

# Evaluation of Tensile Strength and Percentage of Elongation for Pigmented and Non-Pigmented Maxillofacial Silicone Before and After Aging

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**Abstract:** Background: Silicone advantageous properties made it to become the material of choice in construction of maxillofacial prosthesis but its properties may change after addition of pigments and usage. This study aimed to define the optimum concentration of 2 types of intrinsic pigments that added to VST-50 silicone and study their effects on tensile strength and percentage of elongation before and after aging. Materials and methods: According to the pilot study, 0.1% of rayon flocking and 0.2% of burnt sienna intrinsic pigments concentrations were selected because of improvement in tested mechanical properties of VST-50 silicone. Sixty samples were prepared in order to measure (tensile strength and percentage of elongation) before and after aging and divided into 6 subgroups with 10 samples made for each one of these sub groups. After that the samples are tested before and after (75 and 150 hrs.) of aging and the data were analyzed with a descriptive statistical analysis, student t-test and one -way ANOVA test. Results: The results for pigmented samples before aging show that tensile strength had improved while percentage of elongation was decreased. After (75 and 150) hours of aging, percentage of elongation highly significantly decreased while tensile strength had highly significantly decreased. Conclusion: The addition of intrinsic pigments had improved tensile strength of maxillofacial silicone which was on the reverse for percentage of elongation. After subjecting the maxillofacial silicone to aging, both had been adversely affected.

**Keywords:** aging, tensile strength, intrinsic pigments, maxillofacial silicone, percentage on elongation

## 1. Introduction

When maxillofacial defects cannot be surgically fulfilled, maxillofacial prostheses are constructed by maxillofacial prosthetists and technologists (MPTs), as an alternative treatment<sup>[1]</sup>. Silicone elastomer is the material of choice, because it offers biological, physical and mechanical properties better than other materials<sup>[2]</sup>.

One of the most important factors relative to clinical success of maxillofacial prosthesis is its ability to reproduce color, texture, form and translucence of surrounding tissue making it inconspicuous to any observers. To achieve this, it is necessary to add colors to silicone, as they are mostly available in transparent form. Coloration techniques are basically divided into two groups: intrinsic or extrinsic<sup>[3]</sup>. Extrinsic coloring can change the look of the prosthesis only to some extent that's why the basic shade of the prosthesis must be achieved by intrinsic coloration<sup>[4]</sup>. The intrinsic pigments cannot be rubbed off easily since they cure with the silicone<sup>[3]</sup>.

Tensile strength and percentage of elongation are among the mechanical properties that are important for the success of prosthesis<sup>[5]</sup>.

Maxillofacial materials are not perfect materials. Many of them are subjected to discoloration, deterioration of properties, difficulty in repair process, and short service lifetime of the prosthesis, considering the patient's habits, the climate, and the environment<sup>[6]</sup>. Weather, environmental factors, smoking, chewing tobacco, and household cleaning

agents such as solvents, bleaches, detergents, and scouring powders all can degrade maxillofacial materials<sup>[7]</sup>.

The amount of sunlight, the average temperature, the moisture level to which the prosthesis is exposed, and the presence of dust and pollutants in the air are considered the main components of weather that affect the prosthesis<sup>[8]</sup>. Artificial weathering is often used to approximate outdoor performance and predict the average lifetime of polymers. Because the effect of weather varies considerably by location, season, time of day, amount of cloud, and orientation at which the specimens are exposed<sup>[9]</sup>.

The artificial accelerated weathering extensively applied to produce the same normal life weathering condition for testing the overall deterioration of the prosthesis material and produce the long term effect of outdoor weathering condition but in accelerated way, that was achieved by using aggressive component of the weathering condition UV, heat, moisture and water spray.<sup>[10]</sup> The average service-life of the maxillofacial prosthesis from 4-14 months<sup>[11]-[12]</sup>.

## 2. Materials and Methods

### Sample fabrication

According to the conducted tests (tensile strength and elongation percentage tests), 60 samples were made to measure the mechanical properties before and after weathering. It includes 6 subgroups with 10 samples made for each one of these sub groups. These sub groups represent the following:

- 1) Control samples without weathering: (A, non-pigmented and B, pigmented samples).
- 2) After 75 hrs. of weathering: (C, non-pigmented and D, pigmented) samples.
- 3) After 150 hrs. of weathering: (E, non-pigmented and F, pigmented) samples.

The dimensions of samples were designed using Auto CAD 2013 then processed using the CNC machine. Clear acrylic sheets were used in fabrication of molds.

The two pigments and the platinum RTV (Room-Temperature-Vulcanized) VST 50, platinum catalyzed, vinyl terminated RTV silicone, obtained from Factor II Inc.,(Lakeside, USA ). The mixing ratio for the silicone is 10:1 base (part A) to cross linker (part B) by weight according to manufacturer's instruction (Figure 1).



**Figure 1:** A: Rayon flocking (tan color), B: Burnt sienna (F1) pigment, C: VST 50 silicone base, D: VST 50 silicone catalyst

In this study two groups of samples were fabricated. The maximum amount of silicone to be mixed each time was 200 g.. The first group was the non-pigmented group, which is fabricated by mixing 200 g of silicone base and 20 g of silicone catalyst without adding any pigments. The second group was the pigmented group which is fabricated by mixing of 199.40 g of base, 19.94 of catalyst and adding 0.2g of rayon flocking with 0.4 g of burnt sienna. These pigments concentration were chosen according to previous pilot study. For each of the experimental groups, the pigment was placed first in a clean plastic beaker. The rayon flocking was added by a clean metal spatula while the burnt sienna was added as drops from the pigment dropper-type bottle after shaking of it [13]. The rayon flocking was easily spread through the air, thus the addition of silicone over it prevented it from spreading. To fabricate the specimen, a vacuum mixer multivac 3 (Degussa, Germany) was used, mixing is performed for 7 minutes at speed 360 rpm under vacuum - 10bar (Figure 2)<sup>[14]</sup>.



**Figure 2:** Degussa Multivac vacuum machine. A: Vacuum meter, B: Multivac machine, C: Mixing time gauge.

For better incorporation of pigments into silicone, pigments was mixed with the base for 2 minutes by mechanical mixer without vacuum followed by 5 minutes  $\pm$  5 seconds mixing under vacuum by the mechanical mixer according to the pilot study and other studies<sup>[15]-[16]</sup>. The catalyst drops were dispensed in different places and not in the center only, to aid in gaining homogeneity rapidly while the glass beaker was on the digital scale.<sup>[15]-[16]</sup> The mixture mixed under vacuum for 5 minute<sup>[14]</sup>. Pouring of the material was done at a controlled temperature of  $(23\pm 2^\circ\text{C})$  and  $50\pm 10\%$  RH as recommended by the manufacturer and standards<sup>[19]</sup>. Before closing the mold, the mold cover was coated with petroleum jelly and hex nuts are prepared and screws are inserted in each hole at the corner of the mold. Then silicone mixture is poured gradually. When pouring was finished, the air bubbles were removed by using fine needle. When all the air bubbles were broken-up, the mold cover, which was previously coated with petroleum jelly using tissue papers, was laid onto the matrix<sup>[20]-[21]</sup>. After that, the mass of 1 kg was applied on the center and the cover was tightened by screws and nuts at each corner of mold then four (4) G-clamps was added at the middle of each side.

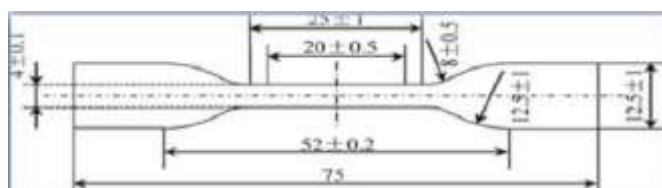
Mold stored away from light at  $23^\circ\text{C} \pm 1$  for 24 hours to complete vulcanization of RTV silicone. All samples were removed carefully from their molds after complete set of the silicone. Care was taken not to strain samples during demolding procedure. The flash was removed with a scalpel and sharp surgical blade # 10<sup>[21]</sup>.

### Mechanical properties testing

Two tests were used to evaluate mechanical properties of pigmented and non-pigmented VST-50 silicone elastomer before and after artificial weathering which were percentage of elongation and tensile strength tests. The tests were done at optimum temperature of  $23^\circ\text{C} \pm 2$ . The minimum time of testing should be 16 hours after vulcanization.<sup>19</sup> The samples were stored in air free bag and light proof box to avoid change in the properties of samples<sup>[22]</sup>.

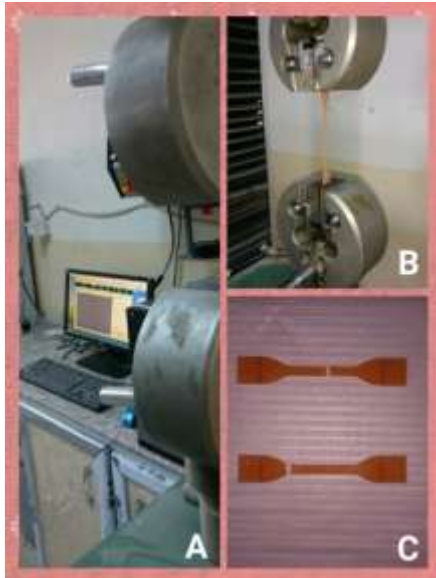
### Tensile strength test

Test was performed according to ISO 37 (2011) and dumbbell-shaped samples (type 2) were prepared (Figure 3).<sup>[23]</sup>



**Figure 3:** Tensile strength type 2 sample dimensions in mm (adopted from ISO 37, 2011).

The thickness measurements were made at each end and at the center of the test length of the sample, using a digital caliper and the median value of three measurements was taken. Tensile strength testing was conducted using a computer controlled universal testing machine. The sample was clamped by grips of the machine to distribute the tension equally over the cross-section. The speed of stretching of sample was 500 mm/min and the maximum stretching force at break was recorded by computer software (Figure 4)<sup>[23]</sup>.



**Figure 4:** A, B, Tensile sample on instron machine. C, Two measured tensile samples; the upper one should be included in the study, the lower one should be discarded.

According to ISO 37 (2011), the ultimate tensile was calculated from the maximum stretching force at break divided by the original cross sectional area of the sample (width × thickness of the narrow portion) using the following formula:

$$T_s = F/W \times T$$

Where:

F=maximum force in Newton

W= width of narrow portion of the sample in millimeters

T=thickness of narrow portion of the sample in millimeters.

#### Elongation percentage test

In accordance with ISO 37 (2011), elongation percentage was calculated at the time of measuring tensile strength (before failure)<sup>[23]</sup>.

The original length was measured before testing using the digital caliper by placing two benchmarks on the narrow part of the dumbbell-shaped sample using the thin-line permanent marker. The marks were 20±0.5 mm apart, equidistant from the center and perpendicular to its long axis.

The break elongation was measured from the original length of tensile sample and the length of the sample at break using the equation:

$$\text{Elongation percentage at break} = 100(L_b - L^\circ) / L^\circ$$

Where:

L<sup>°</sup>: The original length in mm.

L<sub>b</sub>: Extension at break in mm.

#### Artificial accelerated weathering- using weather-Ometer Device

Weather-Ometer device (QUV) promote the same condition of outdoor weathering but in accelerated way, so many hours in device chamber equivalent to many days in outdoor weathering according to the site of study. Samples were arranged in the machine's rulers and then the rulers were inserted in the QUV machine.

The weathering standardization used in this study is ASTM (G 154) which is the most popular accelerated weathering.<sup>(24)</sup> According to cycle 7 of ASTM G 154 the samples exposed to 25 cycles of 340 ultra-violet light (8 hour) at a temperature of 60°C followed by 4 hours (spray for 15sec.), and condensation (dew) for 3:45 minutes at a temperature of 50°C. Artificial weathering, accompanied with UV irradiation 1.55 W/m<sup>2</sup>/nm and water spray<sup>[25]</sup>.

The samples were exposed to artificial weathering, then tested after (75) and (150) hours and compared with the samples that were tested before artificial weathering. There was no exact number of hours in weather-Ometer chamber equivalent to one year of outdoor weathering or one year of clinical use of maxillofacial prosthesis, because that depends on many factors, related to weather condition, site of study and type of material being used, but there was some important facts can be depended i.e. the yearly cumulative global radiation mean for Baghdad city that equivalent to 216 MJ/m<sup>2</sup> per year<sup>[26]</sup>.

The irradiance that controlled at narrow wavelength range in Weather-Ometer device according to cycle 7 of G 154 standardization, that is equivalent to 1.55 W/m<sup>2</sup> at 340nm, then used the following equation: KJ/m<sup>2</sup> = W/m<sup>2</sup> × 3.6 × Hours.<sup>(25)</sup> According to this information, the one year of outdoor weathering equivalent nearly 387 hours of artificial accelerating weathering according to the Baghdad city, 75 hours of accelerated weathering equivalent nearly 70.73 days, and that means 2.35 months in outdoor weathering and 150 hours of accelerated weathering equivalent nearly 141.47 days, and that means 4.71 months in outdoor weathering.

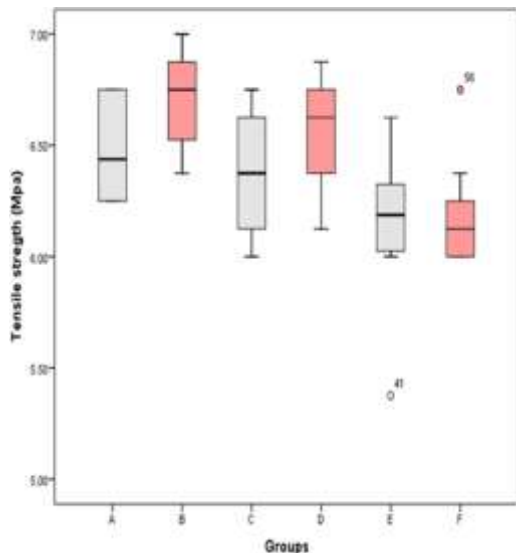
#### Statistical analysis

The study data were analyzed by descriptive statistics (Mean, Standard deviation, Box plots) and inferential statistics (Student t-test and One- way ANOVA) was used to compare mean value of all groups. A highly significant value was considered when (P) value ≤ 0.01, while P ≤ 0.05 was considered significant, and (p) value > 0.05 was considered statistically as non-significant.

### 3. Results

#### Tensile strength test

The highest mean value of tensile strength test appeared in group B (6.710 MPa), followed by group D (6.550 Mpa), group A (6.488 MPa), group C (6.350 MPa), group F (6.188 MPa) while group E mean value was the lowest among the groups (6.160 MPa) (Figure 5 and Table 1).



**Figure 5:** Box plot presentation of tensile strength mean values of all study groups in MPa.

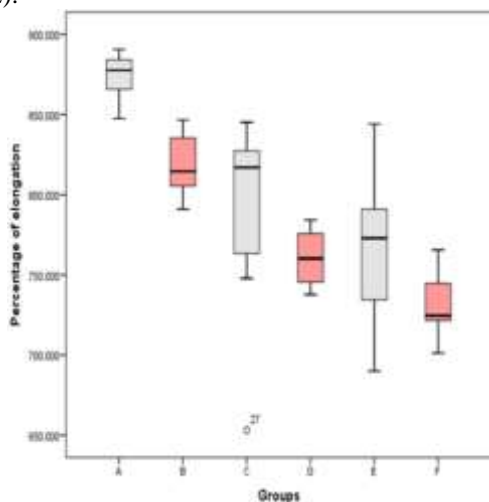
Table 1 show descriptive statistics for tensile strength values and significant difference between two independent means using Students-t-test at 0.05 levels. P values showed non-significant difference between all groups.

**Table 1:** Descriptive statistics for tensile strength test results in MPa

	Tensile (MPa)		
	Mean	SD	t- test P value
A	6.488	0.216	0.062
B	6.710	0.230	
C	6.350	0.275	0.093
D	6.550	0.251	
E	6.160	0.340	0.815
F	6.188	0.238	
ANOVA	0.0001# (HS)		

**Elongation percentage test**

The highest mean value of elongation percentage test appeared in group A (874.515 %), followed by group B (817.656 %), group C (789.279 %), group E (767.068 %), group D (760.207 %), while the mean value of group F (713.92 %) was the lowest among the groups (Figure 6 and Table 2).



**Figure 6:** Box plot presentation of elongation percentage (%) test mean values of all study groups

Table 2 show descriptive statistics for percentage of elongation values and significant difference between two independent means using Students-t-test at 0.05 levels. P values showed high significant difference between group A and B. while between C and D was non-significant. Finally between E and F groups were significant.

**Table 2.** Descriptive statistics for percentage of elongation test results in (%).

	Percentage of elongation (%)		
	Mean	SD	t- test P value
A	874.515	14.243	0.0001*
B	817.565	18.594	
C	789.279	58.538	0.056
D	760.207	16.536	
E	767.068	45.081	0.022*
F	731.920	18.889	
ANOVA	0.0001# (HS)		

**4. Discussion**

Clinicians should treat the manufacturers' values with caution when choosing a material to fabricate an extra-oral maxillofacial prosthesis because it's not representative of how silicone elastomers are used for extra-oral maxillofacial prostheses. Therefore, manufacturers should report the mechanical value of their silicone material with pigment additives<sup>[15]</sup>.

Rayon flocking and functional intrinsic pigment by Factor II Inc. were selected because they were the top two commonly used intrinsic pigments by maxillofacial prosthodontists<sup>[27]</sup>. The service life and mechanical properties of maxillofacial silicone prosthesis was significantly weakened by artificial accelerated weathering<sup>[28]</sup>.

The artificial accelerated weathering was selected in this study because the changes produced are greater than outdoor weathering or normal prosthesis use<sup>[6]</sup>. In this study, the effects of incorporation of intrinsic pigments into VST-50 silicone on some mechanical properties before and after weathering were evaluated. The proper concentration of each pigment was selected previously in the pilot study.

**Tensile strength**

Tensile strength indicates the maximum stress to which a material can be subjected before it begins to fail by localized accelerated deformation<sup>[29]</sup>. It is an important property of maxillofacial silicone in service because when the patient removes the prosthesis, high tensile forces are applied, particularly in thin areas<sup>[30]</sup>. The result of tensile strength test showed non-significant increase in the mean value of tensile strength when both rayon flocking and burnt sienna(FI) were added as compared with the control group (Figure 5 and Table 1).

The increase in tensile strength mean value after rayon flocking incorporation may be due to stress transfer from the weaker resin matrix to the much stronger fibers and the effective restraining of the matrix by fibers, which led to hindering of the growing crack<sup>[31]</sup>.

Another explanation for the increase of tensile strength mean value when rayon flocking was incorporated, the tensile strength was increased because the space between fibers decreased with fiber aspect ratio and content. It is found that short-fiber incorporation accelerate the crystallizing behavior of elastomers and this strain-induced crystallization will improve the tensile property<sup>[32]</sup>.

Results are in agreement with Haug et al. (1999) who found that there was a non-significant increase in tensile strength with the addition of flocking to Silicone A-2186<sup>[11]</sup>.

On the other hand, the results disagree with Haug et al. (1999) because the tensile strength of Medical adhesive A silicone was significantly decreased by the addition of liquid pigment<sup>[11]</sup>. This may be due to differences in manufactured pigment within a company, types of silicones or techniques, as the stretching speeds of jaws of the universal testing machines were different.

The results of tensile strength in (Figure 5 and Table 1) show a high significant decrease in tensile values with artificial accelerated weathering after 75 and 150 hours when compared with the samples before artificial weathering.

Tensile strength of vulcanized maxillofacial silicone mainly depends on the cross-linking system, cross-linking density and interaction between filler and polymer chain<sup>[33]</sup>.

The reduction in tensile strength with artificial accelerating weathering (75-150 hours) could be explained in two ways, the first one, with the time and accelerated UV light, the cross-linking of polymers will continue and increase, and associated with more volatile decomposing from catalyst, that produced denser elastomer. Silicone when exposed to high energy of irradiation undergo cross linking that cause degradation of silicone physical and mechanical properties and became brittle and inelastic and easily deformed with lower forces<sup>[20]</sup>.

The elasticity and strength of maxillofacial silicone are greatly dependent on the degree of cross-linking and the molecular weight of polymers<sup>[32]</sup>.

The second one, decrement in tensile strength may be due to photo oxidative effect of artificial weathering, with free radical formation and their reaction with each other led to further cross linking, which leads to polymer structural modification and changing in the molecular weight distribution and producing of volatile degradation products<sup>[34]</sup>.

The result of this study was in agreement with Eleni et al., 2009 who found significant decrease in the tensile strength of Cosmosil M511 and CPE (Experimental Chlorinated Polyethylene) when exposed to accelerated weathering for 216 hours<sup>[35]</sup>. The results were in disagreement with Al-Harbi et al., 2015 who found that the tensile strength is not affected by outdoor weathering on A-2186 maxillofacial silicone this may be due to different filler and different weathering environment<sup>[36]</sup>.

### Percentage of elongation

Elongation is useful in restoring defects of the head and neck with deeper undercuts and it's an indication of how far the material stretches before it breaks. In these conditions, high elongation is desirable as it makes the material to withstand larger deformation without breaking.<sup>[37]</sup>

The results of elongation percentage showed that pigments highly significantly decreased the elongation percentage mean value when compared with the control group before weathering (Figure 6 and Table 2).

The decrease in elongation percentage mean value after addition of rayon flocking may be due to that the fibers makes the matrix more restrained due to inhibition of molecular chains flow and thus result in substantially less elongation percentage values.<sup>[31]-[38]</sup>

This decrease could also be explained by the decrease in the space between fibers (fiber aspect ratio and content), therefore the elongation percentage was decreased due to reduced distance between silicone molecules.<sup>[32]</sup>

While the decrease in mean value after adding burnt sienna (FI), is due to the gradual increase of resulting in higher crystallization rates facilitated by the presence of plasticizer. The plasticizer, which is a cross-linking fluid into which the pigment is suspended, could increase the rate of cross-linking. The reduction of elongation percentage with the increased rate of cross-linking can also be explained for the same reason as in tensile strength.<sup>[39]</sup>

Results agree with Lai et al. (2002) who added 0.05 wt.% rayon flocking to MF-606 silicone and reported a non-significant decrease in elongation as compared with control group.<sup>[40]</sup>

Results are in disagreement with Haug et al. (1999) who found that percentage elongation increased after the addition of artist's oil to medical adhesive type A by 26.6% and silastic 4-4210 by 24%. This may be due to differences in pigment and tested types of silicones.<sup>[41]</sup>

The results of elongation percentage in (Figure 6 and Table ) show a high significant decrease in percentage of elongation values with artificial accelerated weathering after 75 and 150 hours when compared with the samples before artificial weathering. The decrease in elongation percentage values after weathering could be as a result of enhanced chemical reaction (photo-oxidation) that will affect the molecular weight distribution and decrease the density of the structural network.<sup>[42]</sup> Moreover, the elevated temperature increases the stiffness of the polymers.<sup>[43]</sup>

Others believes that elastomer became denser after accelerated UV light aging as a result of volatile decomposing from catalyst; however, high levels of cross linking may yield brittle, inelastic and easily deformed material even when applying lower forces.<sup>[17]</sup>

The results were in agreement with Nguyen et al. (2013), Al-Harbi et al. (2015), Zardawi et al. (2015)<sup>[15]-[36]-[44]</sup>.

While disagreements were found with Hatamleh et al. (2011) the disagreement of the present study with all previously mentioned studies may be due to different types of pigments or maxillofacial silicone, and different types of aging conditions and durations<sup>[17]</sup>.

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