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The Effect of Nano Titanium Silicate Addition on Some Properties of Maxillofacial Silicone Material

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ABSTRACT

Background: Silicone elastomers are the main materials used nowadays in fabricating facial prosthesis, due to their high compatibility, chemical inertness, elasticity and ability to be colored by pigments. But many other properties need to be improved in order to have a better clinical performance like increasing tear strength, tensile strength and bonding to acrylic resin. So an increasing number of studies are performed each year trying to improve these properties, some studies concentrated on incorporating different types of nano oxide particles into the silicone matrix.
Aim of the study: The aim of this study was to investigate the effect of adding different concentrations of titanium silicate nano particles into silicone matrix on tear strength, tensile strength and hardness.
Materials and method: Depending on the results of a pilot study, 0.5% and 1% weight concentrations of titanium silicate nano filler were selected, as they had the most improvement in properties of the silicone material. The manufacturer's instructions were followed in mixing and curing of the maxillofacial silicone material, and 90 specimens were prepared, the samples were divided into 3 groups according to the tests (tear strength, tensile strength and hardness), each group contains 30 samples, the groups were subdivided into three subgroups (A, B and C); group A is the control group with 0% of nano filler, while both B and C groups being experimental groups with 0.5% and 1% of nano filler respectively. The collected results of the study were analyzed statistically by using analysis of variance (one way ANOVA) and Post hoc tests.
Results: For tear strength and tensile strength both experimental groups (0.5% and 1%) showed a highly significant increase in values compared to control groups, with the highest mean value being noticed in 0.5% group. While in shore A hardness test both experimental groups showed a highly significant increase in hardness compared to control group, with the highest mean value being noticed in 1% group.
Conclusion: The addition of 0.5% concentration of titanium silicate nano particles into silicone elastomer enhanced some of the material properties with a slight increase in hardness.

Key words: Silicone, Maxillofacial prosthesis, VST-50, Titanium silicate, Nano filler

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INTRODUCTION

Defects in head and neck area are mainly caused by congenital malformations, neoplasia and injuries; these defects prevent the patient from having a normal life in both physiologic and psychologic means [1].

Reconstructive surgery is considered the first line of treatment in these cases, but in many situations it may not be possible, due to unfavorable conditions like the size of the defect or the medical condition of the patient, so the construction of a maxillofacial prosthesis becomes mandatory [2,3].

Multiple materials were used through the history to fabricate maxillofacial prosthesis, but the development of silicone materials was a great invention in the field of maxillofacial prosthetics because of its durability, ease of fabrication, chemical stability and biocompatibility [4], on the other hand, mechanical properties of silicone elastomers are far from ideal, some of these properties, like low tensile and tear strength and short service life, need to be improved. The improvement can be achieved by adding fillers, pigments and other additives, which will create an elastic material with enhanced physical and mechanical properties, this material will be more practical in clinical use of maxillofacial prosthesis [5,6].

In the last few decades, scientists were trying to develop their industrial methods to incorporate fillers into polymeric matrix, creating a new generation of elastomers that combine the flexibility of silicone and the strength of fillers [7]. The variety of the achieved improvements is mainly related to polymer properties, characteristics of added filler (like size, surface area,

structure and activity of filler surface), amount of added filler and processing conditions [8].

Nano oxide fillers were widely used in recent researches as additives to silicone elastomers, as they are more rigid than the polymeric matrix and have a greater shear modulus, multiple studies found some improvement in mechanical properties of the polymeric matrix, this improvement may be attributed to the high surface area and high surface energy of the nano particles, making them more reactive and allowing them to be incorporated into the backbone of the polymer [9,10].

Titanium silicate ($TiSiO_4$) nano particles are one of the mixed metal oxides nano particles, which have a wide range of applications in electronic industry, glass and optical devices, ceramics and other composite applications. The properties of titanium silicate material are size dependent, so their chemical and physical properties are unique compared to the bulk material, but as a general guideline the smaller particles will have a greater surface energy allowing a better incorporation into the polymeric matrix [11]. Considering all the previous reasons, titanium silicate nano particles (<100 nm) were selected in this study to be added to room temperature vulcanized (RTV) silicone as reinforcing filler, in order to investigate its effect on some properties.

MATERIALS AND METHODS

Titanium silicate nano particles (Nanoshel Inc., Willmington, DE, USA) were incorporated into room temperature vulcanized silicone VST-50 (FactorII Inc. Lakeside, USA). Ninety samples were prepared and divided into three groups according to the conducted test with 30 samples for each test. Each group was further sub-divided into 3 sub-groups (A, B and C), each sub-group contains 10 samples. Group A with 0% of filler (control group), groups B and C are experimental groups with nano filler added in 0.5 wt% and 1 wt% concentration respectively.

Pilot study

A pilot study was done to determine the most suitable concentrations of titanium silicate nano filler to be added to the maxillofacial silicone by testing the effect of addition on tear strength and hardness, the pilot study revealed that 0.5 wt% and 1 wt% were the most suitable concentrations.

Mold preparation

Computer software (Auto CAD 2015 ((Autodesk Inc., San Rafael, CA, USA)) was first used to design the mold; followed by a laser engraving cutting machine that was used to cut the molds parts (Figure 1). Acrylic sheets of different thicknesses 2 ± 0.05 mm and 6 ± 0.05 mm were used to obtain the appropriate dimensions of the molds depending on the specifications required for each test to be performed [12].

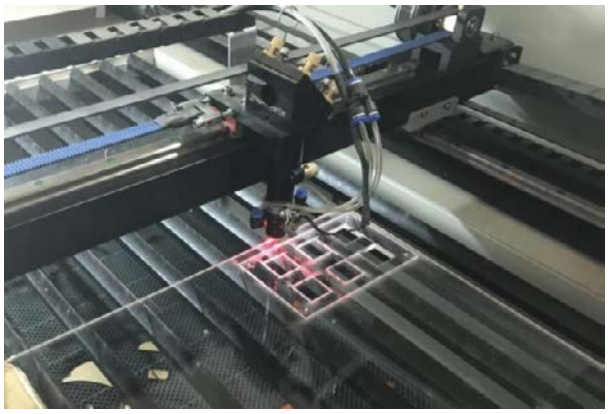

Figure 1: The process of acrylic mold preparation by using Laser engraving cutting machine

Incorporating nano titanium silicate filler into silicone base (part A)

The required concentration of the nanoparticles were accurately weighed in the mixing bowel using electronic balance (0.000 digits) and then part A of the silicone material was added to it and weighed properly, after that both part A and the nanoparticles were mixed together for 10 minutes using the vacuum mixer; to prevent nanoparticles suction; the vacuum part was switched off for the first three minutes and then switched on for the remained seven minutes [6].

Addition of silicone catalyst (part B)

As mixing process generates heat; the obtained mixture was then left to cool down at room temperature before adding part B to prevent the inevitable reduction in the working time that could occur if part B was added to part A before it cooled down. So part B was then weighed (According to the manufacturer's instructions) and added to the mixture and mixed for additional 5 minutes using the vacuum mixer [5].

Sample fabrication

The cover acrylic sheet of the mold was coated with two layers of alginate solution (separating medium) and left over the counter to dry, after that the silicone mixture was then added slowly and carefully using a metal spatula until it filled all samples spaces inside the matrix; to prevent insufficiency, sample spaces was slightly overfilled (Figure 2). The cover part was then placed over the matrix part by applying a moderate continuous hand pressure over the center of the cover until being tightened by screws and nuts at the corners and secured with the G-clamps all around the mold borders. All air bubbles and excess silicone material were supposed to disappear after mold closure and to drain out from the mold borders by the pressure forces that were applied by screws and G clamps (Figure 3); when not; the air bubbles contained sample was excluded [6].

Figure 2: Filling mold with silicone material

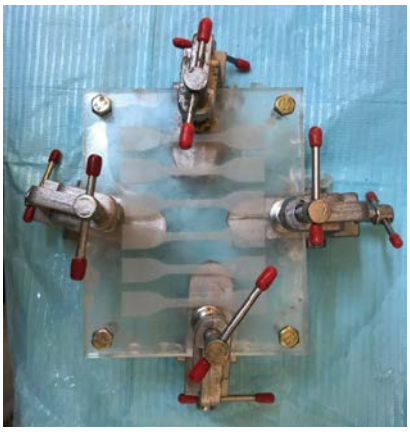

Figure 3: Closing the cover of the mold and tightening screws and G clamps

The silicone material was left to undergo the polymerization process for 24 hours at room temperature (23°C ± 2°C), after that the mold was opened and the samples were carefully removed [10]. To remove the remaining residual separating medium; samples were carefully washed under tap water, dried using paper towel and finished using scalpel and no. 11 blade to remove all excess material [13].

Storage of samples

VST-50 silicone sets after 24 hours according to product descriptions, after that the samples were removed carefully from the molds. All samples were stored inside a cooling box (same box used for vaccine storage). The cooling box was constructed from a tightly closed cover that secured all around from the inner side by a sealing ring. The inner part of the box was lined with insulating foil that had a special six foil pockets in the side walls; holding six gel-type ice containers. This insulating system play important role in controlling the external condition by keeping the humidity and temperature within acceptable values for the longest time possible. An internal plug was used to close a hole crossing the entire box cover thickness. A special sensor was passed through this hole to evaluate the values of temperature and humidity when the box is closed. These values were presented on a digital device screen (digital indoor and outdoor thermometer with a hygrometer) [13,14].

As directed by the manufacturer; silicone samples are better to be stored at (50% ± 10%) RH and (23°C ± 2°C) temperature. This temperature was achieved by either cooling or warming the ice containers. While the favorable RH was achieved by placing a disposable cup containing wet cotton inside the box in order to increase the RH value; on the other hand; silica gel was used to decrease the RH value as it was considered as a material which has a drying ability [6].

Procedures for mechanical tests

Tear strength: An angle un-nicked test sample was used according to the specifications of ISO 34-1:2015 [15]; the sample has one apex (90° angle) and two tab ends with a sample thickness that measure 2 ± 0.2 mm . The un-nicked angle type of sample is utilized for the measurement of tear initiation and propagation; the stress was accumulated at the angle point until tear was initiated; followed by furthers stresses which was responsible for the tear propagation.

The following equation was used to calculate the tear strength (N/mm):

$$\text{Tear strength} = F/d \quad (1)$$

where, F is the maximum force in Newtons; d is the sample thickness in millimeters.

Tensile strength: The samples were fabricated as type 2 dump-bell shaped samples according to the specifications of ISO 37:2017 [16]. The ultimate tensile strength (in MPa) was calculated according to the following equation:

$$\text{Tensile strength} = Fm/Wt \quad (2)$$

where, Fm is the maximum force in Newtons; W is the width of the narrow part of sample in millimeters; t is the thickness of the sample over the narrow part in millimeters.

Hardness: Test samples with dimensions of 40 mm × 40 mm × 6 mm length, width and thickness respectively was fabricated to conduct Shore A hardness test in accordance to ISO 7619-1:2010 [17] specifications. The outer surface area of the dimension should be sufficient to allow for five measurements with 6 mm distance separates each measurement from the other one and 12 mm distance from the margin of the sample. Sample was placed over a flat firm surface, the durometer was held in perpendicular way over the sample surface with pressure foot parallel to the surface (Figure 4). The durometer was firmly pressed for one second at each one of the 5 marked points [10].

Figure 4: Shore A durometer used to measure hardness of silicone sample

Statistical analysis

The data of this study was analyzed using SPSS (statistical package for social sciences) software, one-way ANOVA (Analysis of variance) was used to compare mean values of tested groups. Post hoc test (Tukey HSD) was used to determine the significance of difference between each two tested groups.

A probability (P) value of >0.05 was considered statistically non-significant (NS), while P ≤ 0.05 was considered statistically significant (S), and P ≤ 0.01 was considered as highly significant (HS).

RESULTS

Tear strength test results

Both experimental groups B and C showed a higher mean value of tear strength than control group A, experimental group B showed the highest mean value (27.92 N/mm) among all other groups (Figure 5). One-way ANOVA showed a highly significant difference among groups (Table 1).

In order to compare mean value among study groups, Tukey Honestly Significant Difference (Tukey HSD) test was conducted. There was a highly significant difference between group (A) and group (B) as well as between group (A) and group (C), while there was no significant difference between group (B) and group (C) (Table 2).

Table 1: Statistical test of tear strength among groups using one-way ANOVA

	Sum of Squares	Df	Mean Square	F	P	Sig.
Between Groups	112.335	2	w56.167	16.993	0.362	.000 HS
Within Groups	89.243	27	3.305	-	-	-
Total	201.578	29	-	-	-	-

Table 2: Multiple comparisons of tear strength between groups using Tukey HSD test

(I) groups	(J) groups	Mean Difference (I-J)	Sig.
Group (A)	Group (B)	-4.632	.000 HS
	Group (C)	-3.187	.002 HS
Group (B)	Group (C)	1.445	.196 NS

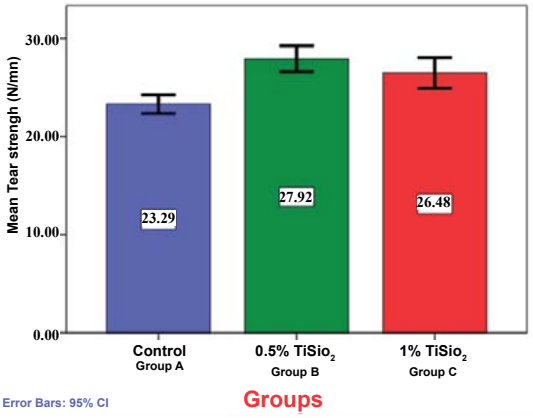

Figure 5: Bar chart representation of mean value and standard deviation of tear strength test of all study groups

Tensile strength test results

Both experimental groups B and C showed higher mean values than control group A, the mean value of group B (8.01 MPa) was the highest among all the other groups (Figure 6). One-way ANOVA showed a highly significant difference among groups (Table 3).

In order to compare mean value among study groups, Tukey HSD test was conducted. There was a highly significant difference between group (A) and group (B) as well as between group (A) and group (C), while there was no significant difference between group (B) and group (C), (Table 4).

Table 3: Statistical test of Tensile strength among groups using one-way ANOVA

	Sum of Squares	Df	Mean Square	F	P	Sig.
Between Groups	5.135	2	2.568	15.16	0.426	.000 HS
Within Groups	4.573	27	0.169	-	-	-
Total	9.708	29	-	-	-	-

Table 4: Multiple comparisons of tensile strength between groups using Tukey HSD test

(I) groups	(J) groups	Mean Difference (I-J)	Sig.
Group (A)	Group (B)	-1.003	.000 HS
	Group (C)	-0.627	.006 HS
Group (B)	Group (C)	0.376	.121 NS

Hardness test results

Experimental group C showed the highest mean value (38.89), followed by experimental group B (37.11), while control group A showed the lowest mean value (34.89),

as shown in (Figure 7). One-way ANOVA showed a highly significant difference among groups, (Table 5).

Tukey HSD test was conducted to compare mean value between the study groups, the test revealed a highly significant differences between all study groups, (Table 6).

Table 5: Statistical test of hardness among groups using one-way ANOVA

	Sum of Squares	Df	Mean Square	F	P	Sig.
Between Groups	80.323	2	40.161	26.264	0.259	.000 HS
Within Groups	41.287	27	1.529	-	-	-
Total	121.61	29	-	-	-	-

Table 6: Multiple comparisons of hardness test between groups using Tukey HSD test

(I) groups	(J) groups	Mean Difference (I-J)	Sig.
Group (A)	Group (B)	-2.22	.001 HS
	Group (C)	-4	.000 HS
Group (B)	Group (C)	-1.78	.009 HS

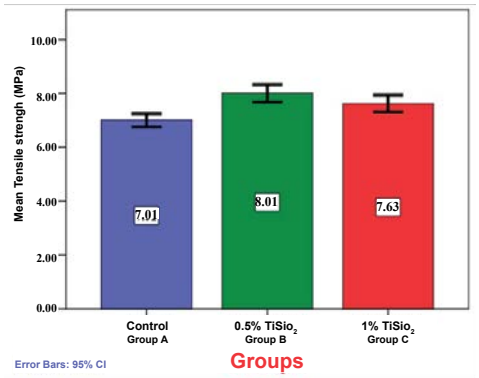

Figure 6: Bar chart representation of mean value and standard deviation of tensile strength test of all study groups

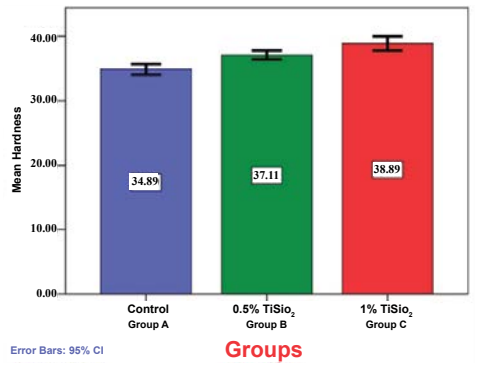

Figure 7: Bar chart representation of mean value and standard deviation of hardness test of all study groups

DISCUSSION

In general, maxillofacial silicone materials have a long list of physical, mechanical, esthetic, biologic and processing properties. Among this list are the most basic properties which must be available in the ideal maxillofacial silicone material such as high tear strength, high tensile strength, and adequate hardness to permit the flexibility of the material [18]. The results of many studies suggested that no commercially available material had fulfilled all of these ideal properties; this explains the continuously growing number of researches aimed to find a better maxillofacial silicone material, either by altering the formulas of the previous materials or by adding different types of fillers in different concentrations [19].

The results of tear strength test indicate a highly significant increase after the addition of nano titanium silicate in both concentrations (0.5 wt% and 1 wt%) compared to control group, this increase will be explained by the trapped networks that can be formed as a result of the ability of the nano particles to aggregate in three dimensional meshes of fillers inside the polymer matrix and trap some chains of polymers. As a result; a marked increase in polymer stiffness and tearing resistance will be unavoidable [20,21].

In general, rubber has high tear strength; this is mainly due to their ability to disseminate strain energy close to the beginning of growing tear. As the tear propagates; nanoparticles will disseminate their energy inside the polymer matrix this will increase the matrix tear resistance and subsequently increase the load required to break the matrix completely [22].

When the concentration of nano particles is raised from 0.5 wt% to 1 wt%, a slight non-significant decrease in tear strength test results is noticed, this decrease can be explained by the fact that when nano filler concentration is increased; the filler starts to agglomerate which will result in a lower mechanical properties like tear strength [9,10].

The results of tensile strength test indicate a highly significant increase in tensile strength after the addition of nano titanium silicate in both concentrations (0.5 wt% and 1 wt%) when compared to control group, the increase may be attributed to the fact that polymer chains and the incorporated filler particles, when subjected to tensile forces, will slide over each other. The presence of nano filler will aid in preventing polymer chains breakage [23].

Also, if the polymeric matrix is capable of converting the affected force into heat, then there will be fewer forces available to destruct the polymeric chains [23]. Filler incorporation is a good factor in disseminating those forces [24].

Increasing the concentration of titanium silicate from 0.5 wt% to 1 wt% resulted in a non-significant decrease in tensile strength, this can be attributed to the fact that filler content should be kept under proper level, because nano fillers have a high surface energy and good chemical

reactivity, so any increase in nano oxide concentration will lead to agglomeration. When silicone is subjected to forces, the agglomerated particles will be the center of stress concentration, leading to faster breakage and as a result a decrease in mechanical strength [9].

The results of shore A hardness test indicate that the hardness increases after the addition of titanium silicate nano filler, in 0.5 wt% and 1 wt% concentrations. And the increase was gradual as the filler concentration rises. When the concentration of nano filler increases, it will result in filler to filler binding, which will fill the inter-aggregates spaces between polymer chains and making them smaller and smaller as the filler loading increases, leading to more rigid polymer that will be highly resistant to indentation and penetration [25].

CONCLUSION

Within the limitations of the present study, it was concluded that the addition of different concentrations of titanium silicate nano particles into VST-50 maxillofacial silicone elastomer enhanced some of the material properties (tensile strength and tear strength) but on other hand slightly increased hardness. The most optimum enhancement was obtained at 0.5 wt% concentration.

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