



## Effect of Silver-Zinc Zeolite Addition on Mechanical Properties of Maxillofacial Silicone

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### ABSTRACT

**Background:** Deterioration of maxillofacial silicone properties due to microbial colonization is a common problem and leads to the replacement of the prosthesis. Incorporation of the antimicrobial agent into the silicone could be a solution. The purpose of this study was to evaluate the effect of silver-zinc zeolite addition on some mechanical properties of a maxillofacial silicone (VST-50). **Materials and methods:** Total 120 specimens were fabricated and divided into 3 groups: 40 specimens for tear strength test, 40 specimens for tensile and percentage of elongation tests and 40 specimens for Shore A hardness and surface roughness. Each group was divided into 4 subgroups according to the amount of zeolite added (0% "control", 0.5%, 1% and 1.5%). One-way ANOVA and Tukey's HSD tests were used to analyze the study data. FTIR revealed a chemical interaction between zeolite and silicone. **Results:** Study results revealed a highly significant increase in tear strength ( $p < 0.01$ ) in all experimental subgroups. The tensile strength of 1% filler was significantly higher than the control group ( $p < 0.01$ ) while 0.5% and 1.5% showed a non-significant increase. All experimental groups demonstrated a highly significant decrease in the percentage of elongation and a highly significant increase in roughness ( $p < 0.01$ ). The subgroup with 1.5% zeolite showed a highly significant increase in hardness, other subgroups showed a non-significant increase. **Conclusion:** The addition of 1% Ag-Zn zeolite improved some mechanical properties of VST-50 maxillofacial silicone.

**Keywords:** Ag-Zn zeolite, VST-50 maxillofacial silicone, Tear strength, Tensile strength, Shore A hardness, Roughness

### INTRODUCTION

Patients with facial defects and deformities can be rehabilitated with maxillofacial prosthetics [1]. The most important goals of the maxillofacial prosthesis are to restore the natural appearance of such patients and improve their self-image [2,3].

Over the past few decades, the need for prosthetic rehabilitation has proportionally increased since surgical intervention may not always be possible because of the location and size of the defect [4,5]. Maxillofacial prostheses are fabricated using various types of polymeric materials such as polyvinyl chloride, polyurethanes, poly (methyl methacrylate), chlorinated polyethylene and silicones [6,7].

The ideal physical and mechanical properties of the maxillofacial material should be comparable to that of the tissue to be replaced. These materials should be non-toxic and tissue-compatible, should be colored with intrinsic and extrinsic pigments, should have easy manipulation and processing and should not deteriorate during patient use [3,8].

Silicone has overtaken other materials as the material of choice for facial prostheses construction [9]. This is mainly due to its strength, ease of manipulation, chemical inertness, durability, and the comfort that they offer to the patient compared to older materials. But yet still, its mechanical properties are far from the ideal requirements [10,11]. Prostheses made of silicone need to be replaced periodically because their color and physical properties deteriorate rapidly over time and repairing of silicone are very difficult [4,6]. Also after a few months of usage, microorganisms start to colonize the prosthesis causing unpleasant appearance and may encourage infection of the adjacent tissues [12,13].

The required physical and mechanical properties of silicone elastomer depend on both the nature and concentration of the filler used with the polymer. Fillers can be custom-added and adjusted to produce a strong material and elastic at the same time and fulfills the required clinical properties [14-16].

It was stated that the addition of fillers with small particle size and the large surface area could enhance the physical and mechanical properties of maxillofacial silicone. Such small sized fillers have the potential to improve tear strength, tensile strength, the percentage of elongation and hardness [17].

Since the usual methods for infection control such as daily cleaning with different disinfection solutions affect the mechanical properties of silicone elastomers and shorten the service life of the prosthesis, it was proposed that the addition of antimicrobial agents to elastomeric silicone, to obtain antifungal and antibacterial properties, would prolong the elastomer life [18,19].

Most antifungal agents used in past studies were organic compounds such as benzimidazole and methylsulfonyl. Such organic agents are vulnerable to temperature, light, radiation, and humidity, thus they can be deteriorated easily and their effect no longer last [13].

Antimicrobial zeolites have been used as filler with dental materials to prevent or reduce bacterial, fungal and yeast contamination [20]. In a previous study, zeolite was added to improve the mechanical properties of vulcanized rubber. It should be pointed out that natural zeolite could be the appropriate filler for the applications where a moderate tensile strength but good abrasion resistance are needed. The improvement in the rubber properties is due to the small particle size of the zeolite and the large filler-matrix interface adhesion [21].

Many authors found zeolites containing silver and zinc ions are excellent candidates to be added to elastomeric silicone to obtain antimicrobial property because zeolites have prolonged antimicrobial activity, low toxicity, no odor or taste and they are chemically stable against temperature and humidity change [12,13,20].

The porous structure of Ag-Zn zeolite facilitates the slow release of antimicrobial metals and it allows regeneration by secondary ion-exchange when metals are depleted [22].

The purpose of this study was to evaluate the tear strength, tensile strength, and percentage of elongation, shore A hardness and surface roughness of a maxillofacial silicone elastomer after the addition of a prepared silver-zinc zeolite powder. The null hypothesis was the addition of Ag-Zn zeolite powder would not affect the properties of maxillofacial silicone.

## MATERIALS AND METHODS

In this study, an RTV (room temperature vulcanized) maxillofacial silicone (VST-50, Factor II Inc., USA) and prepared silver-zinc zeolite powder (average particle size of 1 micron) were used. The preparation and characterization of Ag-Zn zeolite powder are described in a previous study [23]. The Ag-Zn zeolite was added to the silicone in 4 percentages: 0% (control), 0.5%, 1% and 1.5% by weight. Total 120 specimens were fabricated and divided into 3 groups according to the mechanical properties tested as follows:

Group 1: 40 specimens for tear strength test.

Group 2: 40 specimens for tensile strength and elongation at break tests.

Group 3: 40 specimens for shore A hardness and roughness tests.

Each group was then divided into 4 subgroups according to the weight percentage of Ag-Zn zeolite as follows:

Subgroup A: 10 specimens with 0% Ag-Zn zeolite.

Subgroup B: 10 specimens with 0.5% Ag-Zn zeolite.

Subgroup C: 10 specimens with 1% Ag-Zn zeolite.

Subgroup D: 10 specimens with 1.5% Ag-Zn zeolite.

Total 3 plastic mold were fabricated using laser cutting machine (JL-1612, Jinan Link Manufacture and Trading Co., Ltd., China); 1 for each group of specimens. Each mold consisted of 3 plastic layers. The thickness of the top and bottom layers was 6 mm. The middle layer had a thickness of 2 mm for group 1 and group 2 molds and of 6 mm for group 3 mold.

Six holes were engraved in the middle layer of each mold. The shape of the holes was according to the specifications

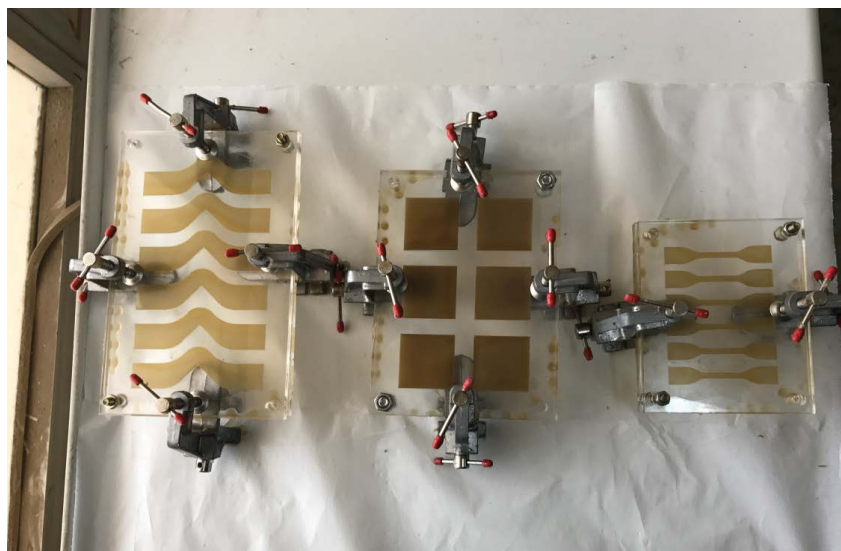
for each test. An angle test specimen without nick with dimensions in accordance with ISO 34-1:2010 was selected for tear strength test [24]. Type 2 dumb-bell specimen was chosen for tensile strength and elongation at break test according to ISO 37:2011 [25]. A 40 mm × 40 mm square test specimen was used for hardness and roughness tests in accordance with ISO 7619-1:2010 [26]. The middle plastic sheet was glued to the bottom sheet of the mold.

The mixing of silicone was done according to manufacturer's instructions. For control subgroup, part A (base) of silicone and part B (crosslinker) was weighed with a ratio of 10:1 and mixed by a vacuum mixer (Multivac 3, Degussa, Germany) for 5 minutes (Figure 1). For subgroup B, C and D, part A of the silicone and the Ag-Zn zeolite were weighed first and then mixed by vacuum mixer for 10 minutes and then part B was added and mixed for another 5 minutes [27].



**Figure 1** Mixing silicone with the vacuum mixer

The mixture was poured into the molds and the top sheet of the mold was secured with screws and nuts. The mold was then pressed by g-clamps and left at room temperature for 24 hours (Figure 2).



**Figure 2** Molding of specimens

Specimens were finished with a scalpel and stored in a light-tight container before testing. All tests were done under standard laboratory conditions, i.e. the temperature of  $23 \pm 2^\circ\text{C}$  and relative humidity of  $50\% \pm 10\%$ , and the minimum

time between vulcanization and testing was 16 hours.

Tear strength test was done by mounting the test specimen on a universal testing machine (WDW-20, Laryee Technology Co. Ltd., China) and stretching it with a speed of 500 mm/min until the break. The thickness of the specimen was measured at the area of the right angle by a digital caliper before testing. The tear strength was calculated by dividing the maximum force obtained from the universal testing machine by the thickness of the specimen.

For tensile strength, the thickness and the width of the narrow portion of the specimen were measured by a digital caliper at 3 areas; at the two ends and in the middle. The average of the 3 readings was considered as the thickness and the width of the specimen. Then the specimen was mounted on a universal testing machine and stretched at a crosshead speed of 500 mm/min until it breaks. The tensile strength was calculated using the following formula:

$$T_s = \frac{F_m}{W_t}$$

Where:

$F_m$  is the maximum force in Newton.

$W$  is the average width of the narrow portion of the sample in millimeters.

$t$  is the average thickness of the sample over the narrow portion in millimeters.

Elongation at break was calculated using an extensometer for each tensile specimen and percentage of elongation was calculated using the following formula:

$$E_b \% = \frac{L_b - L_0}{L_0} \times 100$$

Where:

$L_0$  is the initial test length in millimeters.

$L_b$  is the test length at the break in millimeters.

Shore A hardness test was conducted by making 5 markings on one side of the square specimen using a pen and a ruler. Each marking was at least 0.6 mm apart from each other and 12 mm away from the edge. A digital shore A durometer (HT-6510A, China) with a blunt indenter of the diameter of 1.25 mm was used to measure the hardness. The sample was placed on a flat and rigid surface. Then the durometer was held perpendicular to the sample surface with pressure foot parallel to the surface. The durometer was pressed firmly for 3 seconds at each marked point. The average of the 5 reading was calculated and considered the hardness number of that specimen.

Surface roughness test was done on the other side of the square specimen using a profilometer (TR 220, Beijing Time High Technology Ltd., China). The 3 readings were obtained and the average of the 3 was considered the roughness value of the specimen.

Fourier Transform Infrared Spectroscopy (FTIR) test was performed to determine whether the Ag-Zn Zeolite interacted with the silicone polymer or not. Two samples, one from control subgroup and the other from the 1.5% filler concentration subgroup, were prepared by cutting the thin flushes of the mold into 15 mm × 15 mm square with a thickness of 0.5 mm and then tested by a spectrometer (FTIR 8400S, Shimadzu, Japan).

One-way ANOVA and Tukey's HSD (honest significant difference) test was used to analyze study data using IBM SPSS software (version 24.0). A p-value > 0.05 was considered statically non-significant (N.S.), ≤ 0.05 was considered significant (S.) and < 0.01 was considered as highly significant (H.S.).

## RESULTS

### FTIR Analysis

Spectra for maxillofacial silicone before and after the addition of Ag-Zn zeolite powder are shown in Figure 3 and Figure 4 respectively. Both showed the characteristic vibration peaks for silicone at 600-688  $\text{cm}^{-1}$  assigned for Si-

Si, 711-781  $\text{cm}^{-1}$  for Si-C, 1004-1182  $\text{cm}^{-1}$  for Si-O-Si, 1400-1444  $\text{cm}^{-1}$  for  $\text{CH}_3$  and 3000-4000  $\text{cm}^{-1}$  for Si-OH and H-O-H stretching vibration. Both spectra are quite similar except for shifting for Si-Si and Si-O-Si peaks and  $\text{CH}_3$  stretching vibration in the spectrum of silicone with Ag-Zn zeolite. This shifting and stretching vibration indicated some degree of combination between the silicone matrix and Ag-Zn zeolite powder.

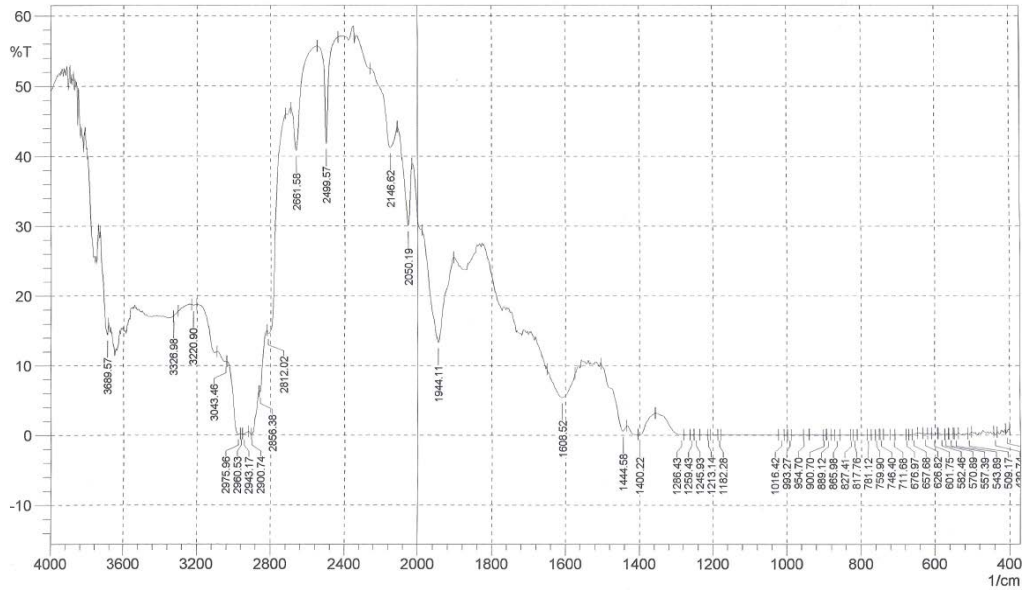


Figure 3 FTIR Spectrum of silicone without Ag-Zn zeolite

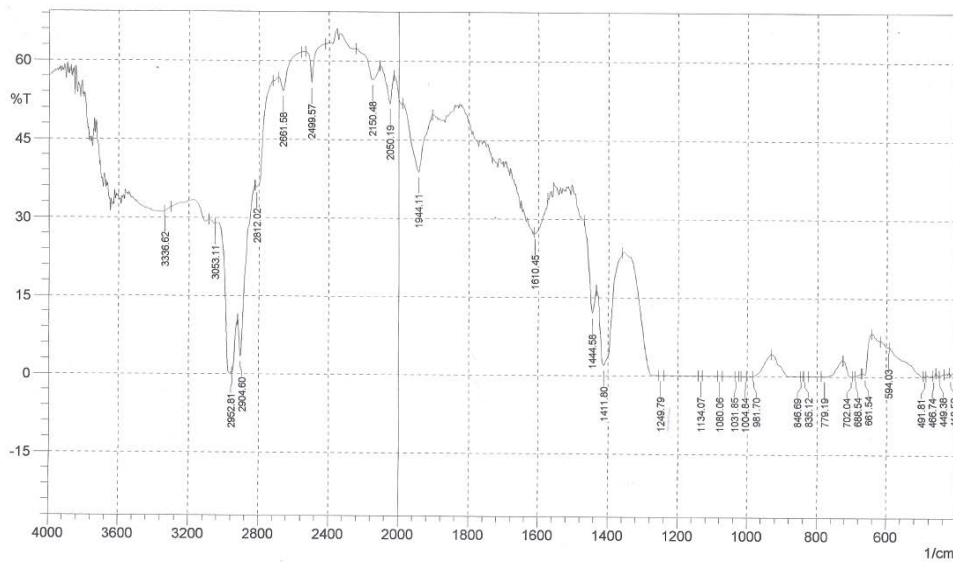


Figure 4 FTIR Spectrum of silicone with Ag-Zn zeolite

**Tear strength**

All experimental subgroups exhibited higher mean value in tear strength than that of the control subgroup. The highest tear strength mean value was subgroup C (1%) (22.57 N/mm) followed by subgroup D (1.5%) (19.18 N/mm) then by subgroup B (0.5%) (19.03 N/mm), while the mean value of control subgroup A (0%) was 14.81 N/mm. One-way ANOVA test showed a highly significant difference ( $p < 0.01$ ) between all subgroups (Table 1).

**Table 1 Descriptive statistics and one-way ANOVA of tear strength test in N/mm**

Subgroup	Descriptive statistics						ANOVA	
	N	Mean	Std. Deviation	Std. Error	Min.	Max.	F-test	p-value
A (0%)	10	14.81	0.66747	0.21107	13.33	15.81	133.827	0.000 (H.S.)
B (0.5%)	10	19.03	0.85373	0.26997	17.61	20.13		
C (1%)	10	22.57	0.81033	0.25625	21.42	24.28		
D (1.5%)	10	19.18	1.08812	0.34409	17.93	21.81		

A Post-hoc Tukey’s HSD (honest significant difference) test was conducted for multiple comparisons between all subgroups of the study. There was a highly significant difference between all subgroups ( $p < 0.01$ ) except for the difference between subgroup B (0.5%) and D (1.5%) was non-significant (Table 2).

**Table 2 Tukey’s HSD test of tear strength results for all subgroups**

Subgroups		Mean Difference	Std. Error	p-value
A	B	-4.216	0.38827	0.000 (H.S.)
	C	-7.756	0.38827	0.000 (H.S.)
	D	-4.37	0.38827	0.000 (H.S.)
B	C	-3.54	0.38827	0.000 (H.S.)
	D	-0.154	0.38827	0.979 (N.S.)
C	D	3.386	0.38827	0.000 (H.S.)

**Tensile strength**

The subgroup C (1%) showed the highest mean value (4.08 Mpa) in tensile strength followed by subgroup D (1.5%) (3.80 Mpa) and subgroup B (0.5%) (3.79 Mpa). The control subgroup A (0%) mean value was 3.63 Mpa. One-way ANOVA test showed a highly significant difference ( $p < 0.01$ ) between all subgroups (Table 3).

**Table 3 Descriptive statistics and one-way ANOVA of tensile strength test in MPa**

Subgroup	Descriptive statistics						ANOVA	
	N	Mean	Std. Deviation	Std. Error	Min.	Max.	F-test	p-value
A (0%)	10	3.63	0.25238	0.07981	3.17	3.91	11.754	0.000 (H.S.)
B (0.5%)	10	3.79	0.1401	0.0443	3.57	3.98		
C (1%)	10	4.08	0.12606	0.03986	3.9	4.25		
D (1.5%)	10	3.8	0.13517	0.04275	3.62	4.09		

Tukey’s HSD test for tensile strength is presented in Table 4. A highly significant difference was shown between subgroups (A and C), (B and C) and (C and D). Other comparisons were non-significant.

**Table 4 Tukey’s HSD test of tensile strength results for all subgroups**

Subgroups		Mean Difference	Std. Error	p-value
A	B	-0.158	0.07664	0.185 (N.S.)
	C	-0.447	0.07664	0.000 (H.S.)
	D	-0.169	0.07664	0.141 (N.S.)
B	C	-0.289	0.07664	0.003 (H.S.)
	D	-0.011	0.07664	0.999 (N.S.)
C	D	0.278	0.07664	0.005 (H.S.)

**Percentage of Elongation**

All experimental subgroups showed a lower mean value in percentage of elongation compared to that of control subgroup. The mean value of subgroup A (0%) was (342.5) while that of subgroup B (0.5%), C (1%) and D (1.5%) were (327.9, 319.9 and 312.9 respectively). One-way ANOVA test showed a highly significant difference ( $P < 0.01$ ) between all subgroups (Table 5).



**Table 5 Descriptive statistics and one-way ANOVA of the percentage of elongation test**

Subgroup	Descriptive statistics						ANOVA	
	N	Mean	Std. Deviation	Std. Error	Min.	Max.	F-test	P value
A (0%)	10	342.5	9.21653	2.91452	330	357	26.656	0.000 (H.S.)
B (0.5%)	10	327.9	6.31489	1.99694	319	338		
C (1%)	10	319.9	6.10009	1.92902	310	329		
D (1.5%)	10	312.9	8.96227	2.83412	300	327		

Tukey’s HSD test demonstrated a highly significant difference between all study subgroups except between subgroup (B and C) and (C and D) the difference was non-significant (Table 6).

**Table 6 Tukey’s HSD test of the percentage of elongation results for all subgroups**

Subgroups		Mean Difference	Std. Error	p-value
A	B	14.6	3.48106	0.001 (H.S.)
	C	22.6	3.48106	0.000 (H.S.)
	D	29.6	3.48106	0.000 (H.S.)
B	C	8.0	3.48106	0.117 (N.S.)
	D	15.0	3.48106	0.001 (H.S.)
C	D	7.0	3.48106	0.203 (N.S.)

**Shore A hardness**

All experimental subgroups showed a higher mean value in shore A hardness compared to that of control subgroup. The increase in hardness was directly proportional to the percentage of filler added. The mean value of subgroup A (0%) was (33.4) while that of subgroup B (0.5%), C (1%) and D (1.5%) were (33.69, 34.01 and 34.89 respectively). One-way ANOVA test showed a highly significant difference ( $p < 0.01$ ) between all subgroups (Table 7).

**Table 7 Descriptive statistics and one-way ANOVA of Shore a hardness test**

Subgroup	Descriptive statistics						ANOVA	
	N	Mean	Std. Deviation	Std. Error	Min.	Max.	F-test	p-value
A (0%)	10	33.4	0.60736	0.19206	32.3	34.2	15.924	0.000 (H.S)
B (0.5%)	10	33.69	0.5646	0.17854	32.8	34.5		
C (1%)	10	34.01	0.40125	0.12689	33.3	34.6		
D (1.5%)	10	34.89	0.44335	0.1402	34.1	35.6		

Tukey’s HSD test showed a highly significant difference between subgroup D and each subgroup of the study. Differences between other subgroups were non-significant (Table 8).

**Table 8 Tukey’s HSD test of Shore A hardness results for all subgroups**

Subgroups		Mean Difference	Std. Error	p-value
A	B	-0.29	0.22861	0.588 (N.S.)
	C	-0.61	0.22861	0.053 (N.S.)
	D	-1.49	0.22861	0.000 (H.S.)
B	C	-0.32	0.22861	0.508 (N.S.)
	D	-1.2	0.22861	0.000 (H.S.)
C	D	-0.88	0.22861	0.003 (H.S.)

**Surface Roughness**

All experimental subgroups showed a higher mean value in surface roughness compared to that of control subgroup. The increase in roughness was directly proportional to the percentage of filler added. The mean value of subgroup A (0%) was (0.3035  $\mu\text{m}$ ) while that of subgroup B (0.5%), C (1%) and D (1.5%) were (0.3375  $\mu\text{m}$ , 0.3953  $\mu\text{m}$  and 0.416  $\mu\text{m}$  respectively). One-way ANOVA test showed a highly significant difference ( $p < 0.01$ ) between all subgroups (Table 9).

Table 9 Descriptive statistics and one-way ANOVA of surface roughness in  $\mu\text{m}$ 

Subgroup	Descriptive Statistics						ANOVA	
	N	Mean	Std. Deviation	Std. Error	Min.	Max.	F-test	p-value
A (0%)	10	0.3035	0.0087337	0.0027618	0.289	0.316	350.817	0.000 (H.S.)
B (0.5%)	10	0.3375	0.0084492	0.0026719	0.326	0.353		
C (1%)	10	0.3953	0.0078606	0.0024857	0.385	0.411		
D (1.5%)	10	0.416	0.0098093	0.003102	0.395	0.428		

The multiple comparisons of Tukey's HSD test were highly significant between every two subgroups of the study (Table 10).

Table 10 Tukey's HSD test of surface roughness results for all subgroups

Subgroups		Mean Difference	Std. Error	p-value
A	B	-0.034	0.0039095	0.000 (H.S.)
	C	-0.0918	0.0039095	0.000 (H.S.)
	D	-0.1125	0.0039095	0.000 (H.S.)
B	C	-0.0578	0.0039095	0.000 (H.S.)
	D	-0.0785	0.0039095	0.000 (H.S.)
C	D	-0.0207	0.0039095	0.000 (H.S.)

## DISCUSSION

The null hypothesis was rejected because the addition of silver-zinc zeolite significantly affected some mechanical properties of maxillofacial silicone.

High tear strength, high tensile strength, and percentage of elongation, adequate hardness and good color stability are the most important properties of maxillofacial silicone [28]. The addition of filler is needed for achieving improvement in these mechanical properties. The reinforcement depends largely on the polymer and filler characteristic, filler loading (amount of filler) and processing conditions [29,30].

Zeolite is good reinforcing filler as it has large surface area and fine particles which leads to greater interface between the filler and silicone matrix [21]. FTIR test is considered as a qualitative and quantitative analysis of material properties [31]. The FTIR results of this study showed a chemical interaction between the added Ag-Zn zeolite and silicone. This may explain for some instance the improvement in some of the silicone mechanical properties.

Tear strength results of this study indicated that tear strength significantly increased when adding silver-zinc zeolite in amounts of 0.5%, 1% and 1.5% to the silicone. In all experimental groups, the filler interacted with the silicone matrix and associated strongly with the polymer chains thus increasing tear strength values. Tear strength of a material depends on the ability of the polymer to scatter energy at the area of the crack as tearing propagates. Small sized fillers dissipate strain energy within the matrix of the polymer, thus making it more resistant to tearing and a higher applied force is needed to completely break the polymer chains. This explains the increase in tear strength [32,33].

Tensile strength and elongation of silicone depend greatly on the crosslinking between the silicone chains [3]. Zeolite framework consists of aluminosilicate [34]. The silicate groups act as multifunctional crosslinks by the formation of bonds with silicone chains. These multifunctional crosslinks increase the overall crosslinking density of the cured silicone and make it stiffer and stronger. When the polymer is subjected to tensional forces, these crosslinks do not allow the chains to slide over each another and prevent them to break, thus increasing the tensile strength [35]. The decrease in the percentage of elongation could also be explained by increasing polymer stiffness and crosslinking density due to the incorporation of filler which formed multifunctional crosslinks and trapped entanglements. That in fact hindered and restricted the movement of the polymer chains and reduced their stretching ability [36,37].

The decrease in tear strength, tensile strength and percentage of elongation at a concentration of 1.5% zeolite could be due to the formation of filler agglomerates. These agglomerates are formed when two or more filler particle aggregates bind together by weak electrostatic forces called Van der Waals forces. These agglomerates act as stress concentration areas within the polymer matrix. When external forces are applied to the polymer, the agglomerates break and weaken the matrix leading to propagation of crack [17,38].



Shore A hardness results of this study indicated a directly proportional increase in hardness with an increase in filler concentration. This can be attributed to the fact that increasing filler concentration increases the adsorption of the polymer chain to the filler surface and increases the intermolecular forces. This leads to a rigid polymer with high elastic modulus and more resistance to permanent deformation by penetration [39].

In addition, this increase in hardness may be due to the dispersion of the zeolite particles in the silicone matrix, which increases the crosslink density, thereby leading to increased hardness [40]. Regarding the surface roughness test, the increase in roughness values as the zeolite loading increases could be caused by the variation in the microstructural characteristics of the two materials and the formation of filler agglomerates which led to a greater nodular appearance on the surface [41].

### CONCLUSION

Within the limitations of this study, it can be concluded that the addition of 1% antimicrobial Ag-Zn zeolite powder to an RTV maxillofacial silicone improved its tear and tensile strength with a non-significant increase in hardness, but there was an increase in roughness and a decrease in the percentage of elongation.

### DECLARATIONS

#### Conflict of Interest

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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