

RESEARCH PAPER

Influence of Adding Zirconia-Yttria Nanoparticles on Tear Strength, Tensile Strength and Elongation Percentage of Maxillofacial Silicone Type A-2186

Mohammed Fadhil Noori *, Jwan Fateh Abdulkareem

Prosthodontics Department, College of Dentistry, University of Sulaimani, Madam Mitterrand Street, Sulaimani, Iraq

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ABSTRACT

The purpose of this study is to assess the impact of incorporation of Zirconia-Yttria (ZrO_2 -3Y) nanoparticles on certain mechanical properties of A-2186 maxillofacial silicone elastomer, such as tear strength, tensile strength and elongation percentage. A-2186 Platinum silicone elastomer was modified by adding ZrO_2 -3Y nano powder at two weight percentages (1% and 1.5%). A total of 60 specimens were prepared and subsequently divided into three groups: two experimental groups (1% and 1.5% ZrO_2 -3Y) and one control group. Each group was further subdivided into three identical subgroups in accordance with the testing that was intentionally conducted. Ten specimens were employed for each test (tear strength, tensile strength test and elongation percentage). A one-way ANOVA followed by Tukey's HSD post-hoc test was conducted after confirming homogeneity of variance, with a significance level of $p < 0.05$. Both experimental groups (1% and 1.5% of ZrO_2 -3Y) revealed a highly significant increase in tear strength compared to control group. Additionally, the 1.5% ZrO_2 -3Y silicone group showed a significant improvement in tear strength compared to the 1% ZrO_2 -3Y group. Regarding tensile strength, groups (1% and 1.5% ZrO_2 -3Y) exhibited a significant increase in tensile strength compared to the unmodified silicone group ($p < 0.05$). A non-significant difference was also noted between group (1% ZrO_2 -3Y) and group (1.5% ZrO_2 -3Y). On the other hand, both groups (1% and 1.5% ZrO_2 -3Y) showed a highly significant decline in their percentage of elongation compared to the control group. The addition of ZrO_2 -3Y nanoparticles to A-2186 platinum silicone significantly enhanced tear and tensile strengths, but led to a notable reduction in elongation, indicating a trade-off between mechanical strength and flexibility.

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INTRODUCTION

Reconstruction may not always be an effective treatment for facial deformities resulting from congenital, trauma, or surgery. In such instances,

maxillofacial prostheses can be used to restore both function and appearance, particularly in the case of head and neck defects [1]. Prosthetic materials for facial reconstruction showcase a

* Corresponding Author Email: Mohammed.fadhil@univsul.edu.iq



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wide variety of chemical compositions, leading to an extensive range of physical characteristics. These characteristics differ greatly, encompassing the rigidity and resilience of metals and synthetic materials to the adaptability of flexible materials and pliable substances. Examples of these materials include latexes, poly (methyl methacrylate) (PMMA), poly(vinyl chloride) (PVC), polyurethanes, and silicone rubber materials [2]. Silicone elastomers are considered the most preferable material for the production of facial prostheses [3-5]. Silicone provides adaptability, enhancing the individual's overall health and ease. Furthermore, it features characteristics like a texture that closely mimics human skin, stability under heat exposure and the capability to inhibit bacterial growth by repelling water, blood, and organic materials. The characteristics of silicone render it an excellent option for creating facial prosthetics, guaranteeing superior performance and patient contentment [6,7]. Silicone has limitations, particularly in terms of early material deterioration, despite its advantages. Modified texture, ill-fitting margins as a result of shape changes, and decreased tear strength are all potential issues that silicone prostheses may encounter within one to three months. These factors underscore the necessity of consistent maintenance and replacement to guarantee the longevity and efficacy of silicone-based maxillofacial prostheses [7-9]. It is recommended that silicone prostheses be replaced on a regular basis due to the rapid deterioration of their mechanical and physical properties over time, and the complexity of repairing such prostheses [10]. A variety of methods, such as the incorporation of nanoparticles, nano-oxides, and colours and opacifiers, have been employed to prevent the disintegration of silicone [11,12]. The mechanical and physical properties of polymers can be improved by reinforcing them with nanoparticles. This is due to the nanoparticles' high surface energy, polarity, reactivity, and large surface area, which facilitate strong interactions with polymer chains and the formation of a distinctive 3D composite [13,14]. The nanoparticles of zirconium oxide are biocompatible and possess exceptional mechanical strength, durability, and resistance to corrosion and attrition [15]. In addition to possessing the highest hardness of any oxide, nano- ZrO_2 is noted for having high mechanical qualities that enable it to resist the propagation

of cracks [16]. It has been demonstrated that the mechanical properties of maxillofacial silicone are substantially improved by the addition of zirconia nanoparticles. The incorporation of these nanoparticles enhances the tensile strength, shear resistance, and hardness of the material, making it more durable and resilient for long-term prosthetic use [17-19]. Zirconia assumes primarily a tetragonal structure upon yttrium oxide (Y_2O_3) stabilization, which is important for the improvement of the mechanical performance by the inhibition of crack propagation. Yttria, a chemically and thermally stable oxide, is used in ceramic processing to improve toughness and thermal degradation resistance. It is also important in maintaining the crystalline structure of zirconia [19,20]. Moreover, incorporating yttrium oxide nanoparticles into maxillofacial silicone has been demonstrated to substantially improve its mechanical performance. By enhancing tear resistance, increasing tensile strength, and increasing surface hardness, these nanoparticles render the material more suitable for extended use in facial prosthetics [21,22].

Zirconia-yttria poly crystals has been made available to dentistry through the CAD/CAM technique. Stabilized zirconia by yttria ceramics are used among other applications for hip joint prostheses and have been shown to have excellent mechanical performance and superior strength and fracture resistance compared to other ceramics [23]. Yttria-stabilized tetragonal zirconia poly crystal has been widely used in dentistry (dental ceramic) as a core material for crowns and fixed prostheses due to the enhanced mechanical properties (Young's modulus, flexural strength, fracture toughness and hardness) and higher biological stability compared to other dental ceramics [24]. Given these synergistic properties, this study was designed to investigate the influence of a zirconia-yttria nanoparticles (ZrO_2 -3Y) on the mechanical behaviour of room-temperature-vulcanized (RTV) maxillofacial A-2186 platinum silicone elastomer. Three crucial mechanical properties were examined in this study: tear strength, tensile strength and elongation percentage. The aim is to ascertain whether adding this nanoparticle can increase the silicone's mechanical strength without negatively affecting its flexibility and improving its clinical applicability for long-term prosthesis use. It was hypothesized that the addition of the zirconia-yttria nanoparticles would not change

the tear strength, tensile strength, or elongation percentage of the material.

MATERIALS AND METHODS

In this study, zirconia-yttria nano powder (US Research Nanomaterials, Inc, USA) and A-2186 Platinum room-temperature vulcanized silicone elastomer (Factor II Inc., USA) were used.

Specimen Grouping

A total of 60 specimens were prepared and randomly assigned to three primary groups based on the concentration of ZrO₂-3Y nanoparticles incorporated into the silicone matrix:

Group A: Control group (pure silicone, 0% nanoparticles), Group B: 1 wt% ZrO₂-3Y silicone, Group C: 1.5 wt% ZrO₂-3Y silicone.

Each group was further subdivided into three subgroups (n = 10) corresponding to the specific mechanical test performed: tear strength, tensile strength, and elongation at break.

The G*Power software version 3.1.9.7 was employed to calculate the sample size, which was based on the results of previous studies [25]. (alpha: 0.05, power: 0.90, and effect size f: 0.7); the sample size for each group was 10.

Mold fabrication

Laser engraving machines (JL-1612, Jinan Link Manufacture and Trading Co., Ltd., China)

were employed to cut acrylic sheets (2-6 mm) based on the thickness of each test. Cutting was executed in accordance with the predetermined specifications for each test, as determined by the computer software AutoCAD 2019 (Autodesk Inc., San Rafael, CA, USA). The mold is composed of a base, matrix, and cover, which are secured by bolts and screws. Further tightening was achieved by employing G-clamps at the edges to ensure dimensional stability during curing process (Fig. 1)

Mixing procedure

For the control group, the silicone base and catalyst were mixed in a 10:1 weight ratio, as recommended by the manufacturer. The mixture was blended using a vacuum mixer (Renfert, Germany) at 360 rpm and placed under a -10 bar vacuum for 5 minutes, following the guidelines of ISO 23529:2016 [26]. The procedure was executed at a temperature of 23 ± 2°C and a humidity of 50 ± 10%.

For 1% and 1.5% ZrO₂-3Y silicone specimens, an electronic balance with an accuracy of 0.0000 (KERN & Sohn, Germany) was employed to accurately measure the fillers concentration. Filler was introduced initially, followed by silicone Part A, and the mixture was stirred for 10 minutes. To prevent particulate suction, the initial three minutes were mixed without vacuum, followed by seven minutes under vacuum. After allowing

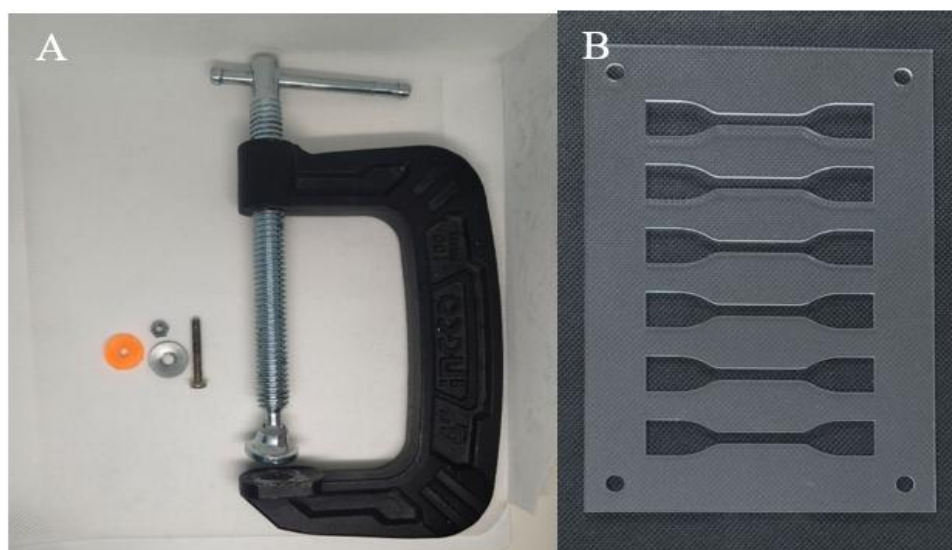


Fig. 1. A) G-clamp, bolts, nuts, metal and rubber washers. B, acrylic mold.

the mixture to cool to room temperature, Part B (catalyst) was added and mixed under vacuum for an additional 5 minutes [17].

Packing, Curing, and Storage of specimens

To prevent air bubbles from forming on the specimens, a disposable plastic syringe was used to inject the silicone mixture into the molds. The mixture was then placed in a closed position and sealed with bolts and G-clamps, (Fig. 2). Subsequently, molds were allowed to set for 24 hours in a laboratory setting. Specimens were later demolded and checked for defects such as air bubbles, edge defects, or other irregularities (Fig. 3). To avoid any form of degradation before undergoing mechanical testing, specimens were stored inside a dark container where both

temperature and humidity were regulated.

Materials Characterization

Fourier transform infra-red (FTIR)

The potential chemical interactions between the silicone polymer and the ZrO₂-3Y nanoparticles were evaluated using FTIR spectroscopy (Shimadzu, Japan) analysis. From each subgroup, one specimen was assessed to characterize functional groups and compositional shifts through spectra evaluation.

Scanning electron microscopy (SEM)

SEM imaging (Thermo Fisher Scientific, Netherlands) was used to assess the dispersion of ZrO₂-3Y nanofiller particles within the silicone

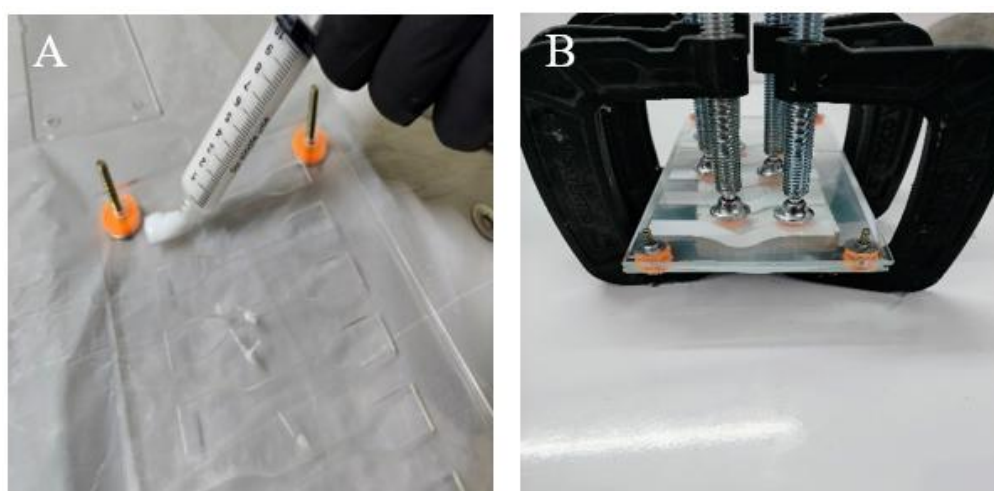


Fig. 2. A) injection of silicone into mold. B) the mold is secured with screws, nuts, and G- clamps after pouring the mixed silicone inside.

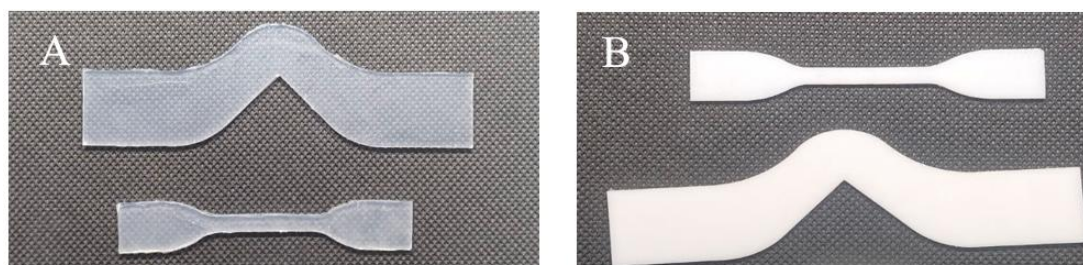


Fig. 3. A) Specimen of control group. B) Specimen of experimental group.

matrix. One specimen from each group were imaged at a magnification of 50 μm .

Testing procedures

Tear strength test

Tear strength testing was performed in accordance with ISO 34-1:2015 [27]. Unnicked angle-shaped specimens were mounted on a universal testing machine (Hongjin, China) and subjected to tear loading at a crosshead speed of 500 mm/min until failure. Maximum force (N) and specimen thickness (mm) were recorded to calculate tear strength (N/mm).

Tensile strength and elongation at break tests

Tensile and elongation testing was conducted

using dumbbell-shaped specimens according to ISO 37:2017 [28]. Samples were clamped in a universal testing machine (Hongjin, China), and elongation was measured using a digital caliper (China). The test was performed at a crosshead speed of 500 mm/min. Maximum force at break (N), initial gauge length (L_0), and final length (L_b) were recorded to calculate tensile strength (MPa) and elongation at break (%).

The statistical analysis

All statistical analyses were conducted using IBM SPSS Statistics (version 26; IBM Corp., Armonk, NY, USA). One-way analysis of variance (ANOVA) was employed to assess group differences. Tukey's Honest Significant Difference (HSD) test was

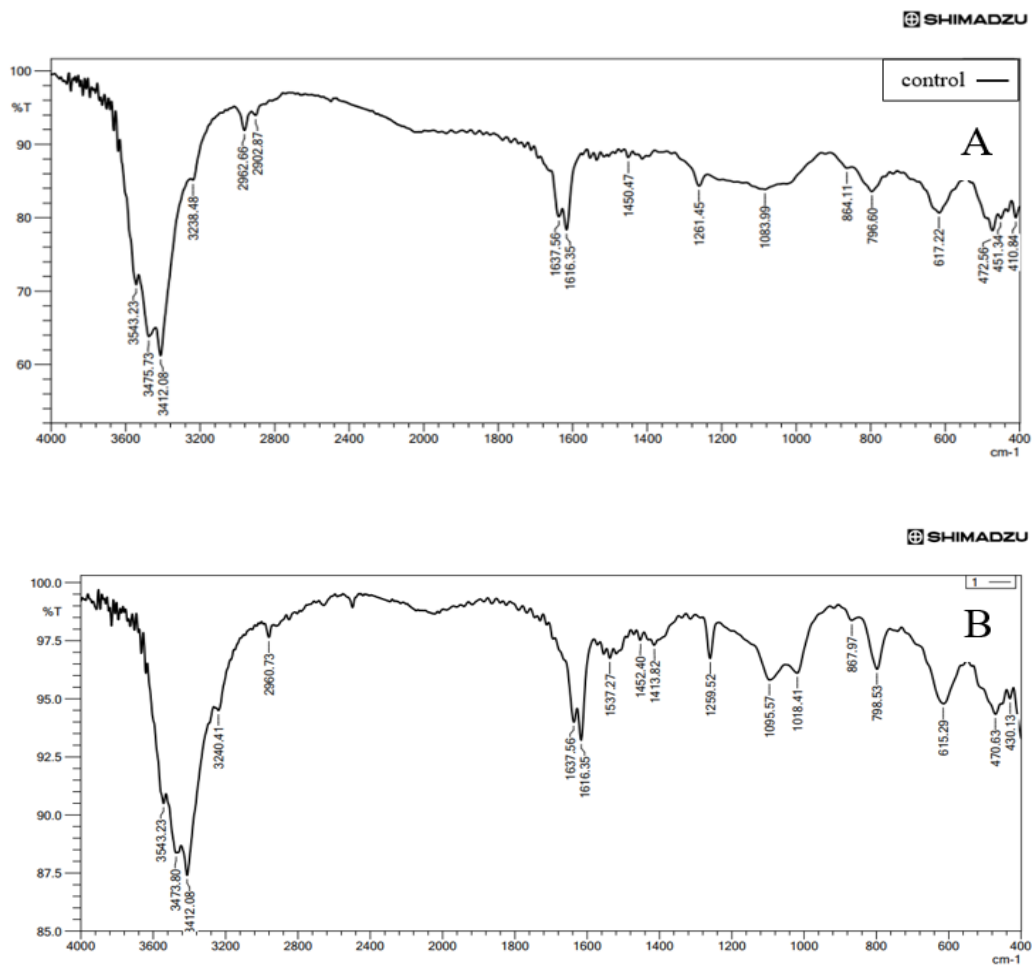


Fig. 4. A) FTIR pattern of control sample. B) FTIR pattern of 1% ZrO_2 -3Y silicone sample.

employed for post hoc pairwise comparisons, when found effects were significant. Homogeneity of variances was evaluated using Levene's test. Statistical significance was set at $p < 0.05$, with $p < 0.01$ indicating a highly significant difference.

RESULTS AND DISCUSSION

Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR results found no chemical variations between the samples before and after ZrO_2 -3Y nanoparticles addition, (Fig. 4).

Scanning Electron Microscopy (SEM)

SEM images confirmed that the ZrO_2 -3Y

nanoparticles were generally well-dispersed within the silicone matrix. However, minor particle agglomeration was noted in the 1.5% group, particularly at higher filler concentrations, (Fig. 5).

Mechanical tests results

Tear strength

The experimental group (C) exhibited the highest mean tear strength followed by the group (B) while the control group (A) recorded the lowest mean value. One-way ANOVA revealed a statistically significant difference in tear strength among the groups ($p < 0.001$). Post hoc analysis demonstrated highly significant differences between the control

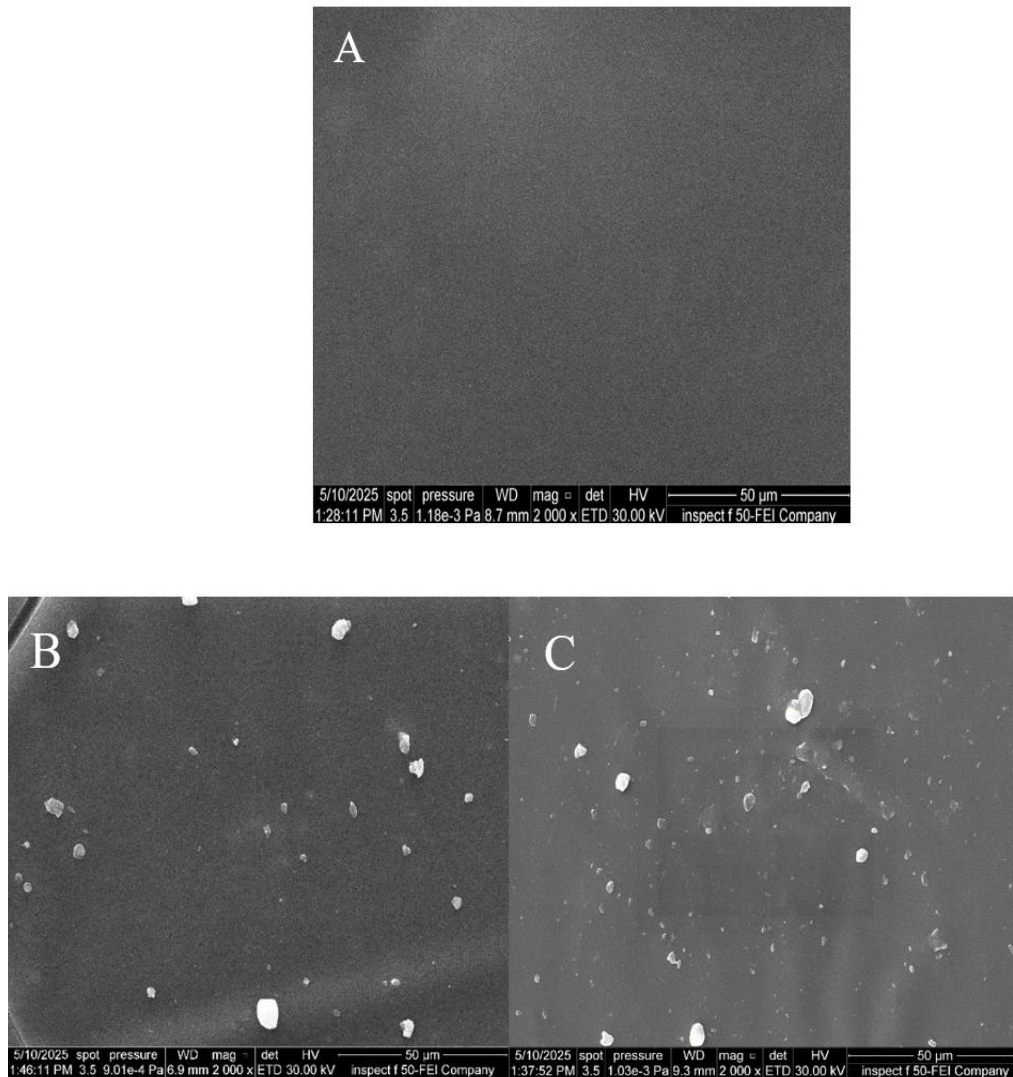


Fig. 5. SEM image (50um magnification) A) control group. B) 1% ZrO_2 -3Y group. C) 1.5% ZrO_2 -3Y group.

group (A) and both experimental groups (B and C), as well as between (B and C) groups (Table 1).

Tensile strength

The (C) group showed the highest mean tensile strength followed by (B) group, while the control group (A) had the lowest. One-way ANOVA revealed a significant difference among groups ($p < 0.05$). Tukey's post hoc test showed significant differences between the control and experimental groups, but no significant difference between (B and C) groups (Table 2).

Elongation percentage

The control group (A) exhibited the highest mean elongation percentage followed by group (B), while group (C) showed the lowest value. One-way ANOVA revealed a statistically significant difference among the groups ($p < 0.001$). Tukey's HSD post hoc test showed highly significant

differences between the control group and both experimental groups, as well as between (B and C) groups (Table 3).

Clinical durability of maxillofacial silicone prostheses depends on materials that can endure mechanical load without compromising flexibility. Although A-2186 silicone is widely used, it also has recognized limitations, i.e., tear and tensile strength. In addressing this, this study investigated the effect of incorporating zirconia-yttria (ZrO₂-3Y) nanoparticles, and the results clearly support rejecting the null hypothesis: the nanoparticles did have effects on the mechanical properties of the material.

The SEM micrographs confirmed good dispersion of the ZrO₂-3Y nanoparticles within the silicone matrix at the 1% level. Some particle clustering was observed at 1.5%, which may explain some of the trends in performance. Notably, FTIR spectroscopy was unable to reveal

Table 1. Minimum values, maximum values, means, standard deviation, ANOVA. (one way), and post-hoc test of tear strength.

Groups	Tear strength N/mm			ANOVA		Tukey HDS	
	Mean $\pm$ SD	Max	Min	F	P value	Groups	P value
A	14.71 $\pm$ 0.826	15.81	13.12	36.950	0.000	AB	0.000 HS
B	16.53 $\pm$ 0.744	17.64	15.21			AC	0.000 HS
C	17.71 $\pm$ 0.781	18.84	16.82			BC	0.006 HS

Levene statistics=0.03973 p value=0.961 [NS]

Table 2. Minimum values, maximum values, means, standard deviation, ANOVA (one way), and post-hoc test of tensile strength.

Groups	Tensile strength Mpa			ANOVA		Tukey HDS	
	Mean $\pm$ SD	Max	Min	F	P value	Groups	P value
A	5.11 $\pm$ 0.515	5.84	4.23	4.85	0.016	AB	0.049 S
B	5.57 $\pm$ 0.355	6.02	4.98			AC	0.021 S
C	5.63 $\pm$ 0.32	6.17	5.01			BC	0.924 NS

Levene statistics=1.407 p value=0.262 [NS]

Table 3. Minimum values, maximum values, means, standard deviation, ANOVA (one way), and post-hoc test of Elongation percentage.

Groups	Elongation percentage %			ANOVA		Tukey HDS	
	Mean $\pm$ SD	Max	Min	F	P value	Groups	P value
A	565.91 $\pm$ 21.86	592.34	523.92	27.44	0.000	AB	0.005 HS
B	539.78 $\pm$ 16.9	571.85	512.31			AC	0.000 HS
C	509.48 $\pm$ 10.4	524.09	495.52			BC	0.001 HS

Levene statistics=1.324 p value=0.283 [NS]

chemical bonding between fillers and polymer, suggesting that reinforcement is primarily physical in nature.

Concerning tear strength, both modified groups were superior to the control, and the highest values were observed for the 1.5% ZrO₂-3Y silicone group. This could be a result of the nanoparticles forming a microstructural network that resists crack propagation and contributes towards energy dissipation under loading [29-32]. The same findings have been observed in previous work with zirconia and other nano-oxides [17,33]. However, some differences are to be found in the literature: for instance, those with other fillers like TiO₂ or chitosan composites showed no or even negative effects on tear strength [12,34,35] supporting the hypothesis that filler type as well as concentration are critical factors.

Tensile strength also showed notable improvement in both experimental groups. While 1.5% ZrO₂-3Y showed slightly higher values than 1%, the difference wasn't statistically significant. One possible explanation is that excessive filler content may lead to nanoparticles agglomeration, creating weak zones within the matrix and limiting further strength gains [14,36]. This trend has also been noted in other studies involving zinc oxide or halloysite fillers [33,37] emphasizing the importance of optimized dosing.

Conversely, the percent elongation decreased significantly in both experimental groups compared to the control. This is not entirely surprising as nanoparticles restrict polymer chain mobility and provide sites of cross-linking, they spontaneously reduce material extensibility. [29,38,39]. While this loss can be undesirable in very flexible prosthesis, it may be an acceptable trade-off for prosthetic regions where toughness is superior to elasticity. Previous research supports this finding, reporting similar patterns with the incorporation of strontium titanate, yttrium oxide, and other nanoparticles [12,17,40].

Overall, while the improvement in strength properties is encouraging, the subsequent loss of elasticity demands balance. Future formulations might benefit from combining ZrO₂-3Y with plasticizers or hybrid fillers to preserve elongation while retaining mechanical reinforcement. Furthermore, although the mechanical improvements are promising, clinical durability in actual use depends on more than initial strength tests. Future research should investigate the long-

term performance of these modified silicones under realistic use conditions, including prolonged exposure to ultraviolet radiation, moisture, and physiological environments, to ensure their stability and safety for implantation in human tissue.

CONCLUSION

Within the findings of this study and in its limitation, the following conclusions were drawn:

1. The zirconia-yttria nanoparticles were successfully incorporated into A-2186 maxillofacial silicone without altering its chemical structure, as confirmed by FTIR analysis, and with good dispersion as shown by SEM imaging.

2. Both 1% and 1.5% concentrations of ZrO₂-3Y significantly improved the tear and tensile strength of A-2186 silicone elastomer, with 1.5% concentration having the best resistance to tearing.

3. Addition of ZrO₂-3Y nanoparticles caused a significant drop in the percentage of elongation, with a greater effect when the concentration was at 1.5%, indicating a decrease in flexibility.

These findings suggest that zirconia-yttria nanoparticles is a suitable reinforcement for enhancing the mechanical performance of maxillofacial silicones. However, optimization of filler concentration is essential to balance mechanical strength with the required flexibility for clinical applications.

CONFLICT OF INTEREST

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

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