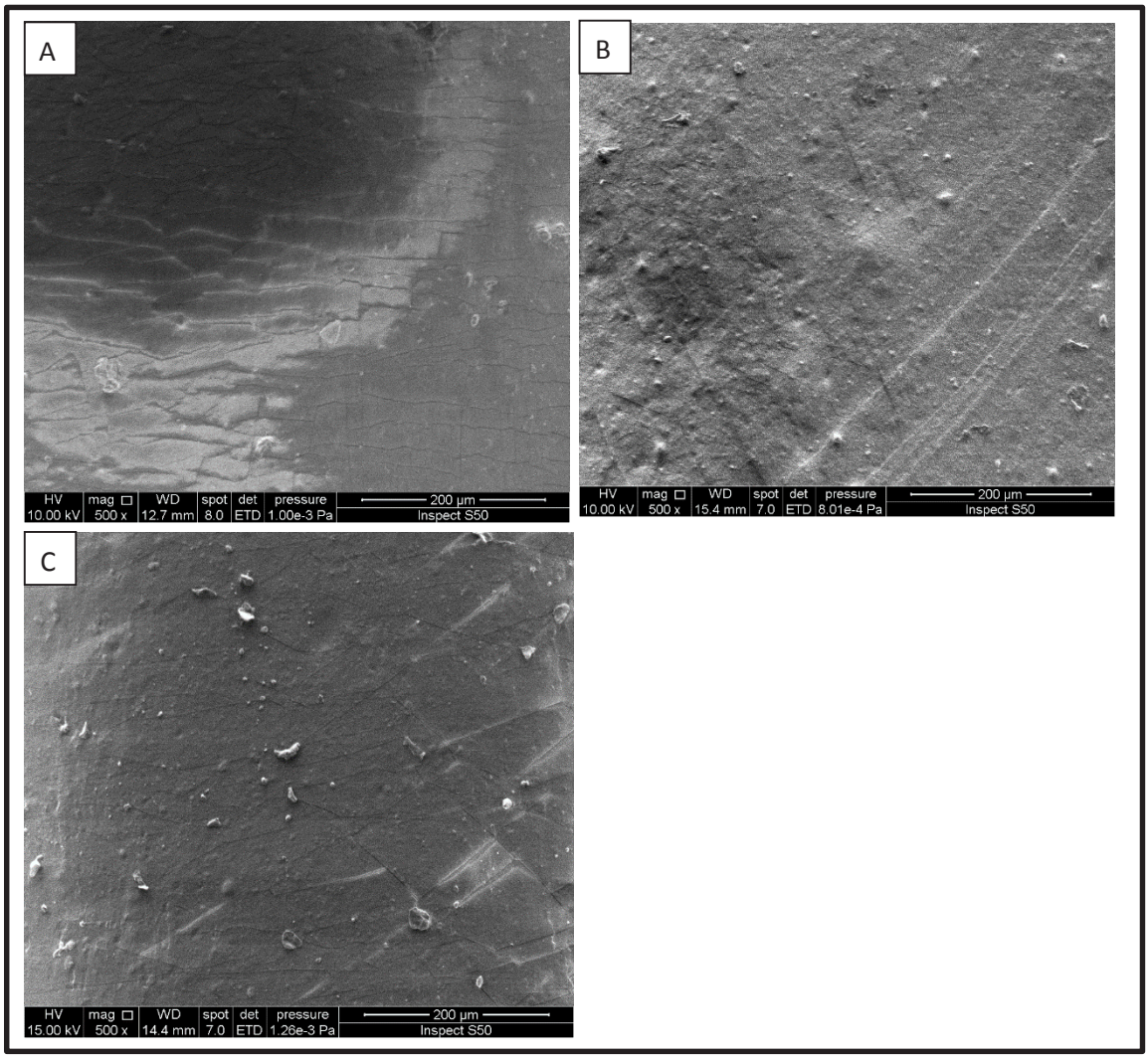

Figure 3: SEM image of VST-50F silicone elastomer. A: Before addition, B: after addition 0.5PA/1SiO2 C: after addition 0.25PA/1SiO2.

Discussion

Many types of research showed that most of the commercially present materials had not satisfied all of the required, ideal properties and the highest chance of early deterioration was the major limitation of silicone material that may exhibit reduced tear strength, somewhat fitting margin, discoloration of material and modified texture. It clarifies the constant increase in the number of researches to develop enhanced maxillofacial silicone material, by changing the formulation of previous materials or by reinforcement with various types of fillers in different percentages 11 . Silica particles can adsorb polydimethylsiloxane chains to its surface and form strong hydrogen bonds between its surface hydroxyl group and the oxygen in the polymer chain. These bonds increase polymer chains resistance to rupturing when subjected to tearing forces due to high shear strength between the nanofiller and the polymer chains 12 . Also, the PA-6 could form multiple hydrogen bonds among adjacent elements because of the high polarity natural of fillers due to the amide (-CO-NH-) groups. It is leading to an alteration in the whole density and more tear resistance of the polymer due to the formation of fillers with a three-dimensional network in the polymer matrix. On the other hand, rubber can scatter stress-energy close the apex of the growing cracks and thus increasing the strength in elastomers. So, this may explain the increase in tear strength after SiO2 and PA-6 filler reinforcement 13 . The hardness increase might be referred to the difference in the crosslink density with filler contents; the filler particles may cause a reduced in the distance among the crosslinks of the polymeric matrix thus the reduction of the softness of the material

contributed to the polymer and filler interaction 14 . As mentioned early, the SiO2 nanoparticles increase the cross-linking network that leads to improve the overall stiffness of the polymer and increase its resistance to tearing, but the higher cross-linker tightened the net to upper limit so reducing the flexibility and deteriorating tensile properties obtaining extremely brittle samples. The decrease in elongation could also be clarified by the reduction in the space between the fillers aspect ratio and contents. Therefore the elongation at fracture was decreased by the relatively reduced distance of rubber molecules 15 . On the other hand, the cross-linking was reversible for PA-6 particles with temperature and UV light inside the Weather-Ometer chamber and could result in a polymer with altered physical and mechanical properties 16 . The decrease in cross-linking leads to a reduction in tensile strength and an increase in the percentage of elongation. It may be because of chain scission that results from the generation of free radicals which react with oxygen leading to the formation of polymer oxy- and peroxy radicals via the enhanced chemical reaction (photo-oxidation). It influences the distribution of molecular weight and reducing the density of the network structure by breaking the bonds, commonly C-H bonds, between two diverse chains or within the same main chain due to strength value of material reduced 17 .

The particles of the nanofiller (SiO2) may be associated strongly with the polymeric chains even after severe condition. If these particles were detached, an increase in the porosity of the polymer and reduction in the hardness would be expected. So the surface roughness increase results from the formation of microcracks and pits on the surface level of material 10 .

Conclusions

Within the limitations of the study, the following were drawn: The addition of various types of fillers in appropriate proportions to silicone elastomer could enhance some of the mechanical properties. The composite of Nano-SiO2 and micro PA-6 fillers tend to increase in tear strength, hardness and surface roughness but a decrease in tensile and percentage of elongation. The composite of different types of fillers reinforcement is promising to improve the anti-aging properties of silicone and maintain some of the mechanical properties to enhance the service life.

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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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