2018

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Studying some mechanical properties of maxillofacial silicone elastomer before and after incorporation of intrinsic pigments and artificial aging

Noor M. Shihab, Faiza M. Abdul-Ameer*

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1. Introduction

Maxillofacial prostheses are constructed as an alternative treatment when maxillofacial defects cannot be surgically corrected [1]. Silicone elastomer is commonly used because it offers excellent biological, physical, and mechanical properties [2]. Color reproduction, texture, form, and translucency are considered the most important factors affecting the clinical success of maxillofacial prosthesis. To achieve these factors, adding colors to silicone is necessary using intrinsic or extrinsic techniques [3]. The intrinsic pigment cannot be rubbed off because they are added before curing to the silicone base. Although the extrinsic coloring allows for changing of the look of the prosthesis, the basic shade of the prosthesis is mostly attributed to intrinsic pigmentation [4]. Tear strength, hardness, tensile strength, color stability, and elongation are important mechanical properties for prosthesis, but such properties may also deteriorate over time [5,6].

Pigments play an important role of transfer the color to the maxillofacial prostheses. Intrinsic pigmentation is longer lasting and is preferred but is predicated to achieve. Several studies conducted to evaluated the effect of intrinsic pigments on mechanical properties using different type of maxillofacial silicone elastomers with different types of pigments in order to improve the properties and durability of this material [7,8]. Nevertheless, the effects of many popular pigments agents on the mechanical properties of popular maxillofacial silicone elastomers have not been evaluated.

Understanding the effect of aging and different disinfecting agents on the mechanical properties of pigmented maxillofacial silicones may help eliminate the current uncertainty as to the best follow-up suggestions for the patients treated with silicone prostheses [9].

Sunlight, moisture, and presence of dust and pollutants in the air are considered the main components of weather affecting the prosthesis. Other factors such as smoking, chewing tobacco, and household cleaning agents can degrade prosthesis materials as well [10,11].

To simulate the long-term effect of outdoor weathering condition, artificial accelerated weathering is extensively applied by using aggressive component of the weathering condition, such as UV, heat, moisture, and water spray [12].

Different weathering conditions used to evaluate their effects on different properties of maxillofacial silicone elastomers, some reports showing no effect while the other reported degradation of color and mechanical properties or causes the material to be inelastic and brittle [13–15].

This study was conducted to define the optimum concentration for a mixture of two types of intrinsic pigments (rayon flocking and burnt sienna) that could be added to VST-50 maxillofacial silicone elastomer and to evaluate their effects on some mechanical properties before and after artificial aging. The null hypothesis tested was that the addition of these pigments to this type of maxillofacial silicone elastomer have no detected differences in tear strength, hardness and surface roughness before and after subjected of material to artificial aging.

2. Materials and methods

2.1. Pilot study

Platinum room-temperature-vulcanized (RTV) VST 50, platinum-catalyzed, and vinyl-terminated silicones, as well as two pigments (rayon flocking, burnt sienna), from Factor II Inc. (Lakeside, AZ, USA), and were used in this study (Fig. 1).

A preliminary study was conducted to determine the optimal concentrations of the mixed pigments to be used in the main study. Pigments are usually incorporated in maxillofacial silicone in a concentration of 0.2 wt %; thus, concentrations higher and lower than 0.2 wt % were tested in terms of tear strength and hardness [16]. The concentrations that improved tear strength with the least effect on hardness were selected. High priority in studies was given to the concentrations that improved tear strength [5]. The pilot study showed that incorporation of 0.1 wt % of rayon flocking and 0.2 wt % burnt sienna had the highest tear strength mean value and least effect on hardness. Thus, these concentrations were selected for the main study.

2.2. Sample fabrication

A total of 180 samples were prepared and divided into 3 equal
groups. According to the tests for tear strength, hardness, and surface roughness, 60 samples were prepared for each group to measure the mechanical properties before and after weathering. Each group includes 6 subgroups with 10 samples. These subgroups represent the following:

1) Non-pigmented subgroups, which include A: samples without weathering (control), C: samples with 75 h weathering, and E: samples with 150 h weathering.

2) Pigmented subgroups, which include B: samples without weathering (control), D: samples with 75 h weathering and F: samples with 150 h weathering.

The dimensions of samples were designed using AutoCAD 2013 (Autodesk Inc., San Rafael, CA, USA), and then processed using the CNC machine (Tengzhou Jianda CNC Machine Co., Ltd, China) to form the acrylic mold (Glass-look acrylic, France) into which the material was poured.

Two groups of samples were fabricated. The first group was the non-pigmented group, which was fabricated by mixing 10:1 of base to crosslinker by weight (according to manufacturer’s instruction). The mixture was mixed under vacuum for 5 min by a Multivac 3 vacuum mixer (Degussa, Germany) (10 bar at speed 360 rpm) [17,18]. The second group was the pigmented group, which was fabricated by the same mixing ratio of the non-pigmented sample but with added pigments (Factor II Inc., Lakeside, AZ, USA). Mixing procedure was performed by weighing first 0.1% of rayon flocking and 0.2% of burnt sienna (according to pilot study), and then adding the silicone base and mixing using a Multivac 3 vacuum mixer (2 min without vacuum).

Then, the catalyst drops were dispensed in different places and the mixture was mixed again for 5 min under vacuum [17]. The material was poured at a controlled temperature of 23 ± 2 °C and 50 ± 10% relative humidity (RH) as recommended by the manufacturer and standards [19]. The silicone mixture was poured gradually, and air bubbles were removed by using fine needle (Pai Fang, China). Then, mold cover, which was coated with petroleum jelly (Kudu Co., Saudi Arabia), was laid on to the matrix [20].

A 1 kg weight (Aksan Co., Turkey) was applied on the center, and the cover was tightened by screws and nuts (Posco Co., Korea) at each corner of mold. Four G-clamps (Jumbo co., China) were added at the middle of each side. The mold was stored away from light at 23 ± 1 °C for 24 h to complete vulcanization of the RTV silicone. All samples were removed carefully from their molds so as not to strain the samples. The flash was removed with a scalpel and sharp surgical blade #10 (manufactured by Dr. Quillel Surgicals, Pakistan) [20].

2.3. Mechanical properties testing

The mechanical properties of pigmented and non-pigmented VST-50 silicone elastomer before and after artificial weathering were evaluated using the following techniques:

1) Tear strength test.  
2) Hardness test.  
3) Surface roughness test.

The tests were conducted at optimum temperature of 23 ± 2 °C. The minimum time of testing was 16 h after vulcanization [19]. The samples were stored in air-free bag (RZ products, Amman, Jordan) and light proof box (Jumbo products, China) to avoid variation in the properties of samples [21].
2.3.1. Tear strength test
   Samples preparation and testing were conducted according to ASTM D624 (2012) (Fig. 2) [19].
   The thickness of each sample was measured by a digital caliper (Lai Co, China) at three places across the width (at the center and at the end of each tab); the median of three measurements was taken. The sample was attached in the grips of a computer-controlled universal testing machine (Laryee Technology Co., Ltd., China) and symmetrically positioned in axial alignment with the direction of pull. The depth of insertion of the sample in the grips was consistent and sufficient to prevent slipping. The sample was stretched at constant crosshead speed of 500 mm/min, until the sample was ruptured, and the maximum force after break was recorded by the computer software [19]. According to ASTM D624 (2012), the following equation was used to measure the tear strength in kilo-Neutons per meter of thickness:
   
   Tear strength = F/D, where F is the maximum force required for sample breaking in kilo-Neutons, and D is the median thickness of each sample in meter.

2.3.2. Hardness test
   According to ASTM D2240-05 (2010), the hardness test must allow five points of reading at least 6 mm between each other and 12 mm away from the edge. The samples should be at least 6 mm thick. ‘The dimension of sample fabricated in this study was (40 mm × 40 mm × 6 mm)’ The samples were marked at the center and four points were marked around the central point; each point 6 mm away at each direction. Shore A hardness durometer (HT-6510A, China) was used for measurements. Based on penetration of durometer indent on the surface of the sample at five points previously marked, the durometer was pressed firmly for 1 s, and the mean of 5 readings was recorded [22].

2.3.3. Surface roughness test
   In the surface roughness average (Ra) test, ‘the sample dimensions were (10 mm × 10 mm × 2 mm)’. Profilometer tester (TR200, Time High Technology Ltd., China) was used, and the mean value of three measurements was considered as the surface roughness of the sample [23]. All the measurements were recorded from the side of the sample opposing to acrylic part of the mold because it was smoother than the glass cover [18].

2.3.4. Artificial accelerated weathering using a weather-Ometer device
   Weather-Ometer device (QUV) simulates the same condition of outdoor weathering but in an accelerated manner. Samples were arranged in the machine’s rulers, which were then inserted in the QUV machine (Atlas Electric Devices Company, Chicago, USA). The weathering standardization adopted in this study is ASTM (G154) [24]. According to cycle 7 of ASTM G154, for the samples exposed to 25 cycles, each aging cycle was accomplished in 12 h. The first 8 h (light cycle) included irradiance of (340 nm) of (1.55 W/m2) and temperature of (60 ± 3°C). The following (4 h) representing dark cycle included irradiance of (340 nm) of (1.55 W/m2) and temperature of (60 ± 3°C). The following (4 h) representing dark cycle included irradiance of (340 nm) of (1.55 W/m2) and temperature of (60 ± 3°C). The following (4 h) representing dark cycle included irradiance of (340 nm) of (1.55 W/m2) and temperature of (60 ± 3°C). The following (4 h) representing dark cycle included irradiance of (340 nm) of (1.55 W/m2) and temperature of (60 ± 3°C). The following (4 h) representing dark cycle included irradiance of (340 nm) of (1.55 W/m2) and temperature of (60 ± 3°C). The following (4 h) representing dark cycle included irradiance of (340 nm) of (1.55 W/m2) and temperature of (60 ± 3°C). The following (4 h) representing dark cycle included irradiance of (340 nm) of (1.55 W/m2) and temperature of (60 ± 3°C). The following (4 h) representing dark cycle included irradiance of (340 nm) of (1.55 W/m2) and temperature of (60 ± 3°C). The following (4 h) representing dark cycle included irradiance of (340 nm) of (1.55 W/m2) and temperature of (60 ± 3°C).
   There was no exact number of hours in the weather-Ometer chamber equivalent to one year of outdoor weathering, because that depends on factors related to weather condition. The factors include site of study and type of material being used. Nevertheless, there were some important facts that can be used as references, such as they early cumulative global radiation mean for Baghdad city of 216 MJ/m2 per year according to (Al-Douri et al., 2016). Another factor is the irradiance that is controlled at narrow wave length range in the weather-Ometer device according to cycle 7 of G154 standardization, which is equivalent to 1.55 W/m2 at 340nm. These values are then used in the following equation:
   
   \[ KJ/m^2 = W/m^2 \times 3.6 \times h. \]  [25,26].

   According to this information, one year of outdoor weathering is equivalent to nearly 387 h of artificial accelerating weathering. For Baghdad city, 75 h of accelerated weathering is equivalent to nearly 70.73 days (2.35 months) in outdoor weathering.

2.3.5. Additional tests
   2.3.5.1. Fourier transform infrared (FTIR) spectroscopy. Two samples (non-pigmented and pigmented) were analyzed by FTIR spectrometry (Shimadzu, Japan).
   2.3.5.2. X-ray diffraction. XRD 6000 (Shimadzu, Japan) was adopted to determine the degree of crystallization of the pigmented and non-pigmented samples.

2.3.6. Statistical analysis
   Data were statistically analyzed (SPSS v. 24.0 IBM, Chicago, IL, USA) by descriptive statistics (Mean, Standard Deviation, Box plots) and inferential statistics using student t-test (significant difference between two independent means at 0.05 level) and one-way ANOVA (significant difference among independent means at 0.05 level).

3. Results

3.1. Tear strength test
   Table 1 shows the descriptive statistics for tear strength values and significant differences between two independent means was evaluated using student t-test at 0.05 level. All P values showed non-significant difference at p > 0.05, and one-way ANOVA for tear strength results also showed non-significant difference at p > 0.05. The highest mean value of tear strength was measured in group B (28.268 kN/m), followed by group D (28.048 kN/m), group A (27.906 kN/m), group C (27.022 kN/m), group F (26.781 kN/m), and then group E (25.773 kN/m).

<table>
<thead>
<tr>
<th>Group</th>
<th>Mean</th>
<th>SD</th>
<th>t-test P value</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>27.906</td>
<td>0.792</td>
<td>0.684</td>
</tr>
<tr>
<td>B</td>
<td>28.268</td>
<td>1.358</td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>27.022</td>
<td>1.357</td>
<td>0.251</td>
</tr>
<tr>
<td>D</td>
<td>28.048</td>
<td>2.076</td>
<td></td>
</tr>
<tr>
<td>E</td>
<td>25.773</td>
<td>1.897</td>
<td>0.259</td>
</tr>
<tr>
<td>F</td>
<td>26.781</td>
<td>3.349</td>
<td></td>
</tr>
<tr>
<td>ANOVA</td>
<td>0.055 (NS)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

3.2. Hardness test
   Table 2 shows the descriptive statistics for hardness values and significant difference between two independent means was evaluated using student t-test at 0.05 level. P values showed non-significant difference (p > 0.05) between group C and D, whereas the significant difference was found between [A and B] and [E and F] (p < 0.05). The highest mean value of hardness test was measured in group F (33.100), followed by group D (30.960 kN/m), group A (30.630 kN/m), group C (29.122 kN/m), group D (28.781 kN/m), and then group E (25.773 kN/m).

<table>
<thead>
<tr>
<th>Group</th>
<th>Mean</th>
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<th>t-test P value</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>27.906</td>
<td>0.792</td>
<td>0.684</td>
</tr>
<tr>
<td>B</td>
<td>28.268</td>
<td>1.358</td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>27.022</td>
<td>1.357</td>
<td>0.251</td>
</tr>
<tr>
<td>D</td>
<td>28.048</td>
<td>2.076</td>
<td></td>
</tr>
<tr>
<td>E</td>
<td>25.773</td>
<td>1.897</td>
<td>0.259</td>
</tr>
<tr>
<td>F</td>
<td>26.781</td>
<td>3.349</td>
<td></td>
</tr>
<tr>
<td>ANOVA</td>
<td>0.055 (NS)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

3.3. Surface roughness test
   Table 3 shows the descriptive statistics for roughness values, and significant difference between two independent means was evaluated using student t-test at 0.05 level. P values showed non-significant difference (p > 0.05) between all groups. One-way ANOVA for roughness test results showed highly significant difference (p < 0.05).

<table>
<thead>
<tr>
<th>Group</th>
<th>Mean</th>
<th>SD</th>
<th>t-test P value</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>27.906</td>
<td>0.792</td>
<td>0.684</td>
</tr>
<tr>
<td>B</td>
<td>28.268</td>
<td>1.358</td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>27.022</td>
<td>1.357</td>
<td>0.251</td>
</tr>
<tr>
<td>D</td>
<td>28.048</td>
<td>2.076</td>
<td></td>
</tr>
<tr>
<td>E</td>
<td>25.773</td>
<td>1.897</td>
<td>0.259</td>
</tr>
<tr>
<td>F</td>
<td>26.781</td>
<td>3.349</td>
<td></td>
</tr>
<tr>
<td>ANOVA</td>
<td>0.055 (NS)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
among groups (Table 3). The highest mean value of roughness test was measured in group F (0.773 μm), followed by group E (0.668 μm), group B (0.632 μm), group D (0.631 μm), group C (0.505 μm), and then group A (0.398 μm) (Fig. 5 and Table 3).

3.4. FTIR analysis

FTIR spectra of maxillofacial silicone elastomer before and after the addition of rayon flocking and burnt sienna pigment showed no chemical reaction between VST-50 silicone and between the two pigments (Figs. 6 and 7).

3.5. X-ray diffraction

By performing XRD, the diffractogram (Intensity vs 2θ) of the polymer was obtained. The organize phase of the material was addressed by sharp peaks. Then, extracting the area of the crystalline peaks and the area of the amorphous phase, the crystallinity degree was computed by: Crystallinity = Icrystal/(Icrystal + Iamorphous). Also DSC was used for estimating the crystallinity degree.

The XRD patterns for maxillofacial silicone elastomer before and after the addition of rayon flocking and burnt sienna pigment (Fig. 8) showed a difference in the degree of crystallization. The crystallization of the non-pigmented sample was 67.58%, while that for the pigmented sample was 65.86%. The decrease in the crystallization indicated that the effect of the addition of pigments on silicone was only physical and not chemical.

4. Discussion

It is apparent that the null hypothesis was partly rejected since the hardness and roughness given differences among the tested groups, except for groups C and D presented no difference regarding the hardness test with 75 h weathering as well as there were no differences reported for tear test samples before and after weathering.

It is not easy to directly compare results, as researches varied in maxillofacial prosthetic materials tested with various silicone elastomers being heavily evaluated, experimental testing protocols, standards for the preparation of test samples and parameters used to control simulated aging conditionings [27] so in this study the comparison depend on values obtain from non-pigmented samples as standardized values. All hardness values obtained in the present study were within the acceptable range described in the literature (25–35 units) [24,28].

The major mechanical properties of VST-50 silicone are rarely studied. Rayon flocking and functional intrinsic pigment by Factor II Inc.

Table 2
Descriptive statistics for shore A hardness.

<table>
<thead>
<tr>
<th>Hardness</th>
<th>Mean</th>
<th>SD</th>
<th>t-test P value</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>30.580</td>
<td>0.579</td>
<td>0.0001*</td>
</tr>
<tr>
<td>B</td>
<td>28.980</td>
<td>0.424</td>
<td>0.246</td>
</tr>
<tr>
<td>C</td>
<td>30.630</td>
<td>0.577</td>
<td>0.246</td>
</tr>
<tr>
<td>D</td>
<td>30.960</td>
<td>0.729</td>
<td>0.0001*</td>
</tr>
<tr>
<td>E</td>
<td>31.640</td>
<td>0.615</td>
<td>0.246</td>
</tr>
<tr>
<td>F</td>
<td>33.100</td>
<td>0.787</td>
<td></td>
</tr>
<tr>
<td>ANOVA</td>
<td></td>
<td></td>
<td>0.0001# (HS)</td>
</tr>
</tbody>
</table>

Fig. 3. Box plot presentation of tear strength mean value of all study groups in kN/m.
were selected because they were the top two commonly used intrinsic pigments by maxillofacial prosthodontics [29]. Artificial accelerated weathering was employed in this study because the changes produced are greater than outdoor weathering [30]. The effects of incorporation of intrinsic pigments into VST-50 silicone elastomer on some mechanical properties before and after weathering were evaluated.

Clinically, the most important property of maxillofacial silicone is the tear strength, which indicates the thin margin integrity and durability of maxillofacial prosthesis [31]. The results of tear strength test indicated that the tear strength was increased non-significantly (p > 0.05) when the two pigments were added (Fig. 3) and (Table 1). The increase in tear strength may be attributed to the rayon flocking fibers that obstruct the propagating tear [33]. For burnt sienna (FI), this liquid pigment may likely act as a plasticizer that could enhance the tear strength [16].

Tear strength results the current study agree with the results obtained by Rai and Guttal (2013) who evaluated the tear strength of Cosmesil M511 and Biomed silicone after addition of intrinsic pigments. The tear strength values increased for both silicones after intrinsic pigmentation; medical-grade Cosmesil M511 silicone showed higher tear strength than Biomed silicone [7]. However, the results do not agree with Su et al. (2011) who added intrinsic pigments in varying concentration (0.1, 0.15, 0.2, 0.25, 0.3 wt%, respectively) to MDX-44210 silicone elastomer and found that tear strength was reduced by incorporation of the pigments and they suggest that incorporation of 0.2 wt% of pigments can alter the initial physical and mechanical behavior of the base elastomer [34]. The difference in results may be due to the types of maxillofacial silicones or techniques.

The results of tear strength showed in Fig. 3 and Table 1 indicated that the tear strength non-significantly decreased (p > 0.05) with artificial accelerated weathering after (75 h–150 h) when compared with control non-pigmented and pigmented samples before weathering. And it was noticed that the pigmented groups still reported higher mean values compared with non-pigmented groups which indicated that the addition of intrinsic pigment improved this property. The decrease in tear strength with accelerated weathering (75 h and 150 h) can be attributed to the change in chemical nature of silicone resulting in the degradation of mechanical properties, which was more obvious after 150 h. As a result, the tear strength decreased due to longer duration of weathering.

### Table 3

Descriptive statistics for surface roughness test results in μm.

<table>
<thead>
<tr>
<th>Roughness (μm)</th>
<th>Mean</th>
<th>SD</th>
<th>t- test P value</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>0.398</td>
<td>0.019</td>
<td>0.0001*</td>
</tr>
<tr>
<td>B</td>
<td>0.632</td>
<td>0.001</td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>0.505</td>
<td>0.004</td>
<td>0.0001*</td>
</tr>
<tr>
<td>D</td>
<td>0.631</td>
<td>0.001</td>
<td></td>
</tr>
<tr>
<td>E</td>
<td>0.668</td>
<td>0.002</td>
<td>0.0001*</td>
</tr>
<tr>
<td>F</td>
<td>0.773</td>
<td>0.005</td>
<td></td>
</tr>
<tr>
<td>ANOVA</td>
<td>0.0001#</td>
<td>(HS)</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 4. Box plot presentation of shore A hardness mean value of all study groups.
aging irradiation, which adversely affects the mechanical properties of maxillofacial silicone [35]. The change in mechanical properties was material dependent. The improved mechanical properties with weathering condition through chemical reaction were mainly due to photo-oxidation of polymer chains and free radical formation and reaction of these radicals with each one another, leading to further crosslinking. Furthermore, the reaction with oxygen leads to formation of peroxy radicals, which resulted in brittle and inelastic material [36].

The results coincide with Zardawi et al. (2015) who studied the effect of different natural and artificial weathering conditions on mechanical properties of silicone polymer (SP) and 3D starch printed infiltrated silicone polymer (SPIS). Their result showed a decrease in tear strength after artificial weathering for both materials [37]. However, our results disagreed with Nobrega et al. (2016) who studied the effect of artificial accelerated weathering on hardness, tear strength, and permanent deformation of MDX4-4210 maxillofacial silicone. They found that tear strength increases with artificial aging. The discrepancy could be due to different types of maxillofacial silicone and pigment additions [38].

Hardness is the resistance of the material to plastic deformation [39]. The hardness is an important property because it determines the flexibility of the material, and a material with similar flexibility to that of the surrounding defect site is desired [40].

The hardness test showed highly significant decrease (p < 0.05) in the mean value of hardness when pigments were added as compared with the control non-pigmented group (Fig. 4, Table 2), because the addition of intrinsic pigment affected the polymerization process of the silicone material, leading to decreased hardness [10].

VST-50 silicone is an addition cure platinum RTV silicone elastomer and the crosslinking reaction of this silicone involves formation of a complex that is capable of competing with the unsaturated carbon—carbon double bond that could inhibit the crosslinking reaction. In addition, hardness may be reduced because the pigments mixed with this type of silicone act as impurities that contaminate the platinum catalyst. Such contamination reduces the degree of cure, resulting in reduced hardness [32]. The results agree with Lai and Hodges (1999) who added 0.05 wt % rayon flocking to a A-2186 silicone and concluded that inhibition of cure by contamination, even in very small quantities, would result in significantly inferior hardness [41]. And also agree with Abudullah and Abdul -Ameer (2018) who found that highly significant decrease in the hardness of VST30 (RTV) silicone elastomer when each of rayon flocking (0.1 wt %) and burnt sienna (FI) (0.2 wt %) was added separately in comparison with the control group [18]. However, the results disagree with Goiato et al. (2012) who showed that the addition of make-up and ceramic powder as intrinsic pigments to Silastic MDX4-4210 silicone leads to a highly significant increase in hardness, which is assumed to be due to the rigid pigments [42]. This difference may be due to the use of different types of pigments, technique and duration for aging.

The hardness values in Fig. 4 and Table 2 indicated that hardness had increased significantly (p < 0.05) with artificial accelerated weathering after (75 and 150 h) when compared with control pigmented and non-pigmented samples before weathering. It was obviously that the pigmented groups reported higher mean values compared with non-pigmented groups which indicated that the addition of intrinsic pigment adversely affected the hardness of material and its durability. The increase in the hardness may be due to continuous polymerization as function of the aging process (UV exposure). In addition, evaporation of
Fig. 6. FTIR spectrum for non-pigmented sample.

Fig. 7. FTIR spectrum for pigmented sample.
polymer ingredient and cross linking of the material produces high temperature, thereby increasing the conversion rate and crosslinking density of the silicone elastomer, resulting in higher hardness value [23,40]. The result agrees with Goiato et al. (2012) who studied the effect of pigment addition, disinfection, and accelerated artificial weathering on the hardness of the maxillofacial silicone MDX4-4210; they found a significant increase in hardness value of all samples [42].

However, the result disagrees with Nguyen et al. (2013) who stated that after adding pigments and opacifiers, artificial accelerated weathering softens maxillofacial silicone (MDX4-4210). This difference could be due to the use of different techniques in setting process, as well as different types of silicone material and additives [13].

Surface roughness is often a good predictor of the performance of a mechanical component, given that irregularities in the surface may form nucleation sites for cracks or corrosion. It is defined as the measure of the finer irregularities of surface texture that are inherent in materials [43]. The result of surface roughness test showed a highly significant increase ($p < 0.05$) in the mean value of roughness when both rayon flocking and burnt sienna (FI) were added as compared with the control group (Fig. 5 and Table 3). Rayon flocking has short whiskers protruding from its surface [44]. Burnt sienna (FI) uses a vehicle for the pigment, which allows the pigment to be handled in liquid form. When the material hardens through evaporation or absorption of the vehicle, the distributed pigment particles on the silicone surface may increase the surface roughness [45,46]. The results of this study agree with Al-Dharrab et al. (2013). They found that the addition of liquid pigments causes surface changes of samples [43]. As well as the result agrees with Abudullah and Abdul-Ameer (2018) as they added rayon flocking (0.1 wt %) and burnt sienna (FI) (0.2%wt) to VST 30 RTV separately not as a mixture and they concluded that the addition of each of rayon flocking and burnt sienna changed the mechanical properties and increase the surface roughness of the VST-30 silicone compared to no-pigmented samples, while no superior pigment-silicone combination was revealed in all the conducted tests [18].

The results of surface roughness test shown in Fig. 5 and Table 3 indicated that surface roughness increased significantly ($p < 0.05$) with artificial accelerated weathering after (75 h and 150 h) for pigmented and non-pigmented groups. Pigmented groups shown more increase in roughness than non-pigmented so the addition of intrinsic pigment adversely affected the surface texture of material. This increase in roughness irrespective of the aging duration was most probably attributed to alteration in the material structure by the prolonged exposure to the aging conditions inside the weather-o-meter. These changes may result in the formation of micro-cracks on the surface layer of material, thereby leading to reduction in the capability of the material to withstand the aging conditions that aggravates all other degradation effects [23]. The current results agree with Al-Dharrab et al. (2013) and Fatalla et al. (2017) [43,47], but not with Goiato et al. (2009). The disagreement may be due to the use of different types of maxillofacial silicone, additives, aging conditions and durations [23].

FTIR analysis showed that no chemical reaction had occurred because of the absence of active groups. In addition, pigments were added at very small amounts, which were insufficient for any chemical reaction to occur. The only interaction was physical in nature from pigment particle interaction with the silicones crosslinking mesh, as shown in Figs. 6 and 7. XRD analysis showed little difference in the degree of crystallization, because the pigments were added at very small proportions compared to the total silicone mass. Thus, the XRD results support the FTIR analysis.

5. Conclusions

The addition of intrinsic pigments had improved hardness and tear strength of maxillofacial silicone but it did not protect the silicone material from artificial aging. Thus, all of the tested properties had adversely affected after subjecting the silicone to artificial aging.

References

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Future Dental Journal 4 (2018) 244–252


