

RESEARCH PAPER

Analyzing the Influence of Yttria Stabilized Zirconia Nano Particles Addition on Some Physical and Mechanical Properties of Room Temperature Vulcanized Maxillofacial Silicone

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ABSTRACT

Examining the effects of Yttria Stabilized Zirconia (YSZ) nanoparticles (nps) added at concentrations of 0.5% and 1% on the physical and mechanical properties of maxillofacial silicone elastomers was the primary objective of this research. In order to verify the size of the particles in (YSZ), the particle size analyzer was used. A 0.5% and 1% weight of YSZnps was added to the maxillofacial silicone (VST-50) Room Temperature Vulcanized (RTV). There were 96 samples divided into three groups one control and two experimental, 10 samples were evaluated for each. Each set of criteria included tear strength, tensile strength, and Shore-A hardness. Additionally, three samples were tested for Fourier Transform Infrared Spectroscopy (FTIR) and Field Emission Scanning Electronic Microscope (FESEM). For the purpose of studying the dispersion of YSZnps in silicone elastomer, the following instruments were utilized: particle size analyzer, Field Emission Scanning Electron Microscope (FE-SEM), Energy Dispersive X-ray spectroscopy (EDX), and Fourier Transform Infrared spectroscopy (FTIR). Then SPSS (26.0) version have been used to analyze the data statistically. All the results of the conducted tests were significantly higher after the nano particles addition. The silicone elastomers showed a well-distributed nanoparticle in the FESEM pictures. There was no chemical change as seen by the FTIR spectra. We conclude that the most appropriate addition of YSZ NPs in percentage of 0.5wt% and 1wt% to the VST-50 RTV maxillofacial silicone which enhanced all physical and mechanical properties.

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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 imitating the missing tissues' inherent traits [1]. The majority

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of maxillofacial prosthesis are made of silicone elastomers. Because of their physical properties and flexibility, these materials are ideal for uses where soft tissue movement is required. 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 outweigh those of VST-50 (RTV), the primary issues with silicone are its poor elasticity, low tensile and rip strength, and the degradation of its mechanical, physical, and color qualities as it ages [3]. The properties of silicone elastomers have been improved by using a number of 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. The amount of yttria in zirconia determines the kind of zirconia (TZP, tetragonal zirconia polycrystal) [5, 6].

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 metastable tetragonal zirconia polycrystal (TZP) form is stabilized down to room temperature by the addition of 3 mol% of yttria to zirconia, which is a commonly used stabilizer in zirconia applications like dental restorations. The 3Y-TZP designation is typically given to this zirconia substance [8, 9]. In terms of mechanical qualities and transparency, zirconia with a lower yttria concentration (3Y-TZP, 3 mol% Y-TZP) is preferable, whereas zirconia with a higher yttria content (6Y-TZP, 6 mol% Y-TZP) is less transparent but has superior mechanical capabilities overall. 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 set out to determine how much of an impact the addition of Yttria stabilized

zirconia (YSZ) nanoparticles (NPs) in different percentages which possess exclusive mechanical and physical properties on tear strength, tensile strength, and surface hardness of maxillofacial silicone that has been vulcanized at ambient temperature (VST-50).

MATERIALS AND METHODS

The particle size analyzer was used to confirm the particle size of YSZnps. That must be in the range of 1-100 nanometers.

Getting the samples ready

The samples were made using VST-50, a maxillofacial silicone from Factor II Inc. in the USA, which is room-temperature vulcanized (RTV) and Yttria Stabilized Zirconia (YSZ) nanoparticles (NPs), (US research, nano materials Inc., USA).

Three categories were established for the samples: a) Control group: neat silicone with 0% wt. YSZ nanoparticles, b) Group I: silicone with 0.5% wt. YSZ nanoparticles, c) Group II: silicone with 1% wt. YSZ nanoparticles.

The 96 samples were split into three categories after preparation. There were 30 samples in each group, distributed as follows: 10 samples for each tear strength test, tensile strength test, and shore A hardness; and Fourier Transform Infrared spectroscopy (FTIR) and Field Emission Scanning Electron Microscopy (FESEM), each need three samples. Energy Dispersive X-ray Spectroscopy (EDS) was done also, all data were statistically analyzed using (26.0) version of SPSS.

The samples for the control group were made by combining the VST-50 maxillofacial silicone base and catalyst and then weighing them using a 4-digit electronic scale (0.000), at ratio 10:1 by weight (manufacturer recommended ratio)¹¹ followed by mixing in a vacuum mixer (140±10 rpm under 28-inch Hg pressure for 5 minutes) to remove air bubbles. An amount of 100g of silicone base and 10g of catalyst were mixed using vacuum mixer for 5 minutes to remove air

Table 1. Mixing proportions of silicone and YSZnps.

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

bubbles [11].

Group I and II samples were prepared by weighing on an electronic balance 4 digit, according to the percentage of addition. Subtract the weight of YSZ from the weight of the catalyst. Mixing proportions are shown in (Table 1).

Starting the mixing of catalyst with YSZ NPs by using magnetic stirrer, they were stirred for 10 minutes with mixing speed 150 – 200 rpm to get the nanoparticles evenly distributed and mixed then added to silicone base with the same mixing proportions and the whole mixture was mixed under vacuum for 5 minutes (140 ± 10 rpm under 28-inch Hg pressure for 5 minutes), to prevent air entrapment, then allowed to cool for 5 minutes to avoid heat raising [11].

Measurement of tear strength

Methods outlined in ISO 34-1 (12) were followed for the tear test. The samples were subjected to stretch at speed of 500 mm/min until they failed, while being fastened to a universal testing equipment (WDW-20, Laryee Technology Co. Ltd., China). At the right-angle region, we measured the sample thickness (t) in millimeters using a digital caliper, and we recorded the greatest force (F) at failure in Newtons.

The tear strength test was done by Eq. 1:

$$T_s = f/d \tag{1}$$

Where the T_s stands for tear strength, the maximal force is denoted by (N), the thickness of the specimen is denoted by d (mm).

Tensile strength

Was carried out using type 2 dumbbell-shaped samples in accordance with ISO 37¹³ guidelines. Straining the samples at a speed of 500 mm/min until they broke was done using an extensometer attached to the universal testing equipment which record the elongation and stretch.

The value of the tensile strength as determined by the following Eq. 2:

Where:

$$UT_s = F_m/Wt \tag{2}$$

Where UT_s is the ultimate tensile strength, F_m is the maximum force recorded at break (N), W is the width of the specimen (mm), t is the thickness of the specimen (mm).

Shore A hardness

The Specimens used were prepared with

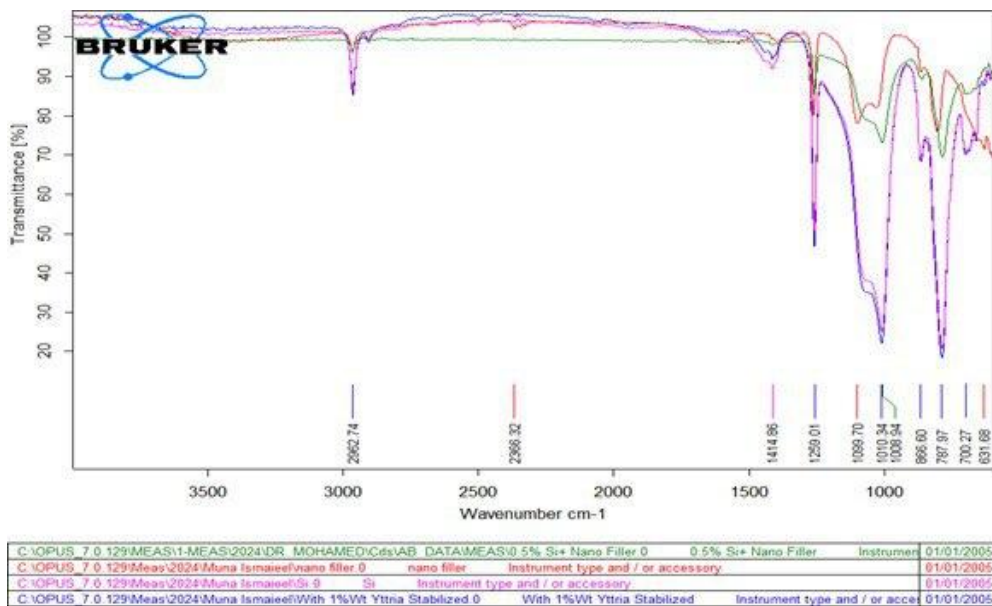


Fig. 1. FTIR for YSZ nano powder, control group, silicone with 0.5% wt. YSZ nanoparticles and silicone with 1% wt. YSZ nanoparticles.

dimensions (25mm length x 25 mm width x 6 mm thickness) according to specification ISO 48-4 (14). The concept of this technique is dependent on the puncturing of durometer stylus by using a blunt indenter with 1.25mm diameter to conduct the hardness test using a digital scale ranging from 0 to 100 into the sample's surface at five places marked formerly. Within each specimen, five points were marked with at center and every point should have at least 6 mm of space between it and the lateral boundaries and adjacent points. The hardness value was given as the average of the five measurements.

The FTIR

Measures the transmission of infrared light at different frequencies, by analyzing the specimen's absorption and transmission spectra, a molecular fingerprint may be derived (15).

FE-SEM and EDX

The surface and morphology of silicone matrix were observed by FE-SEM for YSZ NPs. The distribution and proportion by which each element of YSZ particle in the silicone was also

assessed by EDX (elemental distribution).

RESULTS AND DISCUSSION

Particle size analysis for YSZ powder

The diameter of YSZ powder was proven according to the particle size analyzer, to be 58.8 nanometers, effective diameter 58.8 nm, polydispersity 0.369, baseline index 0.0, elapsed 00:00:30.

FTIR

FTIR spectral results of YSZ NPs powder, VST 50 maxillofacial silicone, both before and after the addition of YSZ NPs powder (0.5% and 1% concentrations) and mix of all, are shown in (Fig. 1). From chemical point of view there is no chemical reaction, due to the absence of new active functional group in both sides.

FE-SEM

FE-SEM image of YSZ powder refer to presence of nanoparticles with size and diameter of particle (Fig. 2 A).

FE-SEM images of VST-50 silicon polymeric matrix before addition of YSZnps particles (control group) was shown in (Fig. 2 B).

FE-SEM images of VST- 50 silicone after

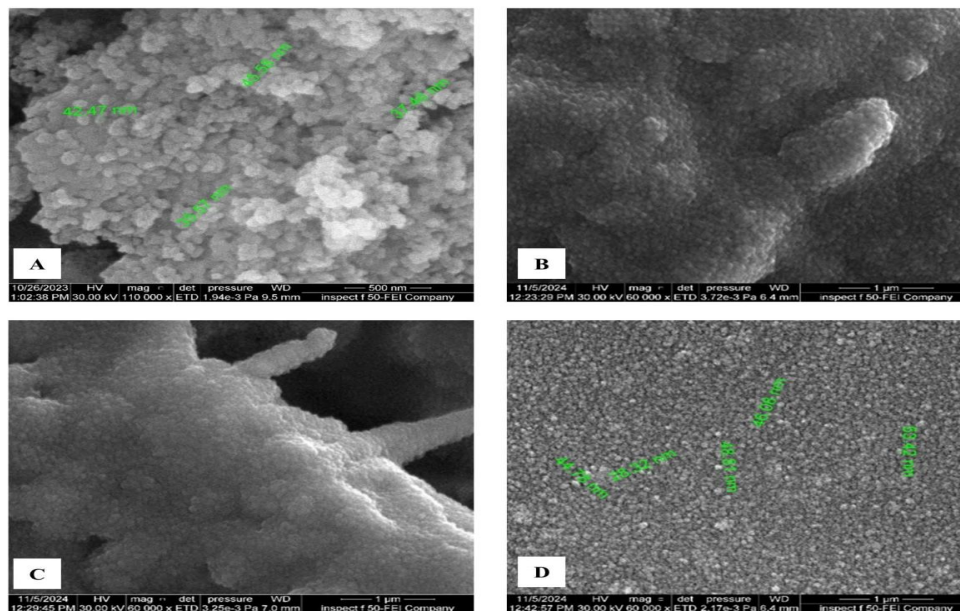


Fig. 2. FE-SEM images of: A, YSZ (NPs). B, silicone control group 0% wt YSZ. C, YSZ distribution in silicone with 0.5% group. D, YSZ distribution in silicone with 1% group.

addition of 0.5 % wt. YSZ nanoparticles, and 1% wt. YSZ presented no agglomeration and well distribution of YSZ NPs within the VST- 50 silicone polymeric matrix after using of magnetic stirrer resulting in improving dispersion of nanoparticles within the silicone material

(Fig.2C and D).

Energy dispersive X-ray spectroscopy (EDX)

EDX mapping for YSZ NPs powder are shown in (Fig. 3) which shows the presence of Oxygen (O), Ytria (Y) and Zirconia (Zr) elements.

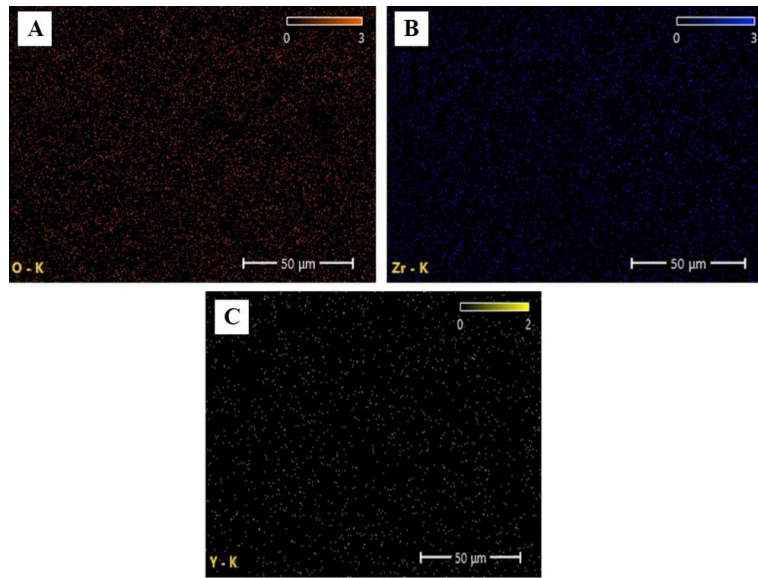


Fig. 3. Count map of YSZ powder specimen: A, spreading of oxygen element. B, spreading of zirconia element. C, spreading of yttria element.

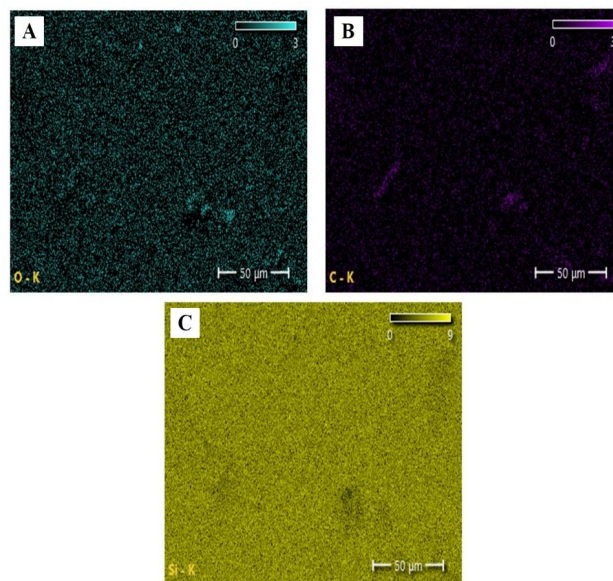


Fig. 4. Count map for silicone VST-50 control group: (A) spreading of oxygen element. (B) spreading of carbon element. (C) spreading of silicone element.

While control sample shows no YSZ NPs but only Si, O and C. As shown in EDX mapping (Fig. 4).

EDX mapping for silicone VST-50 incorporation with 1% YSZ group. As shown in (Fig. 5).

Tear strength

All experimental groups with higher mean value than the control group. Experimental group (1wt% YSZ) showed the highest mean value (24.34N/mm) among all other groups as shown in (Table 2). The Brown-Forsythe test shows a high significant value (non-homogenous). In order to compare the mean values of the

different groups, we used Games-Howell's multiple comparisons test. The results showed that there were a highly significant differences between groups, as showed in (Table 2).

Tensile strength

The control group's mean values were lower than those of all experimental groups. The group treated with 1% YSZ had the highest mean values, at 5.038 MPa, followed by the 0.5% YSZ group (4.801 MPa), while the lowest mean value was for control group (3.976 MPa). The comparative significant F-test by ANOVA test showed a high significant difference between all groups.

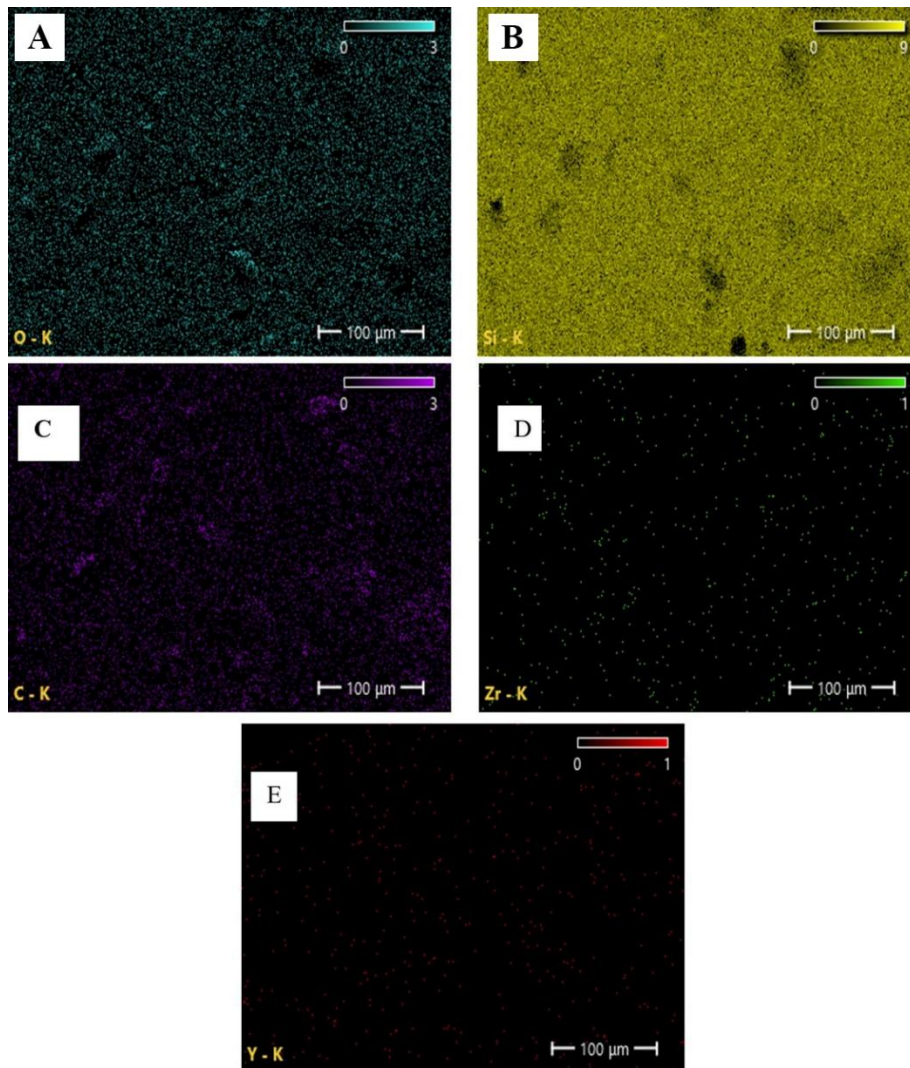


Fig. 5. Count map of silicone incorporation YSZ specimen; A, spreading of oxygen element, B, spreading of silicon element. C, spreading of carbon element. D, spreading of zirconia element. E, spreading of yttria element.

The Brown-Forsythe test was nonsignificant value that mean homogenous, as shown in (Table 3). Bonferroni's multiple comparisons test examined the mean values of the research groups and found that there were a highly significant differences between (Control vs. 0.5wt% and Control vs. 1wt%), while comparison between (0.5wt% vs. 1wt%) was nonsignificant as shown in (Table 3).

Shore A hardness

The result of experimental group of VST-50 maxillofacial silicone elastomer with (1 wt% YSZ) presented the highest mean values (36.00 N/mm), followed by the (0.5wt% YSZ) group with a mean value of (33.14 N/mm), while the control group presented lowest mean value (30.43 N/mm). Games-Howell's multiple comparisons, Table 4 shows the results of a test that compared the mean values between different study

groups; the results showed that there were a highly significant differences between all of the groups. Games-Howell's multiple comparisons test of shore a reveled there is highly significant increase in hardness.

Silicone elastomer VST-50 is the most popular and economical RTV silicone elastomers. This material is a translucent two-component, low viscosity platinum-cured silicone elastomer, addition type polymerization reaction with no by-products and acceptable mechanical properties. The addition of nanoparticles to silicone materials has been found to enhance their physical and mechanical properties to some extent [16]. YSZ NPs was selected in this study because it has good physical and mechanical properties. The mechanical and physical behavior of YSZ 3 mol% materials had much higher strength (high hardness, rigidity and thermal stability) and it provides the highest

Table 2. Statistical Data of tear strength test.

	Control	0.5%	1%	Welch's ANOVA test		Brown-Forsythe test (Homogeneity test)	
				W Value	P Value	F Value	P Value
Number of values	10	10	10				
Minimum	19.42	21.25	23.16	127.0	<0.0001	64.38	<0.0001
Maximum	20.67	24.93	25.56				
Range	1.250	3.680	2.400				
Mean (MPa)	20.02	23.10	24.34				
Std. Deviation	0.4593	1.240	0.7453				
Std. Error of Mean	0.1452	0.3921	0.2357				
Games-Howell's multiple comparisons test				Mean Diff.		Adjusted P Value	
Control vs. 0.5%				-3.075		<0.0001	
Control vs. 1%				-4.320		<0.0001	
0.5% vs. 1%				-1.245		0.0399	

Table 3. Statistical Data of tensile strength test.

	Control	0.5%	1%			
Number of values	10	10	10			
Minimum	3.630	4.420	4.780			
Maximum	4.280	5.260	5.250			
Range	0.6500	0.8400	0.4700			
Mean (MPa)	3.976	4.801	5.038			
Std. Deviation	0.1945	0.2793	0.1371			
Std. Error of Mean	0.06152	0.08831	0.04335			
ANOVA table						
	SS	DF	MS	DF	F	P value
Treatment (between columns)	98.89	2	49.45	27	64.38	P<0.0001
Residual (within columns)	20.74	27	0.7680			
Total	119.6	29				



reinforcement to rubber products which is attributed to its small particle size (high surface area) is also abundant and inexpensive [17].

FTIR analysis revealed the absence of any chemical reaction. When fillers react with silicone, the only contact that occurs is a physical reaction, often known as a hydrogen bond or Van der Waals bond. This physical interaction which is resulted from nanoparticles interaction with the silicones to form cross-linking mesh that appeared as a minimal change in vibration absorption of already existing bonds, with a change in transmittance of silicone matrix for light. This agrees with Ali and Abdul-Ameer¹⁸ who use hexagonal boron nitride microparticles with maxillofacial silicone.

Field emission scanning electron microscope (FE-SEM) showed that the YSZ was evenly distributed throughout the silicone matrix as the filler percentage increased in the samples. The silicone matrix achieved very good nanoparticle dispersion improved without any agglomeration which occur because of magnetic stirring of YSZ NPs with silicone before vacuum mixer, and this disagree with Kareem et al. [11], Kumail and Hamad¹⁹ who add barium titanate nanoparticles 1wt% and 2wt% to the VST-50 RTV maxillofacial silicone and Tukmachi and Safi [20] who use zirconia nano powder with maxillofacial silicone.

The uniform distribution of nanoparticles in the silicone material, as well as the improvement in both mechanical and physical characteristics, may explain why the material is so homogenous that caused by magnetic field effect.

Results of tear strength test revealed enhancements for both experimental groups (0.5wt% and 1wt% YSZ) as compared to the

control group, their proportions increased directly with the increase in percentage, the results agree with Kareem, et al. [11] who add barium titanate nanofillers 0.75wt% to the VST-50 RTV maxillofacial silicone and also agree with Ali [21] who add nano-cellulose fibers 0.5wt% to the VST-50 RTV maxillofacial silicone as they found improvement in tear strength. By physically building 3D networks within the silicone matrix, and certain polymer chains that incorporated with nano particles which producing filler meshes inside the polymer matrix. The polymer chains are unable to move in opposition to the nanoparticles or each other due to the interaction between the nanoparticles and the polymer matrix, that increased in density and tear strength [22].

These findings disagreed with those of Chowdhary [23] who found no change in the tear strength values of the silicone after the inclusion of silver nanoparticles. As each study was applied in different experimental conditions, varying kinds of silicones and nanoparticles employed at varying ratios, might be the reason for the differences, since each investigation was conducted under distinct experimental settings.

The tensile strength test showed an increase with the addition of YSZ NPs. This improvement could be attributed to the physical interconnection between YSZ NPs and the VST-50 silicone matrix. The particles and polymer chains interacted when subjected to tensile forces, leading to enhanced resistance against failure and breakage of the polymer chains [24, 25]. It shows that the addition of YSZ increased the value of tensile strength compared to control group.

The improved tensile strength seen in this

Table 4. Statistical Data of the Shore A hardness test.

	Control	0.5%	1%	Welch's ANOVA test	
				W Value	P Value
Number of values	10	10	10	672.5	<0.0001
Minimum	30.10	32.73	34.83		
Maximum	30.80	33.57	36.63		
Range	0.7000	0.8400	1.800		
Mean	30.43	33.14	36.00		
Std. Deviation	0.1825	0.2498	0.5624		
Std. Error of Mean	0.05770	0.07901	0.1778		

research could be because of the physical attachment of the YSZ NPs to the silicon matrix. Increasing tensile forces resulting in sliding of the polymer chains and the nanoparticles over each other; as the presence of nanoparticles will prevent the polymer chains from breaking, they will move across the polymer chains as the tensile pressures increase. This prevented chain sliding and breakage and improving tensile strength [26].

These findings were agreed with the results of Mashloosh [16] who add copper oxide nanoparticles to RTV maxillofacial silicone and agree with Ahmed [27] who add bisoctrizole organic ultraviolet absorber to RTV maxillofacial silicone, and disagreed with the results reported by ALAnsari [28] using a mix of RTV maxillofacial silicone, polyamide microparticles, and silicone dioxide nanoparticles.

Shore A hardness test shows a highly significant enhancement with addition of (0.5% YSZ) followed by (1% YSZ) when compared with the control group. This means that the hardness increased with acceptable clinical values, Shore A hardness test must use a range of 25–45 IU with the facial missing part dependence [29].

The explanation is that nanoparticles are well dispersed within the polymer while gradually building networks inside polymers, which in turn reduces the inter-aggregate space and makes the material harder and stiffer. This agreed with Kumail and Hamad [3] who added barium titanate nanofillers to maxillofacial silicone. But they disagree with Ali and Abdul-Ameer [18] who used hexagonal boron nitride microparticles and also disagree with Chowdhary [23] who added silver nanoparticle to maxillofacial silicone, when they found a reduction in hardness after the addition of nanoparticles to silicone elastomeric polymer. This disagreement may be due to different in brands of silicones, type of additive materials, percentages and methodology.

CONCLUSION

The addition of (YSZ NPs) in (0.5% and 1%) concentrations to VST-50 maxillofacial silicone significantly improved tear and tensile strength; with this improvement, there was a clear correlation between the increasing in the concentration of additional nanoparticles and the remarkable increase in tear and tensile

strength. The inclusion of YSZ NPs increased surface hardness of maxillofacial silicone and this rise in acceptable clinical range (25–45 IU) at 0.5% and 1% YSZ NPs by weight. The most appropriate addition of YSZ NPs in percentage of 0.5wt% and 1wt% to the VST-50 RTV maxillofacial silicone which enhanced all physicommechanical properties that tested in this study.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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