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Color Stability of Graphene Oxide Incorporated Room Temperature Vulcanizing Silicone At Different Concentrations: An In Vitro Study

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ABSTRACT

Purpose: *Incorporating graphene oxide has been shown to enhance the mechanical and physical properties of specific materials including resin, whereas change in the properties of silicone elastomers is yet to be evaluated.*

Materials and method: *This in vitro comparative study involved 30 samples fabricated using a stainless-steel die designed according to specific dimensions. The samples were divided into three groups: Group I (Sil 0.5-G) with 0.5% graphene oxide in silicone elastomer, Group II (Sil 1-G) with 1% graphene oxide in silicone elastomer, and Group III (Sil 2-G) with 2% graphene oxide in silicone elastomer. All samples were made from a standard RTV silicone elastomer, mixed as per the manufacturer's guidelines, with graphene added at the designated concentrations. The specimens were exposed to artificial aging chamber. Before and after aging, the spectrophotometer reading was measured and the data were examined through statistical analysis using a one-way ANOVA, followed by additional post hoc tests for further evaluation.*

Results: *Delta E values were significantly higher in the group containing 0.5% graphene oxide compared to the group with 1% graphene oxide ($p < 0.0001$). Similarly, a significant increase in delta E values was observed in the group containing 2% graphene oxide compared to the group with 1% graphene oxide ($p < 0.0001$). Statistical analysis revealed no significant differences between the groups containing 0.5% and 2% graphene oxide ($p = 0.1021$).*

Conclusion: *After the addition of graphene oxide to medical-grade silicone, the least level of color variation was observed at a 1% concentration, suggesting it as the most effective level.*

Keywords: *color, graphene oxide, maxillofacial prosthesis, silicone elastomers, time passage*

INTRODUCTION

Nanotechnology has expanded its arms in the field of medicine with its application in drug composition, physiochemistry, tissue engineering and biomedical implants. In the field of dentistry also certain nanoparticles are incorporated into various materials to enhance their biological, physical and mechanical properties.[1] Nanoparticles like graphene oxide, Ag, Au, Fe are used in various materials in dentistry. Graphene oxide is also utilized in resin material and effects of its incorporation on their mechanical and physical properties were compared. Effect of incorporation of various concentration of graphene oxide in silicone and its effect on tear strength have also been documented but its effect on color stability is yet to be tested.[2]

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In clinical practice, maxillofacial prostheses are utilized to reconstruct anatomical defects resulting from trauma, disease, or congenital conditions. Unlike other intraoral prosthesis, they are usually fabricated from silicone elastomers due to natural look and accurate fit. Nevertheless, a major limitation of these materials is their tendency to undergo color changes over time.[3]

Achieving a natural look relies on both realistic sculpting and effective color matching, with the latter being one of the most difficult aspects of facial prosthesis production. By employing objective color selection methods, such as digital color selection devices, researchers can minimize errors that may arise from subjective assessments. Nevertheless, silicone elastomers are often colored using traditional subjective techniques due to lower associated costs.[4,5]

Addressing the issue of color stability in maxillofacial silicone elastomers over time presents a significant challenge, as this color alteration affects the longevity of the prosthesis. While satisfactory aesthetic outcomes can be obtained with these elastomers, they typically require replacement every 1 to 2 years due to colour fading resulting from cleansing agents, exposure to sunlight, bodily fluids, and staining linked to everyday usage.[6] Research is currently focused on the addition of nano-particles of oxides, to improve the physical and mechanical properties.[7] However, the addition of these particles on color variation don't have enough literature to support it.

This study aims to determine the effect of incorporation of graphene oxide nanoparticles on the color stability of maxillofacial silicone elastomers.

Null hypothesis of this study is that the presence of graphene oxide at various concentrations does not significantly alter the color stability of RTV silicones.

MATERIALS AND METHODS

This in vitro study compared the color stability of commonly utilized RTV silicone materials for maxillofacial prostheses, using a total of 30 samples. The sample size was determined through a calculation designed to meet the requirements for one-way ANOVA analysis, considering an effect size of 0.9 (unit for delta E), a statistical power of 80% (0.8), and a significance level (α) of 0.05. The initial calculation indicated that a sample size of 16 would achieve a statistical power of 0.8231. However, to ensure a balanced design across three experimental groups, a final sample size of 30 participants (10 per group) was utilized. The description of each group is as follows: (Table 1).

Table 1: Samples and groups description

Group I (Sil-0.5G)	0.5% concentration graphene oxide
Group II (Sil-1G)	1% concentration graphene oxide
Group III (Sil-2G)	2% concentration of graphene oxide

In this study, a stainless-steel die was prepared according to the specifications with diameter of 20 mm as shown in Figure 1.

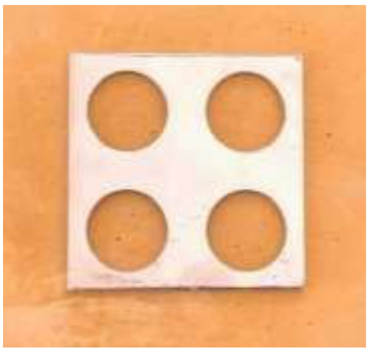


Fig 1: Stainless steel die for sample preparation

The test specimens were fabricated using M511 silicone elastomer (TECTOVENT), as illustrated in Figure 2. This elastomer consists of two components: Base and catalyst, were mixed in a 10:1 ratio by weight, following the manufacturer's guidelines. For the experimental groups, graphene nanopowder was incorporated into the silicone at concentrations of 0.5%, 1%, and 2% by weight, corresponding to the respective study groups, as shown in Figure 3.

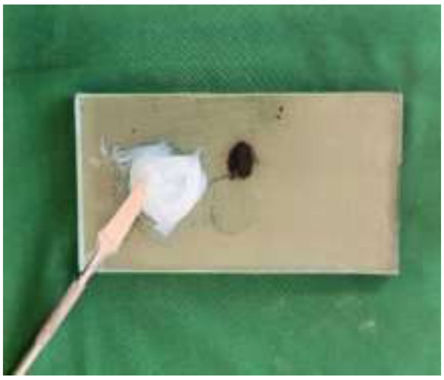
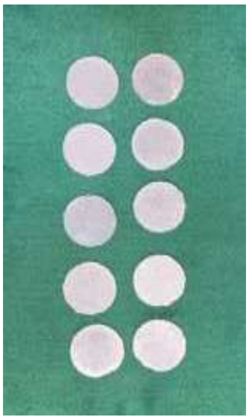


Fig. 2: Medical grade elastomer **Fig 3**: Graphene oxide **Fig 4**: Dispensing

Graphene was weighed using a digital precision balance, and all components were thoroughly mixed on a glass slab with a stainless-steel spatula to ensure uniform dispersion, as depicted in Figure 4. The mixture was then transferred into master molds, and the samples were permitted to set for 24 hours at ambient temperature, following the manufacturer's guidelines.

Fig 4: Dispensing and manipulating base, catalyst and graphene oxide powder

After categorizing the samples into 3 groups as illustrated in figures 5,6 and 7, the samples were placed in artificial aging chamber (QSUN instrument) as per BWS standard 4, for fading change in color 3 gray scale and one light cycle was applied according to ISO 105 BO2. Surface of each specimen was exposed to irradiance at 1.10 w/m^2 at 420 nm, a black panel temperature of 63° Celsius and chamber temperature of 43° Celsius for 16 hours. Approximately 63.3 KJ/m^2 energy was applied. Before and after aging, the spectrophotometer reading was measured and mean of each group was calculated. The mean color difference(ΔE) was calculated.



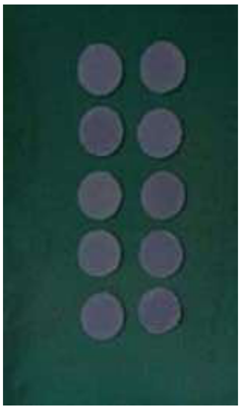


Fig 5: Group I (SIL 0.5g) Fig 6: Group II (SIL 1g) Fig 7: Group III (SIL 2g)

Statistical analysis was done using one-way ANOVA and post-hoc using GraphPad Prism (version 9.4.1), and Dunnett's test was applied for post hoc multiple comparisons. The normality of the data distribution was assessed using the Shapiro-Wilk test.

RESULTS

Post hoc analysis using Dunnett's test demonstrated that delta E values were significantly higher in the group containing 0.5% graphene compared to the group with 1% graphene ($p < 0.0001$) as shown in Table 2. Similarly, a significant increase in delta E values was observed in the group containing 2% graphene compared to the group with 1% graphene ($p < 0.0001$) as shown in Table 3.

TABLE 2: ΔE values for Group I and Group II

Color stability (Delta E values)		
Group I	Group II	P Value
3.22	2.57	<0.0001*

*Statistically significant

TABLE 3: ΔE values for Group II and Group III

Color stability (ΔE values)		
Group II	Group III	P Value
2.57	3.52	<0.0001*

*Statistically significant

TABLE 4: ΔE values for Group I and Group III

Color stability (Delta E values)		
Group I	Group III	P Value
3.22	3.52	0.1021

As shown in Fig 8, the statistical difference can be seen between the Group I and II, Group II and Group III and there is no significance seen among Group I and Group III.

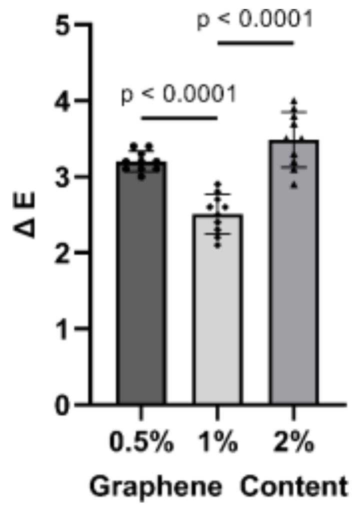


Fig 8: ΔE values for color stability for all the three groups

Though, no statistically significant difference was found between the groups containing 0.5% and 2% graphene ($p = 0.1021$) as shown in Table 4. These findings highlight the influence of graphene concentration on delta E values, with significant differences observed between specific concentrations but not across all groups.

DISCUSSION

This study assesses the color stability for the maxillofacial prosthesis, RTV (Room temperature vulcanized) silicone elastomers following the incorporation of different concentrations of graphene oxide (GO). The results clearly demonstrated that a 1% concentration of GO provided significantly better color stability (i.e., lowest ΔE values) compared to both lower (0.5%) and higher (2%) concentrations, with statistically significant differences observed between the 1% group and the others ($p < 0.0001$).

Our findings align with previous reports emphasizing the importance of optimal nanoparticle concentration in enhancing the performance of silicone materials. The study by Sonnahalli and Chowdhary (2020)[8] suggested that nanoparticles, including graphene oxide, can improve both mechanical and color stability properties of silicone elastomers when incorporated in appropriate proportions. However, they also cautioned that excessive nanoparticle loading may lead to particle agglomeration, reducing uniform dispersion and adversely affecting material performance.

Gupta et al. (2021)[9], in a systematic review, found similar outcomes with TiO_2 nanoparticles, where moderate concentrations improved color stability under environmental aging, while higher doses did not proportionally enhance the effect and sometimes even led to color instability due to optical interference or clustering effects. Although TiO_2 and GO differ chemically, their interaction with the silicone matrix and resulting light-reflective properties appear to follow a similar trend, suggesting that nanoparticle concentration plays a critical role regardless of type.

This concentration-dependent behavior mirrors findings reported by Han et al. (2010)[10], Their study investigated how varying concentrations (1%, 2%, and 2.5%) of nano-oxides,specifically TiO_2, ZnO, and CeO_2 affect the color stability of pigmented maxillofacial silicone elastomers, the research indicated that incorporating 1% CeO_2 and 2–2.5% TiO_2 resulted in the minimal color alteration especially in mixed- pigment groups, while yellow pigments showed the greatest susceptibility to discoloration. Notably, higher concentrations of CeO_2 (2%) exceeded the perceptibility threshold, confirming that excessive nanoparticle loading can be detrimental rather than beneficial due to agglomeration or optical oversaturation.

Graphene's utility has been highlighted in other domains of dental biomaterials as well. Bacali et al. (2019)[11] demonstrated improved physico-mechanical properties, including water resistance and tensile strength, when graphene-silver nanoparticles were incorporated into PMMA denture bases. Although the matrix material differs (PMMA vs. RTV silicone), the study complements our findings by underscoring graphene's functional versatility and biocompatibility across prosthodontic materials.

The present study's results add to the growing body of literature suggesting that the addition of nanoparticles like GO, when optimized for concentration, can significantly reduce the degradation- related color changes observed in long-term prosthesis use. This is especially critical for maxillofacial prostheses, where aesthetic longevity is paramount. Unlike conventional subjective coloring methods, spectrophotometric evaluation, as used in our study, ensures objective assessment and minimizes user bias.

CONCLUSION

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

1. The color stability of RTV maxillofacial silicone elastomer was significantly affected by the concentration of incorporated graphene oxide.
2. The group with 1% graphene oxide exhibited the lowest ΔE values, indicating the best color stability* among the tested concentrations.
3. 0.5% graphene oxide was less effective in minimizing color change, likely due to insufficient UV shielding capability.
4. 2% graphene oxide resulted in increased color change, possibly due to nanoparticle agglomeration and light scattering within the silicone matrix.
5. The dark gray coloration of graphene oxide may influence the final hue of the prosthetic material and should be considered when fabricating prostheses for individuals with lighter skin tones.

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