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Evaluation of Tear Strength of Graphene Incorporated Room Temperature Vulcanizing Silicones at Different Conditions: An *in vitro*Study

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Abstract

Purpose: The study aimed to evaluate the tear strength of commonly used room temperature vulcanized (RTV) silicone for maxillofacial prostheses with and without the incorporation of varying concentrations of graphene.

Materials and Method: This *in vitro* comparative study included 40 samples prepared using stainless-steel die fabricated according to the ANSI/ADA specifications no 20.and ASTM (American Standards for Testing and Material) D624divided into four groups: Group I (NGS)-maxillofacial silicone elastomer without graphene, Group II (0.5GS)-silicone with 0.5% graphene, Group III (1GS)-silicone with 1% graphene, and Group IV (2GS)-silicone with 2% graphene. Samples were prepared using a standard RTV silicone elastomer mixed according to manufacturer instructions. Graphene was incorporated in specified concentrations. Tear strength was measured using an INSTRON Universal Testing Machine, and statistical analysis was performed using ANOVA and post hoc tests.

Results: The highest mean tear strength was observed in the group with 0.5% graphene (39.99±1.15 N/mm), followed by 1% graphene (37.50±1.35 N/mm), no graphene (37.45±2.07 N/mm), and 2% graphene (33.69±1.30 N/mm). Statistically significant differences in tear strength were found among the groups ($p < 0.001$). Pairwise comparisons showed significant differences between 0.5% and 1% graphene ($p = 0.004$) and 0.5% and 2% graphene ($p < 0.001$).

Conclusion: Incorporation of graphene into RTV maxillofacial silicones initially increases tear strength up to 0.5% concentration, after which the strength decreases. The optimal concentration for enhanced tear strength is 0.5% graphene. However, the dark gray color of graphene may not be aesthetically suitable for all patients.

Keywords: Maxillofacial prosthetics, RTV silicone, Tear strength, Graphene, Mechanical properties, Nanoparticles, Prosthesis durability

Introduction

Maxillofacial prosthetics is about fixing problems in the head and neck area when parts are missing or damaged. These issues might be from birth, accidents, or surgeries. Fixing these problems is important because they can affect how the face and neck work together[1]. Advances in plastic surgery techniques are making it easier to fix these issues. The goal of these prosthesis is to make sure the face and neck look and work as they should, so the person's life is better[12].

Maxillofacial silicones are a vital component of prosthodontics, particularly in the realm of maxillofacial prosthetics. Silicone used in maxillofacial prosthesis can be categorized as Room temperatures vulcanized (RTV) & Heat vulcanized (HTV). Both room temperature vulcanized and heat cure silicone materials have been used for more than 50 years to fabricate such maxillofacial prosthesis. These silicones play a crucial role in the fabrication of prosthetic devices designed to restore or replace lost facial structures. Silicone prosthetic materials are very well accepted by patient due to their softness and aesthetics results. But the expected half-life of maxillofacial prosthesis on an average is approximately six months and degradation of physical and color properties of silicone maxillofacial prosthesis is the most common reason for refabrication of prosthesis[3].

Silicone prosthetic materials have some limitations in the form of inferior physical and mechanical properties such as tear strength affecting longevity, colour stability and subsequent deteriorating aesthetics. Most maxillofacial elastomers perform well initially, however as time passes, deterioration associated either with degradation of mechanical properties or changes in appearances occur[4, 5].

Nanoparticles are extremely small particles, typically less than 100 nanometres in size. Graphene nanoparticles are ultra-thin, single-layered carbon structures known for their exceptional mechanical, thermaland electrical properties, making them valuable for enhancing materials like RTV silicone in various applications.

Graphene, the wonder material comprising a single layer of carbon atoms arranged in a two-dimensional honeycomb lattice, has been captivating researchers across various fields for its remarkable properties. In dentistry, graphene holds significant promise for revolutionizing numerous aspects of dental care, from diagnostics to treatment[6-12]. Various Studies that incorporated graphene compounds into dental polymers to positively alter their mechanical properties are available in literature[13-25].

Margins of maxillofacial prosthesis are usually very thin and tend to tear over a period of time, especially with the use of adhesives. Moreover, the color of RTV silicones deteriorates with time. So, addition of graphene may affect the tear strength of RTV maxillofacial prosthetic silicones too. The invention of graphene incorporated RTV silicones may prove to be a boon to debilitated patients leading to longer shelf life of prosthesis. The null hypothesis of this study is that there is no significant difference in the tear strength of RTV Silicones following incorporation of with or without various concentrations of graphene.

Materials and methods

The study was comparative *in vitro* research to evaluate the tear strength of commonly used RTV silicone used for maxillofacial prosthesis with a total sample size of 40. The sample size was calculated by using G*Power 3.1.9.7 software, keeping the significance level at 0.05 and the power of the study at 0.80. The effect size came to be 0.58 with a total sample size of 40 samples which were divided into 4 groups. The description of each groups is as follows: (Table 1).

TABLE 1:SAMPLES AND GROUPS DESCRIPTION

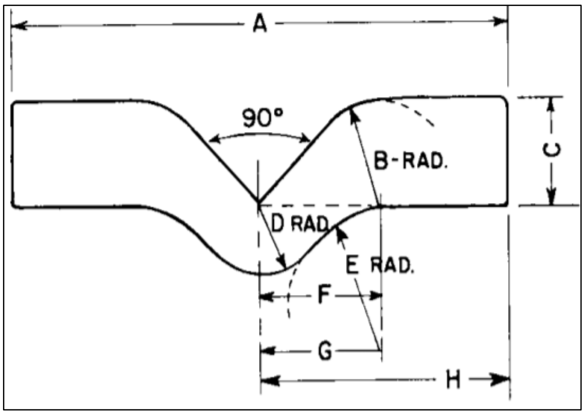
Group I (NGS)	Maxillofacial silicone elastomer without addition of Graphene (control group)
Group II (0.5GS)	Maxillofacial silicone elastomer incorporated with 0.5% Graphene
Group III (1GS)	Maxillofacial silicone elastomer incorporated with 1% Graphene
Group IV (2GS)	Maxillofacial silicone elastomer incorporated with 2% Graphene

In this study, a stainless-steel die was prepared according to the ANSI/ADA specifications no 20.[17]and ASTM (American Standards for Testing and Material) D624[18] as shown in Figure 1 and 2.

The specimens were fabricated using M511 silicone elastomer (TECTOVENT), as shown in Fig.3. This silicone is comprised of two parts: a base (Part A) and a catalyst (Part B), which were mixed in 10:1 ratio by weight according to manufacture instruction. For the control group, only base and catalyst were taken in the manufacturer-recommended ratio without addition of Graphene. For study groups Graphene was incorporated according to different concentration (0.5%, 1%, 2%) pertaining to different groups as shown in Fig.4. Graphene was measured using a digital precision scale. These components were thoroughly mixed on a glass slab using a stainless-steel spatula to ensure a homogeneous mixture as shown in Fig.5.Each sample was made using master mold by placing material in the die and material was removed after 24hr according to manufacturers' instructions as shown in Fig.6&7.The prepared samples were divided into four groups as shown in Fig.8,9,10,11and measured for tear test. Tear test was measured by the universal testing machine (INSTRON Universal Testing Machine) as shown in Fig.12. Constant force was maintained for the separation of jaws at 500 ± 5 mm/min. Force was applied until the test specimen is completely torn. After the maximum tearing force was obtained, tear strength was measured using the formula:

Ts= F/d in N/mm units.

Where,
Ts = Tear strength.
F = Maximum force.
d = Thickness.



Dimensions of specimen,
A = 102 mmB = 19 mmC = 19 mmD = 12.7 mm
E = 25 mmF = 27 mmG = 28 mmH = 51 mm

FIG1:MEASUREMENTS OF SAMPLES FOR TEAR STRENGTH

FIG2: STAINLESS STEEL DIE FOR SAMPLE PREPARATION FOR EVALUATION OF TEAR STRENGTH

FIG3: MEDICAL GRADE SILICONE ELASTOMER

FIG4: GRAPHENE

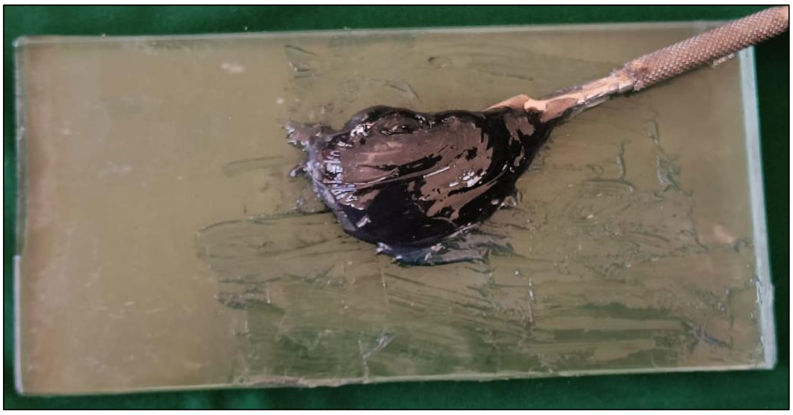


FIG5: MIXING OF ALL BASE, CATALYST AND GRAPHENE

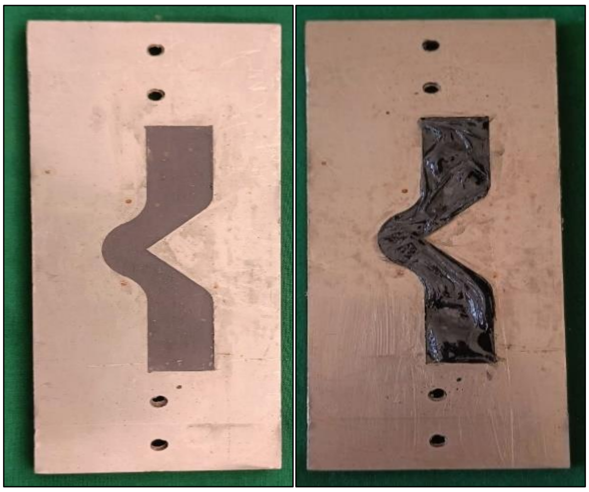


FIG 6 & 7: FABRICATION OF SAMPLE FOR EVALUATION OF TEAR STRENGTH

FIG8: GROUP I (CONTROL GROUP) **FIG9:**GROUP III (1% GRAPHENE)

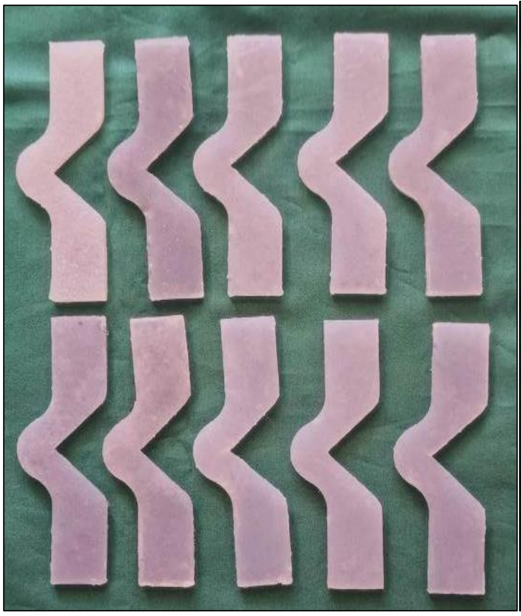



FIG10: GROUP II (0.5% GRAPHENE) **FIG11:**GROUP IV (2% GRAPHENE)

FIG12: INSTRON UNIVERSAL TESTING MACHINES

Statistical analysis was performed using ANOVA test and Post hoc analysis using SPSS (Statistics software Version 20.0; IBM Corp.) Level of Significance $p \leq 0.05$ was set.

Result

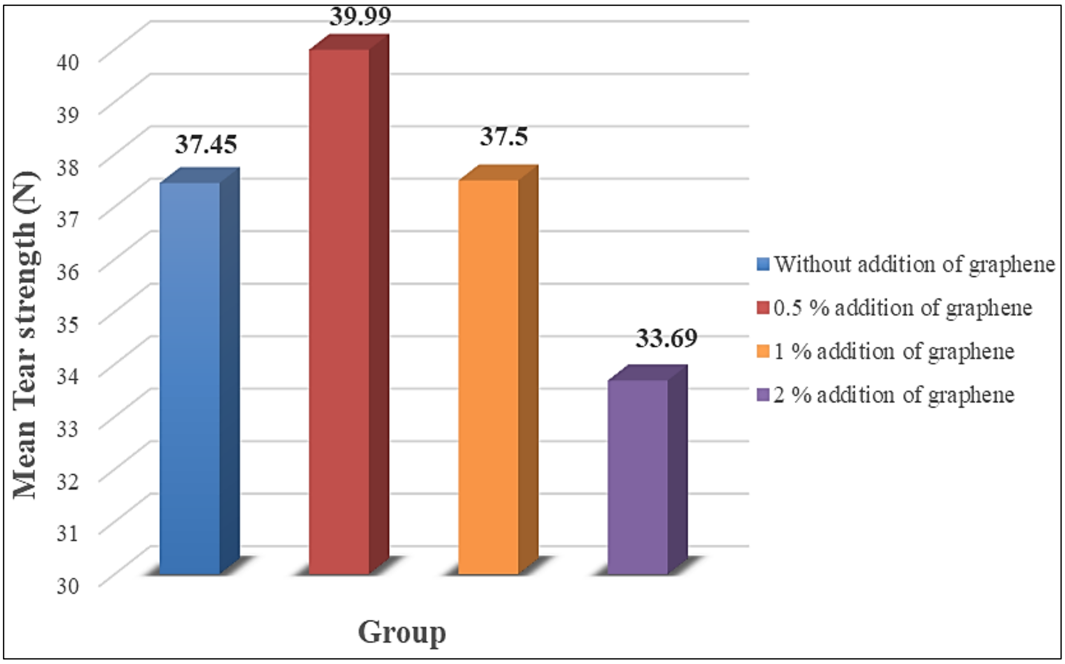
The mean Tear strength score was highest in study groups with 0.5% addition of graphene (39.99±1.15), followed by 1% addition of graphene (37.50±1.35), Without addition of graphene (37.45±2.07) and 2% addition of graphene (33.69±1.30) respectively. A statistically significant difference was observed in mean Tear strength score among various groups.(p value <0.001) **(Table 1 Graph 1).**

TABLE 1: DISTRIBUTION OF STUDY GROUPS BASED ON MEAN TEAR STRENGTH AND WITH ORWITHOUT ADDITION OF DIFFERENT CONCENTRATION OF GRAPHENE

Group	**Number**	**Tear Strength (N/mm)**		**P Value**
		Mean	**SD**	
Group I (NGS)	10	37.45	2.07	<0.001*
Group II (0.5GS)	10	39.99	1.15	
Group III (1GS)	10	37.50	1.35	
Group IV	10	33.69	1.30	

(2GS)				

Level of significance p value ≤0.05, *Significant, **Non-significant



GRAPH 1: DISTRIBUTION OF STUDY GROUPS BASED ON MEAN TEAR STRENGTH AND WITH ORWITHOUT ADDITION OF GRAPHENE

A statistically significant difference was observed in comparison between without addition of graphene and 0.5% addition of graphene (p value = 0.003) and without addition of graphene and 2% addition of graphene (p value <0.001). while statistically non-significant difference was observed in comparison between without addition of graphene and 1% addition of graphene (p value = 1.000) **(Table 2).**

TABLE 2: DISTRIBUTION OF STUDY GROUPS BASED ON TEAR STRENGTH AND WITH OR WITHOUT ADDITION OF GRAPHENE (PAIR WISE COMPARISON)

Tear Strength (N/mm)			
Group		**Difference**	**P Value**
Group I (NGS)	Group II (0.5GS)	-2.54	0.003*
	Group III (1GS)	-0.05	1.000**
	Group IV (2GS)	3.76	<0.001*

Level of significance p value ≤0.05, *Significant, **Non-significant

A statistically significant difference was observed in comparison between 0.5% addition of graphene and 1% addition of graphene (p value = 0.004) and 0.5% addition of graphene and 2% addition of graphene (p value <0.001) **(Table 3).**

TABLE 3: DISTRIBUTION OF STUDY PARTICIPANTS BASED ON TEAR STRENGTH AND WITH ADDITION OFDIFFERENT CONCENTRATION OF GRAPHENE (PAIR WISE COMPARISON)

Tear Strength (N/mm)			
GROUP		**Difference**	**P Value**
Group II (0.5GS)	Group III (1GS)	2.49	0.004*
	Group IV	6.30	<0.001*

	(2GS)		

Level of significance p value ≤0.05, *Significant, **Non-significant

A statistically significant difference was observed in comparison between 1% addition of graphene and 2% addition of graphene (p value < 0.001) **(Table 4).**

TABLE 4: DISTRIBUTION OF STUDY GROUPS BASED ON TEAR STRENGTH AND WITH ADDITION OFDIFFERENT CONCENTRATION OF GRAPHENE (PAIR WISE COMPARISON)

Tear Strength (N/mm)			
Group		**Difference**	**P Value**
Group III (1GS)	Group IV (2GS)	3.81	<0.001*

Level of significance p value ≤0.05, *Significant, **Non-significant

Discussion

The current study rejects the null hypothesis, as it identified a significant difference in tear strength outcomes following incorporation of with or without various concentration of graphene. The greatest tear strength was observed with addition of 0.5% of Graphene, 1% of Graphene, followed by without addition of Graphene and least tear strength by 2% of Graphene. The mean Tear strength score was highest in study groupswith 0.5% addition of graphene (39.99±1.15), followed by 1% addition of graphene (37.50±1.35), Without addition of graphene (37.45±2.07) and 2% addition of graphene (33.69±1.30) respectively. A statistically significant difference was observed in mean Tear strength score among various group. (p value <0.001) **(Table 1 Graph 1).**

Maximum Tear strength:0.5%addition of Graphene > 1% addition of Graphene ≈ Without addition of Graphene > 2% addition of Graphene

Nobrega *et al.*[26] conducted a study on effects of adding nanoparticles on the hardness, tear strength, and permanent deformation of facial silicone subjected to accelerated aging. Results indicated that nanoparticles influenced the properties of the silicone, with a decrease in hardness values and variations observed in tear strength and permanent deformation. Specifically, the addition of $BaSO_4$ resulted in the highest tear strength, while the 1% ZnO group without oil paint exhibited the lowest permanent deformation values. So, the study conducted by Nobrega *et al.*[26]supports the present study.

N.K. Sonnahalli and R. Chowdhary[27]investigated the effects of incorporating silver nanoparticles into maxillofacial silicone elastomer material. They found that adding silver nanoparticles at a concentration of 20 ppm resulted in decreased hardness of the silicone elastomer. However, this addition did not significantly affect tear strength or color stability. These findings highlight the potential for manipulating the physical and mechanical properties of silicone elastomers through nanoparticle incorporation, offering insights for improving material performance in various applications, particularly in the field of maxillofacial prosthetics. However, it should be noted that the findings of this study are not consistent with the results of a separate study on graphene incorporation, which reported an initial increase in tear strength followed by a decrease beyond a certain concentration as in present study. So, the study conducted by N.K. Sonnahalli and R. Chowdhary[27]does not support this present study.

Hussein I.E. *et al.*[28]investigated the effects of adding nanosized Zirconium Oxide (ZrO_2) to maxillofacial silicone elastomer. Results indicated a significant increase in tear strength and tensile strength with 1% and 1.5% ZrO_2 concentrations, while hardness remained within standard limits. However, agglomeration was observed at higher concentrations, suggesting an optimal balance for improved mechanical properties in maxillofacial silicone materials. So, the study conducted by Hussein I. E. *et al.*[28]Supports the present study.

The study conducted by Hussein IE and Hasan RH[29]assessed the different concentrations of (ZrO_2) nanoparticles on tear strength in maxillofacial silicone material. Results indicated a significant increase in tear strength with 1% and 1.5% ZrO_2 concentrations compared to the control group. However, no significant difference was observed between the control group and the 0.5% or 2% ZrO_2 groups. These findings suggest that incorporating ZrO_2 nanoparticles at specific concentrations can effectively enhance tear strength in maxillofacial silicone materials. The study conducted by Hussein IE and Hasan RH[29]obtained similar results to our present study.

Karaman *et al.*[30]investigated the effects of TiO_2, SiO_2, and ZnO nanoparticles on silicone elastomer used in maxillofacial prostheses. Results showed significant improvements in tensile strength, elongation percentage, tear strength, and hardness with nanoparticle additions. These findings suggest that incorporating nanoparticles can effectively enhance the mechanical properties of maxillofacial prostheses. So, the study was conducted by Karaman *et al.*[30] Supported the present study.

Ashika Bk *et al.*[31]conducted research on the tear strength, water absorption, and percentage elongation of a maxillofacial silicone elastomer with and without nanoparticle incorporation. The results revealed a significant decrease in water absorption for both the silver and titanium dioxide nanoparticle groups, along with improved mechanical properties compared to the control group. This highlights the potential benefits of incorporating nanoparticles in maxillofacial silicone elastomers. So, the study was conducted by Ashika Bk *et al.*[31]supports the present study.

Many previous studies evaluated that mechanical properties of medical grade silicon increases by incorporations of nanoparticles[32-37].

Conclusions

Within the confines of the study's limitations, the following conclusions can be inferred:

1. The tear strength of maxillofacial silicones initially increases with the addition of grapheneup to a certain concentration, but then decreases thereafter.
2. The color of graphene, being dark gray, may not be suitable for individuals with fair skin tones seeking maxillofacial prostheses.

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