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ORIGINAL STUDY

Shore A Hardness and Fourier Transform Infrared Spectrophotometer Analysis of Thymol-Modified Maxillofacial Silicone Material

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Abstract

The longevity of any prosthesis depends on the materials from which it was fabricated, which is why defects in the material properties may reduce the service life of the prosthesis and necessitate its replacement. This study aims to test the influence of incorporating different weight percentages of thymol micro-powder into the maxillofacial silicone matrix on Shore A hardness of the silicone. Room-temperature vulcanised VST-50 maxillofacial silicone was used to prepare the study specimens. Thymol micro-powder was incorporated into the silicone matrix in two percentages (0.75 wt.% and 1 wt.%). 30 specimens were prepared and then subdivided equally into three groups, with 10 specimens allocated for each group. These study groups are customised according to thymol content percentage: group A: Control (without additive); experimental groups B and C: 0.75 and 1 wt.% thymol additive, respectively. ANOVA and post hoc honesty significant difference tests were utilised for group comparisons (significance level set at $P < 0.05$). An additional three specimens, one for each study group, were prepared for the fourier transform spectrophotometry analysis. For the Shore A hardness test, there was a non-significant decrease in the reported mean values for both experimental groups (B and C) compared to those of the control group (A) ($P > 0.05$), while there was no chemical reaction between thymol powder particles and the silicone matrix. In conclusion, the application of thymol micro-powder did not yield a statistically significant reduction in the hardness of the silicone facial prosthesis. This outcome may suggest that the use of thymol micro-powder does not contribute to a more realistic or lifelike appearance of the prosthesis.

Keywords: Fourier transform infrared spectrophotometer analysis, Shore A hardness, Thymol micro-powder, VST-50 maxillofacial silicone

1. Introduction

Maxillofacial deformities frequently arise because of congenital anomalies, surgical resection of neoplasms, or traumatic incidents. Silicone prosthetic replacements are commonly employed to restore facial structures that are absent. On average, these prostheses have a lifespan of approximately 1.5−2 years before requiring replacement due to failure [1,2].

The prosthetic replacements help the patients return to their normal lives prior to the defect, boost self-confidence, and relieve a lot of psychological pressure by avoiding possible staring and bullying at people's looks [3]. In order to optimise durability and patient satisfaction, it is imperative for any maxillofacial prosthetic material to possess a significant level of biological, physical, mechanical, and aesthetic attributes [4].

The majority of today's maxillofacial prostheses are made of maxillofacial silicone, which is regarded as the best material for this purpose. This is because of its generally good mechanical and physical properties, low cost, simple management, biocompatibility, and suitable colouring and characterization methods [5]. However, the mechanical properties of the

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silicone prosthesis still do not meet the desired criteria for long-term service and intended longevity. In order to address these shortcomings, strengthening this material may become necessary [6,7].

Hardness is a physical property that implies the surface resistance to permanent deformation, scratching, and indentation [8].

The hardness of an elastomer is the sum of its inherent and treated hardness. These characteristics are chemically dependent on the elastomer. The processing of materials may alter their inherent hardness. The processed, moulded elastomer's hardness is dependent on the amount of inherent cross-linking inside the material that was changed [9].

Facial prostheses need to be strong enough to withstand regular use yet pliable enough to move with the face and seem natural [10,11].

In recent research studies, a variety of fillers, including chemical, herbal, nano-sized, and micro-sized fillers, have been extensively utilised as additives in silicone elastomers. These fillers possess greater rigidity compared to the polymeric matrix and exhibit a higher shear modulus. Several investigations have reported enhancements in the physical, mechanical, and biological properties of the polymeric matrix when these fillers are incorporated. This improvement can be attributed to the filler particles' large surface area and high surface energy, which render them more reactive and facilitate their integration into the polymer backbone [12–14].

Thymol powder is an organic monoterpenoid phenol, characterised by its white crystalline structure and characteristic aroma. It serves as the primary active component in natural thymol oil [15]. The substance exhibits several favourable characteristics, including antioxidant, anti-inflammatory, and antibacterial activity [16].

In the field of dental research, Hatim et al. (2010) conducted a study whereby they discovered that the incorporation of thyme oil into acrylic denture base material resulted in enhancements in the antibacterial, physical, and mechanical characteristics that were evaluated [17].

In recent years, thymol has gained significant attention as a powerful antiseptic that is used in acne treatments, hand sanitizers, and mouthwashes [16].

In addition to the fact that thymol is an antioxidant and an antimicrobial, thymol was chosen to conduct this study because of its natural plant origin as an alternative to synthetic fillers made from materials that may have harmful effects on humans and the environment.

Based on an extensive review of the literature, it seems that there is no published research examining the impact of integrating thymol with maxillofacial silicone elastomer on the physical and mechanical characteristics of the silicone material.

The aim of this research was to examine the impact of thymol micro-powder on the hardness of room-temperature vulcanised (RTV) VST-50 maxillofacial silicone.

2. Materials and methods

Room-temperature vulcanised VST-50 maxillofacial silicone (Factor II Inc., USA) was used to prepare the study specimens. Thymol micro-powder (DHA, Denmark) was incorporated into the silicone matrix in 0.75 wt.% and 1 wt.%, according to the pilot study results. A digital electronic balance (Worner Lab, China) was used to measure the needed weights of the silicone and thymol. The study involved 30 specimens distributed evenly among three groups: the 0 wt.% (group A), 0.75 wt.% (group B), and 1 wt.% (group C) thymol addition groups. The particle size of thymol particles was determined by using a particle size measuring device (Brookhaven, USA), yielding an average particle size of 2 microns.

2.1. Pilot study

A pilot study was conducted according to the study protocol approved by the scientific committee at college of dentistry in the thirteenth session of the College Council held on 3-1-2022.

Different weight percentages (0.25, 0.50, 0.75, 1.25, and 1.50 wt.%) were tested for tensile strength, elongation percentage, and candida adherence. The results of the pilot study nominate 0.75 wt.% and 1 wt.% as the best two weight percentages of thymol powder to be incorporated into the silicone matrix to improve its properties.

2.2. Molds fabrication

Clear acrylic sheets (Perspex cell-cast acrylic, Clairevaux les Lacsrance, France) were used to make the moulds. AutoCAD-2013 (A utodesk Inc., San Rafael, CA, USA) was utilised to specify the size and form of the specimens' mould, and the CNC (JL-1612, Jinan Link Manufacture and Trading Co., Ltd., China) was utilised to create the moulds [14].

2.3. Fabrication of the control silicone specimens

The control silicone specimens were prepared by mixing the silicone catalyst (Part B) and silicone

base (Part A) in a weight ratio of 1:10 (catalyst to base), following the guidelines provided by the manufacturer. The ingredients were mixed using a vacuum mixer (Manfredi, Italy) for a duration of 5 min at a rotational speed of 360 revolutions per minute and a pressure of −10 bar [1].

2.4. Fabrication of the thymol-modified silicone specimens

Thymol powder was mixed with the silicone base (part A) of the experimental specimens at the selected weight percentage using a digital vacuum mixer. As an example, the process included incorporating 0.75 wt.% of thymol powder into a 10 g of silicone base. This was achieved by combining 0.075 g of thymol powder with 9.925 g of silicone base, resulting in a total mass of 10 g for the modified base. This modified base will then be coupled with 1 g of catalyst (part B) at a later stage. Subsequently, the first two components were blended for a duration of 3 min using a digital vacuum mixer, with the vacuum function deactivated to avoid the unwanted suction of powder particles. This was followed by a further 5-min mixing period with the vacuum activated, aimed at eliminating any entrapped air bubbles. The silicone catalyst was afterwards introduced into the mixture and mixed for a duration of 5 min under a vacuum condition [3,18].

A thin coating of separating material was initially applied to the mould's cover, matrix, and bottom (BMS Dendal, Italy). The silicone mixture was then introduced into the mould [19] while maintaining a regulated relative humidity (RH) of 50 ± 10% and 23 ± 2 °C as recommended by the International Organisation for Standardisation (ISO 23529, 2016) and the American Society for Testing and Materials (ASTM D624, 2012). The specimens were retrieved from the moulds subsequent to the completion of the polymerization process. It is essential to allow a minimum of 16 h to elapse between the production phase and the commencement of testing. Then, during the time from vulcanization to testing, these specimens were stored in a storage box away from light exposure and at standard humidity and temperature settings (50 ± 10% RH and 23 ± 2 °C, respectively) that were measured using a hygrometer and a thermometer [20,21].

2.5. Shore A hardness test

Shore A hardness specimens were created in accordance with ISO 7619-1, 2010 criteria [22], and they had the following measurements: 25 mm in length, 25 mm in width, and 6 mm in thickness. The technique was carried out using a digital Shore A durometer with a digital scale (0–100) and a 1.25 mm blunt indenter. It was held upright by a mechanical stand over the specimen, which was supported by a flat, hard surface. For each specimen, readings were taken from five marked spots that were spaced 6 mm apart from one another and from the specimen's lateral boundaries after one second of steady contact over each marked point. The hardness value for a specimen is then calculated as the average of these five values [23].

2.6. Fourier transform infrared spectroscopy (FTIR)

The silicone polymer (VST-50) and thymol powder were tested to see whether there had been any chemical interaction using FTIR equipment (SHIMADZU/FTIR, 1800, Japan) [24]. Three specimens were tested: one for group A (control) and another two from groups B and C (0.75 wt.% and 1 wt.% thymol addition, respectively), as well as the thymol powder alone.

3. Results

3.1. FTIR results

Fig. 1 (A) illustrates the spectral analysis of thymol, whereby the presence of the aromatic benzene ring (C=C) is indicated by the stretching vibration at 1616 cm^{-1} [25]. Additionally, the peak at 1516 cm^{-1} signifies the waging vibration of C–H(CH_3), while the stretching vibrations at 2923 cm^{-1} and 2856 cm^{-1} correspond to C–H bonds [26].

The spectral results of the silicone are shown in Fig. 1 (B), where the peak at 2958 cm^{-1} represents CH_3 stretching vibration, the peak at 1411 cm^{-1} demonstrates the existence of the scissoring vibration ($CH{=}CH_2$), the peak at 1257 cm^{-1} is ascribed to the presence of $Si(CH_3)_3$, and the peak at 1000–1100 cm^{-1} region shows the presence of Si–O–Si [27].

Thymol powder and VST-50 RTV maxillofacial silicone had no chemical interaction, according to the FTIR spectrum data of group B and group C specimens. The sole interaction between thymol and silicone is believed to be physical, as seen by the little shift in the spectrum in the region of 1500 cm^{-1} (Fig. 1; C and D).

3.2. Shore A hardness test results

The findings of this test demonstrated that the mean value of hardness test was lowered as a result

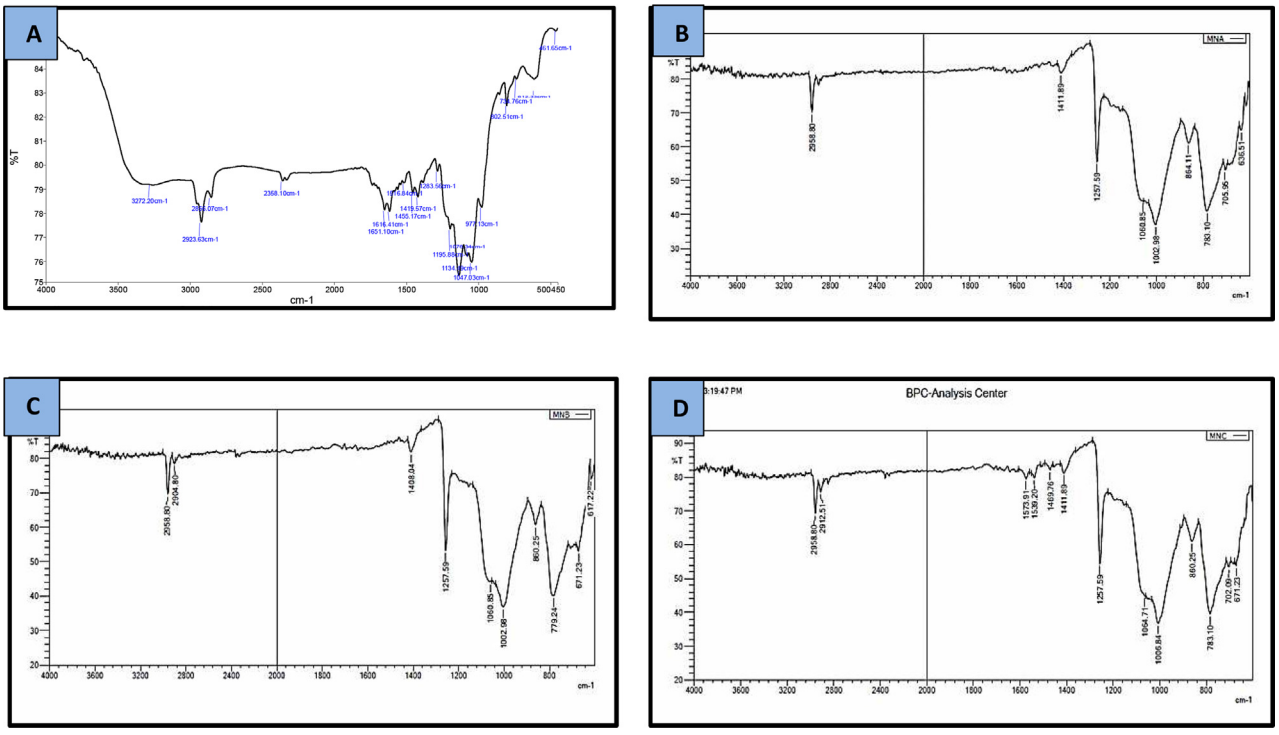

Fig. 1. Fourier transform infrared spectrum of: A (thymol powder), B (VST-50 RTV silicone elastomer without thymol powder addition), C (VST-50 RTV silicone elastomer with 0.75 wt.% thymol powder addition), and D (VST-50 RTV silicone elastomer with 1 wt.% thymol powder addition).

of thymol micro-powder incorporation. Group B had the lowest mean value compared to those of group C and the control group A, respectively. The descriptive statistics derived from the results of the analysis are shown in Table 1.

According to Table 2, the ANOVA test reveals that the effects of the thymol micro-powder addition on the Shore A hardness of the maxillofacial silicone elastomer were significant ($P = 0.05$).

Additionally, Table 3, which illustrates homogeneity of variance (Levene's test), was utilised to determine the appropriate kind of multiple comparisons to use.

Table 4, which contains the Bonferroni post hoc test findings, shows that there is a non-significant difference in hardness results between Groups (A and B), (A and C), and (B and C) as $P > 0.05$.

4. Discussion

When the FTIR spectrum test was done on specimens of silicone before and after thymol fillers were added, no chemical reaction was found between the silicone and the thymol particles. From a chemical standpoint, it is not feasible for thymol particles to combine with the saturated status of silicone rubber because it doesn't have any functional groups. In addition, the amount of thymol micro-filler added was too small to detect the interaction. The only interaction here is said to be a physical one, caused by the entrapment of powder particles with the cross-linking mesh of the silicone [28].

In comparison to the control group, the hardness mean values for both experimental groups (0.75 w.% and 1 w.% thymol powder addition) decreased not

Table 1. Descriptive statistics of the Shore A hardness (IU) test for all study groups.

Group	N	Minimum	Maximum	Mean	Std. deviation
A	10	30.400	32.300	31.417	0.5047070
B	10	30.300	31.700	30.900	0.3887301
C	10	30.300	31.900	30.975	0.5412178

Table 2. One-way analysis of variance (ANOVA) of the Shore A hardness (IU) test among groups.

Groups	Sum of squares	Df	Mean square	F	*P*-value.
Between groups	1.564	2	0. .782	3.358	0.05[a]
Within groups	6.289	27	0.233		
Total	7.853	29			

[a] = Significant at $P = 0.05$.

Table 3. Test of homogeneity of variance.

Levene statistic	df1	df2	*P*-value
0.862	2	27	0.433[a]

[a] = Non-significant at $P > 0.05$.

Table 4. Bonferroni Post hoc test results for every group of the study.

Type of multiple comparisons test	(I) Group	(J) Group	Mean difference (I-J)	Std. error	*P*-value
Bonferroni test.	A	B	0.5175000	0.2158328	0.071[a]
	A	C	0.4425000	0.2158328	0.151[a]
	B	C	−0.0750000	0.2158328	1.000[a]

[a] = Non-significant at $P > 0.05$.

significantly. Thymol is a potent anti-oxidant compound [16], while VST-50 platinum maxillofacial silicone is an addition-type polymerization, as denoted in the manufacturer information. Therefore, when thymol was added to that silicone, its anti-oxidant characteristic may have impeded the addition type silicone's polymerization activity, leading to a silicone matrix that was less fully cured and, as a result, had lower hardness values.

Furthermore, the inclusion of thymol powder particles in conjunction with the silicone has the potential to act as an impurity that can contaminate the platinum catalyst to variable extents. This contamination may subsequently hinder the curing process, leading to a decrease in hardness [28].

In accordance with these findings, research done by Abdulla and Abdul-Ameer (2018) discovered a reduction in the mean Shore A maxillofacial silicone hardness value that has been changed using intrinsic pigments [29].

Moreover, the study conducted by Shihab and Abdul-Ameer (2018) revealed a reduction in the mean hardness of VST-50 maxillofacial silicone material when intrinsic colours were added [28]. Because both act as fillers for the silicone matrix, these intrinsic pigments are the counterparts of thymol.

In contrast to these findings, Salih (2019) showed an increased Shore A hardness mean value of heat-cured soft liner specimens treated with a herbal κ-carrageenan powder compared to silicone specimens from the control group [30]. Furthermore, Ibrahim (2021) saw an increase in the hardness mean value of test silicone specimens modified with herbal κ-carrageenan powder compared to control group specimens [19].

5. Limitations

This research was an *in vitro* study in which patient-related factors were not taken into consideration.

6. Conclusion

The addition of thymol micro-powder into the silicone matrix caused a statistically non-significant reduction in the mean values of the Shore A hardness. Furthermore, there was no obvious chemical reaction between the thymol micro-powder and the silicone elastomer, as denoted by the FTIR spectrophotometric analysis.

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