

Analyzing the Influence of Yttria Stabilized Zirconia Nanoparticles Addition on Some Physical Properties of Room Temperature Vulcanized Maxillofacial Silicone

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Abstract

When acquired or congenital defects need restoration, maxillofacial silicone is the material of choice. Silicone, nevertheless, is far from perfect in quality. The primary objective of this research was to examine the effect of adding yttria-stabilized zirconia nanoparticles (YSZ NPs) (0.5 and 1% by weight) on the wettability, surface roughness, atomic force microscopy (AFM), and X-ray diffractometer (XRD) properties of maxillofacial silicone (VST-50) room temperature vulcanization (RTV). The data were analyzed statistically. The YSZ NPs were introduced to the VST-50 RTV in percentages of 0.5% and 1% by weight. A total of 63 samples of VST-50 RTV maxillofacial silicone were prepared and tested. Each group was subdivided into three identical subgroups. For the wettability test, the mean value of the 1 wt% YSZ experimental group showed the lowest wettability, followed by the 0 wt% YSZ experimental group. The 0.5 wt.% YSZ showed the highest wettability. The mean value of surface roughness decreased as the percentage of YSZ NPs increased. The results of the XRD analysis performed on the YSZ nanopowder revealed the presence of multiple peaks and the crystalline structure of nanoYSZ. Furthermore, studying AFM revealed that, as the percentage of nanoparticles increased, the surface roughness decreased. Conclusion: The addition of 0.5% YSZ NPs to VST-50 RTV maxillofacial silicone enhanced wettability and a non-significant decrease in surface roughness.

Article Info.

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Maxillofacial Silicone, Yttria Stabilized Zirconia, Nanoparticles, Wettability, Surface Roughness.

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1. Introduction

Birth abnormalities, facial trauma, or tumor removal surgery are all potential causes of facial malformation. The size or position of the defect can make surgical repair unfeasible. Ideally, a prosthesis would restore normal function and appearance by mimicking the inherent traits of the missing tissues [1]. The majority of maxillofacial prostheses are made of silicone elastomers. Because of their physical properties and flexibility, these materials are ideal for applications requiring soft tissue movement. Additionally, these materials are biocompatible, long-lasting, chemically inert, simple to work with, and comfortable to wear [2]. Despite the various positive qualities of maxillofacial silicone outweighing those of room-temperature-vulcanized (RTV) VerSilTal (VST-50) maxillofacial silicone elastomer (VST-50 (RTV)), the primary issues with silicone are tensile and tear strength reduction, and the degradation of its mechanical, physical, which are reduced elasticity, tensile strength, surface cracking and increase surface roughness and deformation in thin area, and color qualities as its ages [3]. The properties of silicone elastomers have been improved by employing various methods. Adding nanofibers or nanofillers is one way to enhance the material's mechanical and physical properties, such as its tear and tensile strengths, and make it more applicable to therapeutic applications [4].

Twelve different varieties of yttria-stabilized dental zirconia are available. Yttrium is commonly added to zirconia as a stabilizer in dental restorations. Adding 3 mol% of yttria to zirconia, typically designated as 3Y-TZP, stabilizes the metastable tetragonal zirconia polycrystal (TZP) form even at room temperature [5, 6]. Twelve different varieties of yttria-stabilized dental zirconia are available. A large temperature range is required for the

formation of the cubic phase of the material; however, stabilizing it by substituting some yttrium ions for zirconium ions makes this process possible [7]. The amount of yttria in zirconia determines the kind of zirconia (TZP, tetragonal zirconia polycrystal) [8, 9].

In terms of mechanical qualities and transparency, zirconia with a low yttria concentration (3Y-TZP, 3 mol% Y-TZP) is preferable, whereas zirconia with a high yttria content (6Y-TZP, 6 mol% Y-TZP) is less transparent but has superior mechanical capabilities. Cubic Stabilized Zirconia (CSZ) is a kind of zirconia that has a stable cubic phase at room temperature and contains more than 8 mol% yttria [10]. This study was set out to determine the impact of adding Yttria-stabilized zirconia nanoparticles (YSZnPs), which possess exclusive mechanical and physical properties, of varying percentages, on the wettability and surface roughness of VST-50.

The mechanism for achieving these goals is also determined according to hypothetical theories. The null hypothesis (H0) states that the physical and mechanical properties will not change significantly after the addition of 3 mol% YSZnPs to the maxillofacial silicone material. The alternative hypothesis (H1) suggests that the physical and mechanical properties will be significantly enhanced after the addition of 3 mol% YSZnPs to the maxillofacial silicone material.

2. Materials and Methods

2.1. Sample Preparation

The samples were made using a VST-50 (from Factor II Inc. in the USA), which is room-temperature vulcanized (RTV), and YSZnPs (from US research, Nano Materials Inc., USA). Three groups were established for the samples according to the amount of yttria in the YSZ added to the VST-50:

1. Control group: neat VST-50 maxillofacial silicone with 0 wt.% YSZnps.
2. Group I: VST-50 maxillofacial silicone with 0.5 wt.% YSZnps.
3. Group II: VST-50 maxillofacial silicone with 1 wt.% YSZnps.

Silicone elastomer VST-50 is one of the most popular and economical RTV silicone elastomers. This material is a translucent two-component, low viscosity platinum-cured silicone elastomer. The polymerization reaction is an addition, not a condensation, which means that the reaction is without by-products and with acceptable mechanical properties. VST-50 has the advantage of having an appropriate polymerization duration (6-8 hours) at room temperature [11]. YSZ has not been added previously to the RTV maxillofacial silicone, so the influence of incorporating YSZ into the VST 50-RTV maxillofacial silicone elastomer on certain mechanical and physical properties was evaluated in this study. YSZnps were selected in this study because it has impressive mechanical and physical characteristics. 3 mol% YSZ exhibited significantly greater strength in terms of its physical and mechanical properties, and it provides the highest reinforcement to rubber products, which is ascribed to its small particle size (high surface area); it is also abundant and inexpensive [12]. Sixty-three samples were split into two groups after preparation. There were 30 samples in each test, distributed as follows: 30 for the wettability test, 30 for the surface roughness test, and 3 for the Atomic Force Microscopy (AFM). All data were statistically analyzed using the (26.0) version of Statistical Package for the Social Sciences (SPSS). The control group's samples were created by combining the VST-50 maxillofacial silicone base and the catalyst and then weighing them using a 4-digit electronic scale (0.000). An amount of 200 g of silicone base and 20 g of catalyst, a 10:1 ratio by weight (manufacturer recommended ratio), were mixed in a vacuum mixer (140±10 rpm under 28-inch Hg pressure) for 5 minutes to remove air bubbles [13]. Group I and II samples were prepared by weighing the base and the YSZnps with a 4-digit electronic balance, according to the percentage of addition, subtracting the weight of the YSZnps from the weight of the base. Mixing proportions are represented in Table 1 [13].

Table 1: Mixing proportions of silicone and YSZnps.

Group	Base (g)	Catalyst (g)	YSZ (g)	Total (g)
Control (0wt.% YSZ)	200	20	0	220
0.5 wt.% YSZ	199	20	1	220
1 wt.% YSZ	198	20	2	220

2.1.1. X-Ray Diffractometer (XRD)

XRD is a technique for measuring the resulting diffraction material. The diffraction material provided information about the crystal structure and determined the average crystal size (D). D was around 19.53 nm as calculated by the Scherrer equation, Eq. (1):

$$D = \frac{0.9\lambda}{\beta \cos\theta} \quad (1)$$

where λ , θ , β represent X-ray wavelength, Bragg diffraction angle, and Full Width Half Maxima, respectively. Our calculated average crystallite size of 19.53 nm indeed falls within the established 1-100 nm range for nanoparticles.

2.1.2. Wettability Test

Samples measuring $25 \times 25 \times 6$ mm were made according to the specified dimensions [14]. A static sessile drop method was used in this study to evaluate the effect of adding YSZNPs on the wettability properties of VST-50 maxillofacial silicone material. To conduct the wettability test, a lateral view of a liquid drop placed on a solid substrate over a horizontal flat surface was captured after about 3 seconds; the view was then analyzed using Dino Capture, which is a special computer software (Fig. 1).

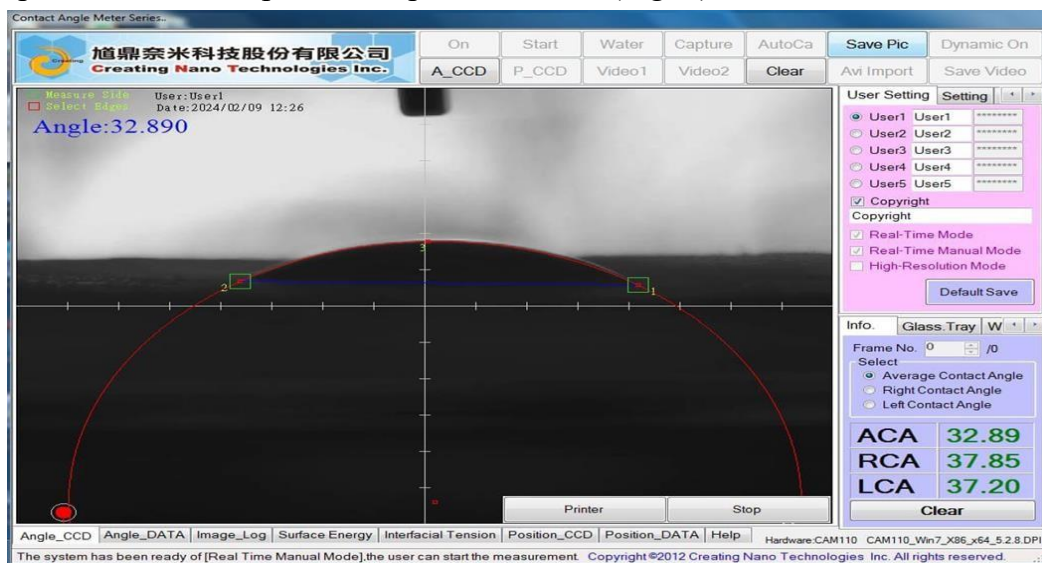


Figure 1: Dino Capture: showing a lateral view of a liquid drop.

The angle formed between the distilled water (liquid), the sample surface (solid), and the surrounding air (atmosphere) is the contact angle. A Dino-Lite digital microscope was utilized to capture a magnified image ($45\times$ magnification) of this contact angle by capturing the lateral view of the sample profile along with the distilled water drop placed over its surface. The Dino-lite microscope was positioned parallel to the sample surface. A micropipette was held vertically over the sample surface to ensure a $40\mu\text{l}$ uniform standard drop of distilled water. The Dino-Lite digital microscope and micropipette are shown in Fig. 2.

A special software system of the microscope (Dino Capture) was utilized to analyze the magnified picture automatically by drawing a tangent to the distilled water drop that take about 3 seconds before evaporation or absorption begin; the angle developed between the baseline of the drop and the tangent (three-phase-lines: air/liquid/solid) was measured and considered as the contact angle value; as the contact angle decreases the wettability increases and vice versa.



Figure 2: The Dino-Lite digital microscope and the micropipette used in the wettability test.

2.1.3. Surface Roughness Test Specimens Design

Specimens with dimensions $25 \times 25 \times 6 \text{ mm} \pm 0.2$ (Fig. 3) were prepared to be used for surface roughness test according to specification [14].

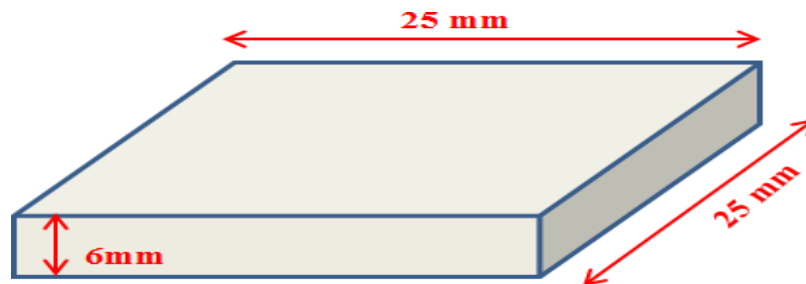


Figure 3: Dimensions of specimens used in Surface Roughness test.

2.2. Testing Procedure

A portable digital roughness tester (Contact profilometer) with an accuracy of $0.001 \mu\text{m}$ was used to assess the surface roughness of the samples. This device contains a sensitive diamond probe (surface analyzer) to trace the surface irregularities. It has been adjusted so that the stylus touches every specimen, which is placed on a solid and stable surface, taking three measurements at different locations on its surface [15]. The R_a parameter (surface roughness) is displayed on the digital scale when the stylus reaches the first point after moving over the sample surface for 11 mm (Fig. 4). Overall, these observations of surface peaks and troughs provide a basis for this metric [16]. Later, the roughness value was calculated as the mean of the three measurements [17].



Figure 4: Surface roughness test (A) Contact profilometer, (B) Surface roughness test specimen.

2.3. Atomic Force Microscopy (AFM)

The topography of the silicone samples' surfaces was analyzed with an Atomic Force Microscope (AFM, model TT-2). The samples surfaces were scanned by the probe, which consists of a sharp tip and a miniature cantilever. The apex of the sharp tip remained in continuous contact with the sample surface during imaging performed in the AFM contact mode, while it was in intermittent contact with the sample in the tapping mode. While operating in non-contact mode, the probe was entering the resonance oscillation which contains a small amount of piezoelectric material. Cantilever bending was in the non-contact mode with the tiny tip positioned at its end [18].

3. Results and Discussion

3.1 X-Ray Diffraction (XRD)

Fig. 5 displays the XRD pattern of YSZnps. All diffraction peaks corresponding to 111, 200, 220, and 311 planes can be attributed to the main peaks of YSZ, as compared to the data from JCPDS-01-082-1241 [19, 20]. The average grain size calculated by Eq. (1) yielded a value of about 19.53 nm. where 2θ (deg.) is the two-theta angle in degrees, C.S (nm) is the crystallite size in nanometers, hkl are the Miller indices of the reflecting plane, and Avg. C.S (nm) is the average crystallite size in nanometers. Characterization shows the phases of the material and crystallinity [22]. XRD analysis results of YSZ nano powder revealed the presence of multiple peaks indicative of its nanocrystallinity and nanostructure. These findings agree with those of Ahmed and Khalaf [22], who added ultraviolet-absorbing bisoctrizole to maxillofacial silicone.

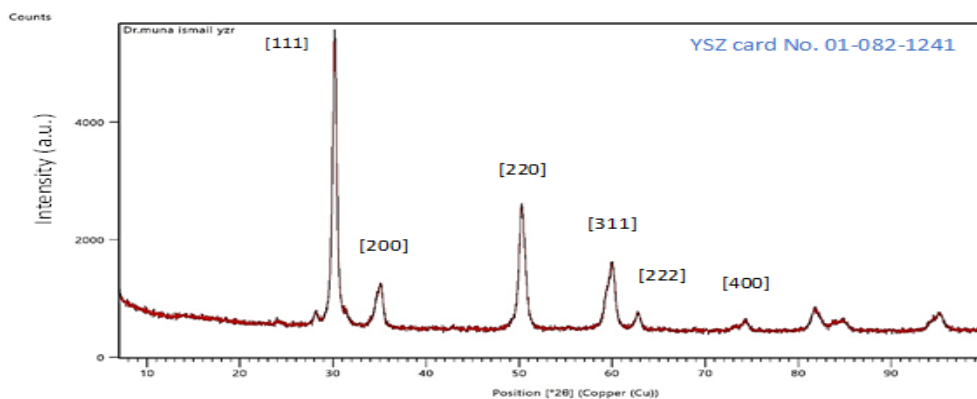


Figure 5: XRD pattern for YSZ nano powder.

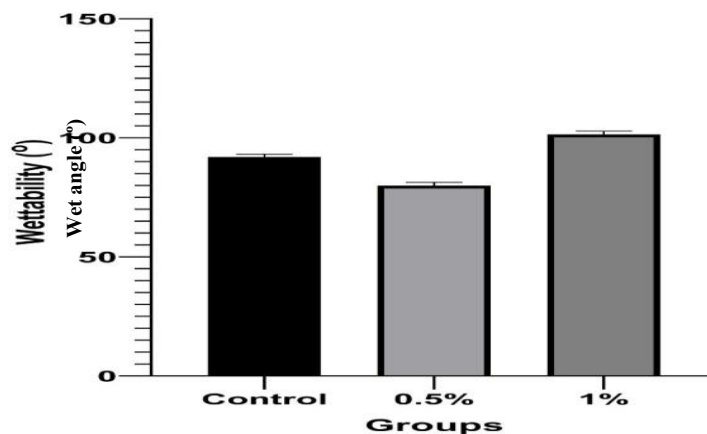
Table 2: XRD analysis for the average grain size of YSZ nanopowder (nm).

Sample	2 θ (degree)	C.S (nm)	hkl	Avg. C.S (nm)
YSZ Nanopowder	29.72	27.83585	111	19.53
	35.09	9.177674	200	
	50.4	12.33914	220	
	60.54	28.79475	311	

3.2. Wettability Test

Experimental group II (1wt% YSZ) showed the highest mean value of static contact angle (101.4°) which means lowest wettability, followed by the control group (0 wt.% YSZ) (91.91°), while group I (0.5wt.% YSZ) showed the lowest mean value of contact angle (79.92°), which means the highest (best) wettability, as shown in Fig. 6. The comparative significant F-test of the wettability test using one-way ANOVA revealed a highly significant difference among groups, as shown in Table 3. Materials used as a maxillofacial prosthesis should have good wettability, as higher wettability means a higher lubricating effect between the prosthesis and the supporting tissues [21]. The results of this study indicated a highly significant decrease in the contact angle test, increase in wettability after the addition of 0.5% YSZnps. This may be that the pores on the sample surface were filled by nanoparticles, leading to a decrease in the surface irregularities and contact angle, thereby enhancing the wettability. This result disagrees with that of AlSmael [21], who added titanium silicate nano filler to maxillofacial silicone.

Conversely, the results of adding 1% YSZnps showed that the contact angle had increased, indicating a decrease in wettability, which may be due to some agglomerations of YSZnps on silicone surface with the increase of nanoparticles concentration, this could also be attributed to the effects of the purification and surface tension of water; a liquid with low tension surface will wet the surface more easily [23]. Also, when the surface free energy of the solid is low, wettability decreases. Therefore, the wettability can be improved by either lowering the surface tension of the liquid or increasing the surface free energy of the solid [23]. The decrease in wettability at 1% YSZNPs may result from reduced surface free energy of the silicone as nanoparticle concentration increases. These findings agreed with those of AlSmael [21]. So, this decrease in wettability with the addition of 1% YSZnps, as a limitation, requires further future investigations

**Figure 6: Bar chart showing the mean and standard deviation values of the wettability test.****Table 3: One-way ANOVA for wettability test.**

ANOVA table	SS	DF	MS	DF	F Value	P value
Treatment (between columns)	2319	2	1160	27	645.2	P<0.0001
Residual (within columns)	48.53	27	1.797			
Total	2368	29				

Bonferroni multiple comparisons test was conducted to compare mean value of wettability between study groups, the test revealed a highly significant differences between all study groups, as shown in Table 4.

Table 4: Bonferroni Multiple Comparison test of wettability test.

Bonferroni multiple comparisons test	Mean Diff.	Adjusted P Value
Control vs. 0.5%	11.99	<0.0001
Control vs. 1%	-9.494	<0.0001
0.5% vs. 1%	-21.49	<0.0001

3.3 Surface Roughness

The surface roughness test of the experimental groups (0wt%, 0.5wt% and 1wt% YSZ) showed that the highest mean value of surface roughness was for the control group (0.384 μm); experimental group II (1wt% YSZ) showed the lowest mean value (0.293 μm) among all other groups, as shown in Fig. 7. Table 5 shows the Welch's ANOVA test result non-significant value. The reduction of the silicone's surface roughness may be due to filling the gaps between the polymer matrix with nanoparticles. Furthermore, well-magnetic stirring at the beginning of the mixing process allowed the nanoparticles to fill the spaces on the sample surfaces, increasing with the YSZnps increase. However, the decrease in surface roughness was statistically non-significant, due to the very small percentages of utilized nanoparticles. The results of this study agreed with the results obtained by Ali and Abdul-Ameer [24], who used hexagonal boron nitride microparticles with maxillofacial silicone. The results of this study disagree with the results obtained by Abdulkareem and Hamad [11], who added aluminum oxide (Al_2O_3) nanoparticles to maxillofacial silicone, disagree with the results of Tukmachi et al. [25], who used zirconia nanopowder with maxillofacial silicone, and also disagree with the results of Ahmed and Khalaf [22], who used ultraviolet-absorbing bisoctrizole with silicone. The limitation of this study was the use of VST-50 RTV maxillofacial silicone, which needs a long setting time of 8-12 hours. Therefore, it is recommended to use VST-30 or VST-50F instead, which have a much shorter setting time.

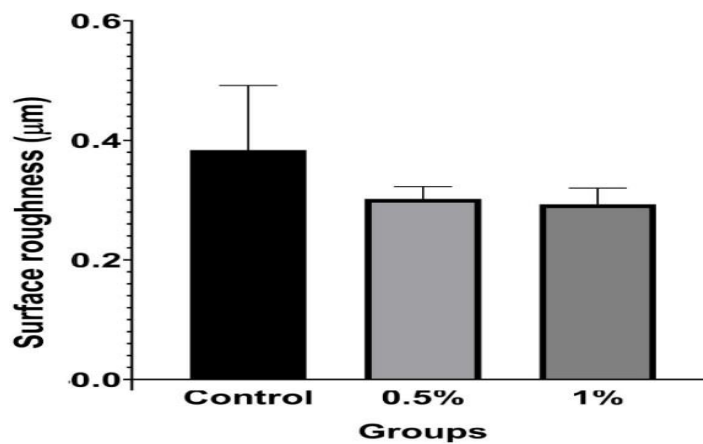


Figure 7: Bar chart shows the mean and standard deviation values of the surface roughness test.

Table 5: Welch's ANOVA for Surface roughness test.

Welch's ANOVA test	
W Value	P Value
3.242	0.0660

3.4. AFM

To quantitatively measure the surface roughness of specimens in three dimensions, studying atomic force at the microscopic level is ideal. As the percentages of nanoparticle grafting increased, the specimen roughness decreased compared to that of the control specimen. Figs. 8, 9, and 10 show two- and three-dimensional images of the control and experimental groups of VST-50 maxillofacial silicone elastomer obtained by AFM analysis, along with bar charts of YSZ powder, illustrating the granularity accumulation distribution of nanograins. The surface roughness values as reported by AFM measurements are listed in Table 6. The roughness of VST-50 maxillofacial silicone elastomer decreased from 5.16 nm in control specimens to 4.81 nm after the addition of 0.5wt% YSZ and to 3.23 nm in 1wt.% YSZ. The reason for this decrease in the surface roughness of the silicone material after increasing the concentration of YSZ may be due to the fact that the nanoparticles of the material filled the microvoids or spaces on the surface of the silicone material. The higher the concentration of YSZ, the more voids were filled, and the roughness decreased further.

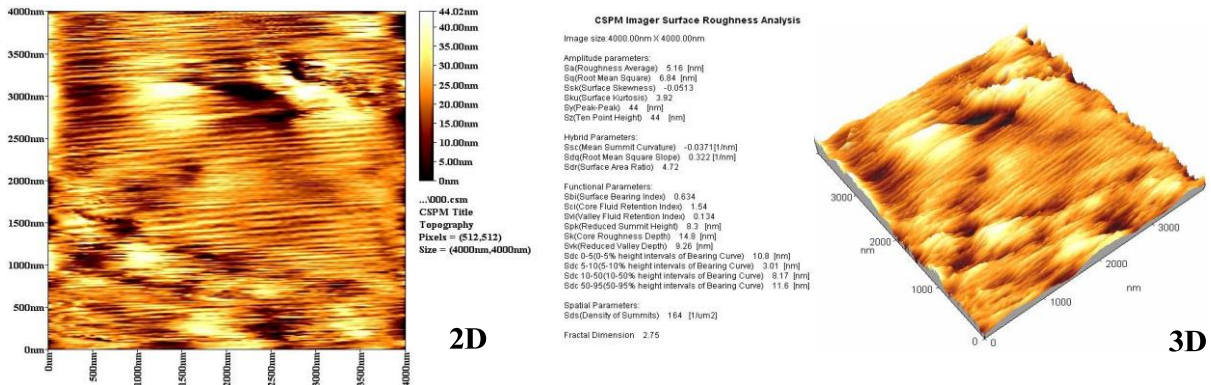


Figure 8: AFM analysis of control specimen (2D and 3D).

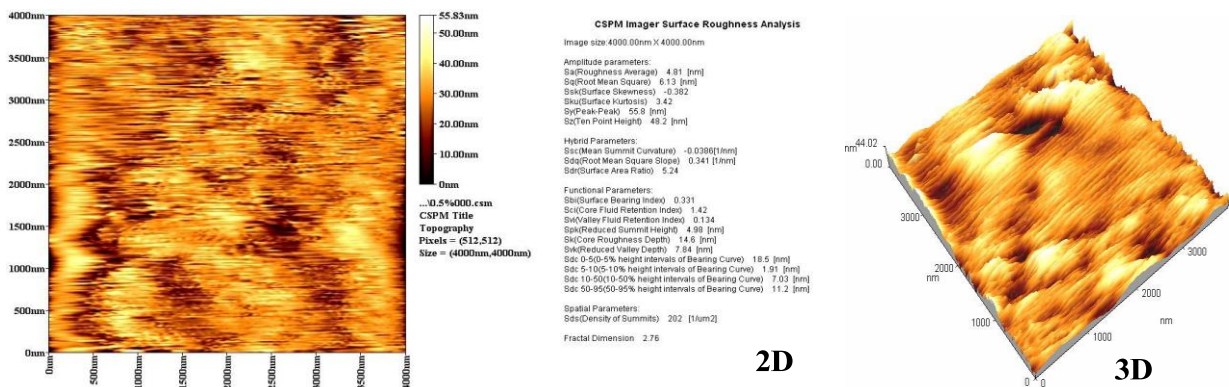


Figure 9: AFM analysis of 0.5wt.% YSZ specimen (2D and 3D).

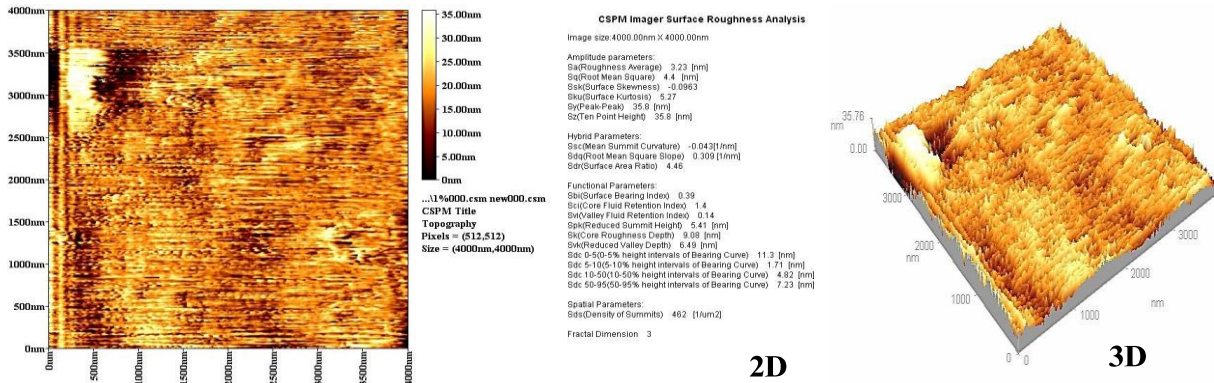


Figure 10: AFM analysis of 1wt.% YSZ specimen (2D and 3D).

Table 6: Surface roughness report of AFM.

Groups	Average roughness(nm)
VST-50 maxillofacial silicone control specimen	5.16
0.5wt% YSZ specimen	4.81
1 wt% YSZ specimen	3.23

4. Conclusions

The wettability was enhanced after the addition of 0.5 wt.% YSZ nanoparticles, showing a highly significant increase. In contrast, the addition of 1 wt.% YSZ nanoparticles resulted in a reduction in wettability. A non-significant decrease was noted in surface roughness with increasing the percentage of YSZ nanoparticle addition. The optimum rate of YSZnps to be added to the silicone is 0.5 wt.%; the decline in the surface roughness was non-significant within the acceptable clinical limit.

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Conflict of interest

The authors declare that they have no conflict of interest.

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تحليل تأثير إضافة خشوة نانوية من الزركونيا المثبتة بالإيتريا على بعض الخواص الفيزيائية لسيليكون الوجه والفكين المعالج بالحرارة عند درجة حرارة الغرفة

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الخلاصة

تُعد مادة السيليكون المستخدمة في جراحة الوجه والفكين الخيار الأمثل عند الحاجة إلى ترميم العيوب المكتسبة أو الخلقية. مع ذلك، لا يزال السيليكون بعيداً عن الكمال من حيث الجودة. الهدف الرئيسي من هذا البحث هو دراسة تأثير إضافة جسيمات أكسيد الزركونيوم النانوية المثبتة بالإيتريا (YSZnps) (بنسبة 0.5% و 1% من الوزن) على خصائص التبلل والخشونة السطحية والفحص بالمجهر القوة الذرية (AFM) وحيود الأشعة السينية (XRD) لسيليكون الوجه والفكين (VST-50) المُعالج في درجة حرارة الغرفة (RTV). تم تحليل البيانات إحصائياً. أُضيفت جسيمات YSZnps إلى سيليكون الوجه والفكين VST-50 RTV بنسبتي 0.5% و 1% من الوزن. تم تحضير واختبار ما مجموعه 63 عينة من سيليكون VST-50 RTV. قُسمت كل مجموعة إلى ثلاث مجموعات فرعية متطابقة. بالنسبة لاختبار التبلل، أظهر متوسط قيمة المجموعة التجريبية التي تحتوي على 1% من YSZ أقل قابلية للتبلل، تليها المجموعة التجريبية التي لا تحتوي على YSZ. بينما أظهرت المجموعة التي تحتوي على 0.5% من YSZ أعلى قابلية للتبلل. انخفض متوسط قيمة الخشونة السطحية مع زيادة نسبة جسيمات YSZnps. كشفت نتائج تحليل حيود الأشعة السينية (XRD) الذي أُجري على مسحوق YSZ النانوي عن وجود قمم متعددة والبنية البلورية لـ nanoYSZ. علاوة على ذلك، أظهرت دراسة AFM أنه مع زيادة نسبة الجسيمات النانوية، انخفضت الخشونة السطحية. الخلاصة: أدت إضافة 0.5% من جسيمات YSZnps إلى سيليكون الوجه والفكين VST-50 RTV إلى تحسين قابلية التبلل وانخفاض غير ملحوظ في الخشونة السطحية.

الكلمات المفتاحية: سيليكون الوجه والفكين، الزركونيا المثبتة بالإيتريا، الجسيمات النانوية، قابلية البلل، خشونة السطح.