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Original Article

Dentistry Section

# Evaluating the Peel Bond Strength of Polymethylmethacrylate and Maxillofacial Silicone Elastomer Reinforced with Titanium Oxide Nanoparticles: A Comparative In-vitro Study

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## ABSTRACT

**Introduction:** Maxillofacial prostheses typically last around six months. The facial prostheses are usually provided with an acrylic substructure. This aids in enhanced retention and stability of silicone material. For good results acrylic and silicone materials must bond well with each other.

**Aim**: The aim of the present study was to evaluate and compare the Peel Bond Strength (PBS) of Polymethylmethacrylate (PMMA) acrylic resin and maxillofacial silicone elastomer reinforced with Titanium oxide ($TiO_2$) nanoparticles.

**Materials and Methods**: The present comparative in-vitro study was conducted in the Department of the Prosthodontics and Crown and Bridge in Manubhai Patel Dental College and Hospital, Vadodara, Gujarat, India. for a time period of six months from February to July in the year 2022. A total of 64 rectangular shaped maxillofacial silicone elastomer specimens with well-formed shaped and no visual irregularity were equally divided into two groups. group A silicone was without $TiO_2$ nanoparticles and group B silicone was added with $TiO_2$ nanoparticles. group A was further subdivided into A1 (PMMA acrylic resin and Silicone elastomer bonded with primer) and A2 (PMMA acrylic resin with retentive holes and Silicone elastomer) with 16 specimens in each subgroup. Similarly, group B was subdivided into B1 (PMMA acrylic resin and silicone elastomer reinforced with $TiO_2$ bonded with primer) and B2 (PMMA acrylic resin with retentive holes & Silicone elastomer reinforced with $TiO_2$) with 16 specimens in each subgroup. The acrylic and silicone specimens were bonded together with primer in subgroup A1 and B1 and with retentive holes in A2 and B2. The PBS was assessed through a 180° peel test on a universal testing machine and the mode of failure between the two materials was visually inspected. The data was statistically analysed using Independent t-test and one-way Analysis of Variance (ANOVA) (p=0.05).

**Results:** The difference between A1 and A2 was statistically significant (p=0.05). However, PBS values in other subgroups pairs (A1-B1; A2-B2; B1-B2) did not show a statistical significance. Tests indicated that specimens bonded with primer A-330 Gold platinum experienced adhesive bond failure.

**Conclusion:** Within the limitations of this study, retentive holes offer significantly stronger PBS between PMMA acrylic resin and maxillofacial silicone compared to primer alone. However, incorporating nanoparticles into maxillofacial silicone does not enhance the PBS between the given two materials.


**Keywords:** Adhesive primer, Bond strength, Maxillofacial prosthesis, Retentive holes

## INTRODUCTION

Maxillofacial prostheses are used to rehabilitate the missing facial deformities either from congenital defects or acquired defects [1]. Success of these silicone prostheses is dependent on retention, stability and proper aesthetics of these prostheses [2]. Starting from being seated in the undercuts, maxillofacial prosthesis has come a long way. Maxillofacial implants have opened new avenues for the maxillofacial prosthesis. Lifelike maxillofacial silicone can be made to reconstruct extraoral defects like nose, auricular, orbital and facial prosthesis. Three-dimensional printing technology or the prosthetic components has eliminated the conventional laboratory steps and reduced the number of stages of the fabrication of a silicone and acrylic prosthesis [3]. Retention for maxillofacial prosthesis can be obtained by retentive matrix, implants and/or both. Retentive matrix is fabricated from various acrylic resin materials to which silicone elastomer is attached.

To maintain the retention of prosthesis for a longer time, the bonding between the 2 substrate should be adequate to sustain the forces of removal. The bond between PMMA and silicone elastomer can be achieved by mechanical (retentive holes), micromechanical (scratches), molecular (primer and adhesive) means [4]. However, continuous usage renders the prosthesis to tensile forces which are peeling in nature. This might lead to tearing of the silicone while pulling it apart. Thus, it is imperative to examine the prosthesis under peeling forces to evaluate the bond strength between acrylic and the maxillofacial silicon [5].

Advancement in Nanotechnology has led to enhancement in mechanical properties of various dental materials. This can be attributed to the fact that nano-oxide particles are rigid and have higher shear modulus. The commonly used silicone elastomers have low tear strength and tensile strength which makes edges of the prosthesis susceptible to tear easily. The addition of nanoparticles improves the tear strength, tensile strength, and percentage elongation, thereby increasing the longevity of the prosthesis. These nano-oxide particles enhance the mechanical properties by diffusing into the silicone elastomer but at a proper level; as they have high surface energy and chemical reactivity [6-8].

The study on evaluation of PBS is generally focused on the efficacy of primers. A few suggest enhanced result when primer was used with retentive holes on acrylic sample [5]. Thus, the present study was undertaken with the aim of evaluating the PBS of PMMA acrylic resin and maxillofacial silicone elastomer and that of PMMA resin and maxillofacial silicone elastomer reinforced with $TiO_2$ nano particles. The primary objectives of the study were to evaluate and compare the PBS between PMMA acrylic resin and Silicone elastomer with primer and retentive holes in both the groups. The secondary objective was to evaluate the type of bond failure between the resin and the elastomer after peeling.

The null hypothesis considered was, there would be no statistically significant difference in the PBS of conventional and nano particles reinforced maxillofacial silicone elastomer. The alternative hypothesis suggested states that the PBS value will be more in the subgroup with retentive holes.

## MATERIALS AND METHODS

The present comparative in-vitro study was conducted in Department of the Prosthodontics and Crown & Bridge in MP Dental College and Hospital, Vadodara, Gujarat, India, for a time period of six months from February to July in the year 2022. The IEC approval with no. IEC/MPDC_215/PROSTHO-30/21 was obtained to commence the study.

**Inclusion criteria:**

- Well-formed and polished acrylic blocks of the given dimensions.
- Well-formed silicone blocks of the given dimensions.

**Exclusion criteria:**

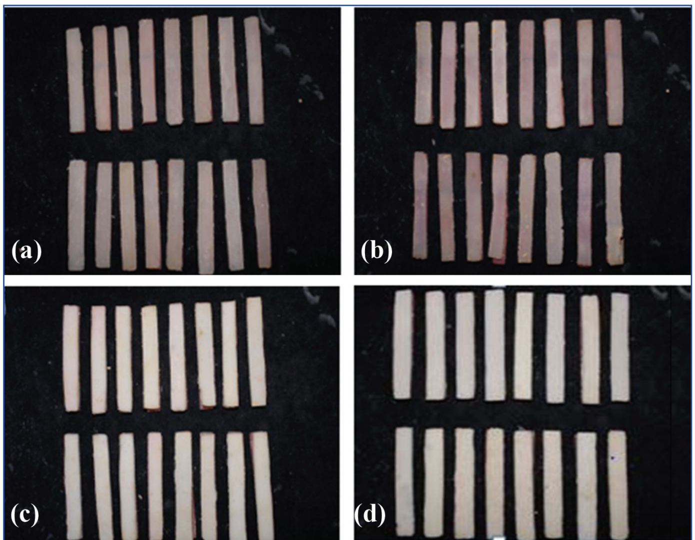
- Rough and irregular blocks of acrylic and silicone with altered dimensions.
- Acrylic blocks with porosities.

### Study Procedure

**Mixing of the Nanoparticles with Maxillofacial Silicone Elastomer:**

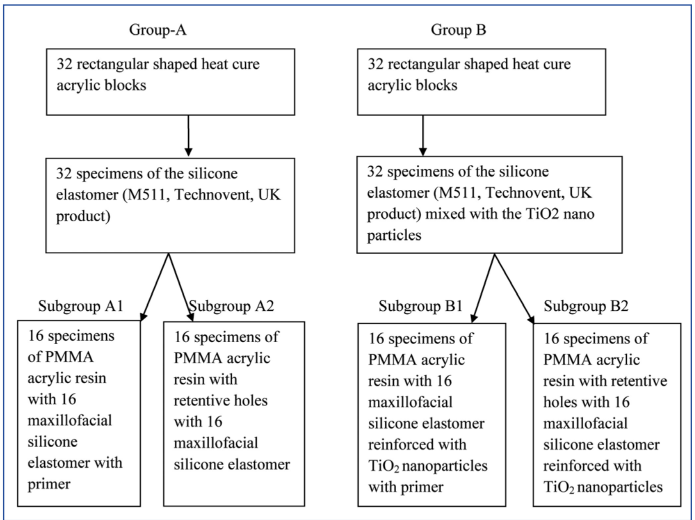
$TiO_2$ nanoparticles of 2% concentration by weight (10 gm) were incorporated into the maxillofacial silicone elastomer (Cosmesil silicone, Technovent, UK) by hand using a wooden tongue depressor mixed in a vacuum mixerfor 15 minutes to ensure a homogenous bubble free mix. The jar was then left for five minutes to allow settling of the mixed silicone [6].

**Specimen Fabrication:** A total 64 rectangular shaped heat cure acrylic blocks (Trevalon H; Dentsply, Mumbai, India) of dimensions 75 mm×10 mm×3 mm [Table/Fig-1] were fabricated by compression moulding technique [5]. A total of 32 specimens of the silicone elastomer (M511, Technovent, UK product) were also fabricated.



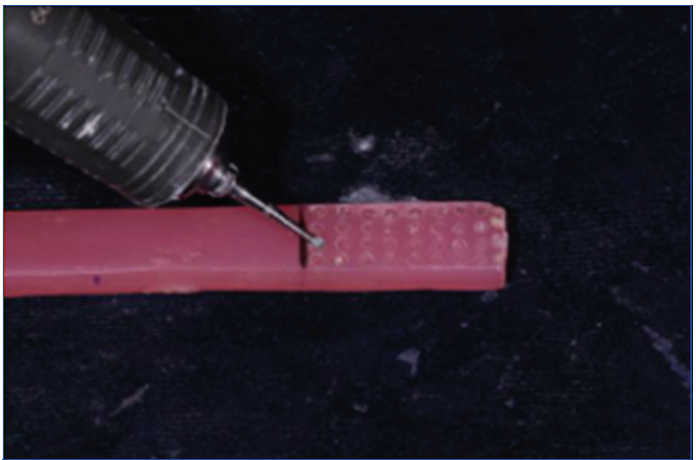

**[Table/Fig-1]:** (a) PMMA acrylic resin with primer (b) PMMA acrylic resin with undercut (c)Maxillofacial silicone elastomer without $TiO_2$ nanoparticles (d) Maxillofacial silicone elastomer reinforced with $TiO_2$ nanoparticles.

Remaining 32 samples were fabricated with the silicone elastomer mixed with the $TiO_2$ nano-particles. Distribution of samples in group and subgroup is explained in [Table/Fig-2].




**[Table/Fig-2]:** Sample distribution.

In subgroup A1 and B1 the adhesion between the resin and silicone material was achieved by a maxillofacial silicone primer (A 330-G). In subgroup A2 and B2 the adhesion between resin and silicone material was achieved by retentive holes on the resin block. These retentive holes were made using a tungsten carbide round bur (no.4) on a micro motor. The holes were 1.5 mm in diameter and 0.5 mm in depth [Table/Fig-3].



**[Table/Fig-3]:** Retentive holes measuring 1.5 mm in diameter and 0.5 mm in depth.

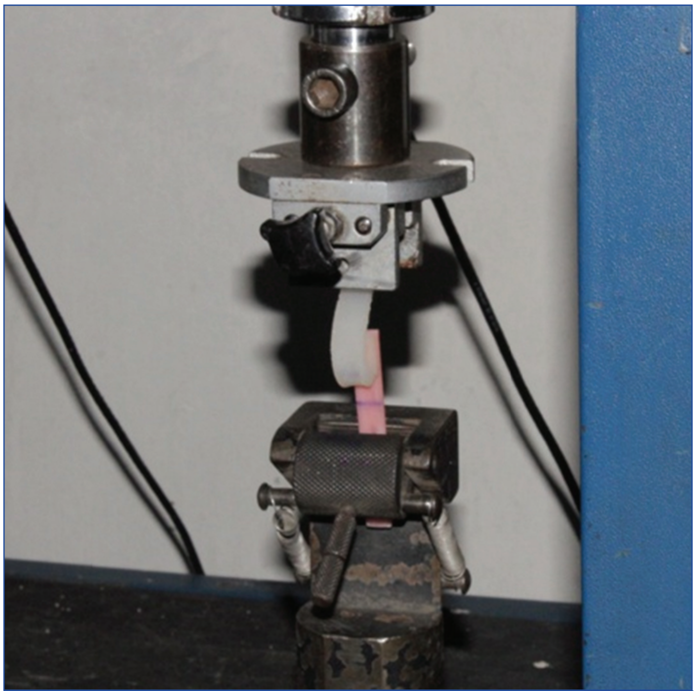
All the resin specimens were adhered to their respective silicone material and subjected to 180° peel bond test on Universal Testing Machine (UNITEST 10) at a crosshead speed of 10 mm/min. The free end of the strip was turned back at 180° so that the hard acrylic bar was clamped in the lower clamp and the soft free silicone strip was gripped in the upper clamp [Table/Fig-4]. The readings were noted for each specimen.

The PBS (N/mm) was determined using the formula [5]:

Peel strength=F / W (1+λ/ 2+1)

{Where, F=maximum force required to cause bond failure recorded (N); W=Width of specimens (mm); λ=Extension ratio of silicone elastomer (the ratio of stretched to primary length)}.

Application of force on the specimen gives us the value of flexural strength. Thus, value is included in the table. After the peel test, each acrylic resin and silicone specimen were visually checked to evaluate the failure of the materials to peel completely. The failures were categorised as adhesive, cohesive and mixed. On complete separation of the resin and silicone, the



**[Table/Fig-4]:** Prepared acrylic and silicone specimen placed in the universal testing machine for the peel bond test.

failure was categorised as adhesive failure. Cohesive failure was characterised by separation occurring entirely within the silicone material. Mixed failure was characterised when both adhesive and cohesive failures are present [4]. A full separation of the substructure and silicone was described as adhesive failure. Cohesive failure described as failure that occurs solely inside the silicone substance. Cohesive and adhesive failures coexisted in a mixed failure.

## STATISTICAL ANALYSIS

The obtained values were evaluated using descriptive statistics for mean±Standard deviation (SD), Confidence interval (CI), Independent t-test and One-way ANOVA or intragroup and intergroup comparisons.

## RESULTS

[Table/Fig-5] shows that the difference between the mean PBS (MPa) in Subgroup A1 and Subgroup A2 is statistically significant as p-value is 0.05 and t value is 2.4. The p-value for the difference in PBS in Subgroup B1 and Subgroup B2 is 0.341 which is statistically insignificant.

[Table/Fig-6] shows the comparison of the mean PBS between the subgroup A1 and B1 and A2 and B2. The result shows no statistically significant p-value between the compared sets (p-value 0.073 and 0.181, respectively). In [Table/Fig-7] One-way ANOVA test was applied to compare the sample means of A1, B1, A2 and B2 for the peel bond group. The difference between the four means was statistically non-significant (p-value 0.064 and 0.130, respectively). All the four groups provide strong evidence to conclude that there is no change between any groups. All groups have means similar to PBS. The type of bond failure for every specimen in the study was assessed visually and designated as adhesive, cohesive and mixed. Test showed that specimens of subgroup A1 and B1 were separated in adhesive type of bond failure. The specimens of subgroup A2 and B2 separated in cohesive and mixed type of failure.

## DISCUSSION

In the present study, the PBS of PMMA acrylic resin and maxillofacial silicone is found to be significantly better in group A with retentive holes. Thus, the null hypothesis was rejected. In group B, it is found that $TiO_2$ nano particles do not contribute in improving the PBS.

The bonding between maxillofacial silicone and acrylic resin substrate is the key for a successful facial prosthesis as detachment of acrylic resin housing from the silicone, tearing of the silicone and poor fitting of the prosthesis are the main reasons of remaking the prosthesis. Patients generally remove prostheses by grabbing a part of the prosthesis (i.e., silicone body) or rotating or peeling it away from the skin. Bonding of facial prostheses is affected by various types of stresses and directions of forces during removing and cleaning. The bond strength test for maxillofacial silicones to an acrylic resin has been tested in the past using different testing methods such as the peel bond test, the shear bond test and the tensile bond test [9,10].

| Group | | N | Mean | Std. deviation | Std. Error Mean | Mean difference | p-value | t value |
|---|---|---|---|---|---|---|---|---|
| Peel bond | A1 | 16 | 0.192 | 0.124 | 0.031 | -0.250 | 0.05 | -2.4 |
| | A2 | 16 | 0.442 | 0.475 | 0.119 | | | |
| **Group** | | **N** | **Mean** | **Std. deviation** | **Std. error mean** | **Mean difference** | **p-value** | **t value** |
| Peel bond | B1 | 16 | 0.362 | 0.345 | 0.086 | 0.092 | 0.341 | 0.97 |
| | B2 | 16 | 0.269 | 0.164 | 0.041 | | | |

**[Table/Fig-5]:** Comparison of mean PBS in MPa between the subgroups within the same group.
Independent t-test (p-value <0.05), Peel Bond Strength (PBS)

| Group | | N | Mean | Std. deviation | Std. error mean | Mean Difference | p-value | t value |
|---|---|---|---|---|---|---|---|---|
| Peel bond | A1 | 16 | 0.192 | 0.124 | 0.03 | -0.170 | 0.073 | -1.86 |
| | B1 | 16 | 0.362 | 0.345 | 0.08 | | | |
| Peel bond | A2 | 16 | 0.442 | 0.475 | 0.11 | 0.172 | 0.181 | 1.38 |
| | B2 | 16 | 0.269 | 0.164 | 0.04 | | | |

**[Table/Fig-6]:** Comparison of mean PBS between the similar subgroups of the two different groups.
Independent t-test (p-value <0.05), Peel Bond Strength (PBS)

| Group | | Sum of squares | df | Mean square | F | ANOVA p-value |
|---|---|---|---|---|---|---|
| Force (N) | Between groups | 13.998 | 3 | 4.666 | 2.551 | 0.064 |
| | Within groups | 109.730 | 60 | 1.829 | | |
| Peel bond | Between groups | 0.568 | 3 | 0.189 | 1.957 | 0.130 |
| | Within groups | 5.805 | 60 | 0.097 | | |

**[Table/Fig-7]:** Comparison of PBS between the two groups and within the group.
One-way Analysis of variance (p-value <0.05)

In the present study, the PBS between the two groups {group A (PMMA and maxillofacial silicone elastomer) and group B (PMMA with maxillofacial silicone elastomer reinforced with TiO2 nanoparticles)} was tested by Molecular Adhesion using primer {Subgroup A1 (PMMA acrylic resin with maxillofacial silicone elastomer with primer) & Subgroup B1 (PMMA acrylic resin with maxillofacial silicone elastomer reinforced with TiO2 nanoparticles with primer)} and Micromechanical Adhesion using retentive holes {Subgroup A2 (PMMA acrylic resin with retentive hole with maxillofacial silicone elastomer) & Subgroup B2 (PMMA acrylic resin with retentive hole with maxillofacial silicone elastomer reinforced with TiO2 nanoparticles)}. The test was done on a Universal Testing machine (Unitest 10) at a crosshead speed of 10 mm/min according to ASTM D-903 specifications [5].

In this study, the mean PBS in the subgroup A1 and A2 was found to be statistically significant with p-value of 0.05. Thus, authors can infer that as compared to primer, retentive holes provide significant PBS. These results concur with that of Craig RG and Gibbons P stated that roughened surface is recommended to enhance the adhesive bond between acrylic and a resilient liner [11]. Findings from their study indicate that the adhesive values obtained from roughened surfaces were approximately double those of smooth surfaces. This increase in adhesion is attributed to the slightly irregular surface, which allows for mechanical locking with the softer material.

On the contrary, Jagger RG et al., claimed that roughening the resin surface with an acrylic bur weakened the bond because of the stress concentration caused by discontinuities of the surface and entrapped air or gas at the interface, which could further weaken the bond by the created voids [12]. However, the errors can be decreased by preventing air or gas entrapment, with incorporating various factors like definite pattern of surface roughening with adequate intervals of plain surface and roughness, reducing the possibility of stress concentration and weakening of the acrylic resin substrate. Also, appropriate equalisation of pressure while packing of silicone into the mould will ensuring the proper flow of silicone elastomer into the depressions created on the acrylic substrate, will prevent air or gas entrapment [5].

However, Hatamleh MM et al., Meran Z and Anwar OK, Hatamleh MM et al., mentioned in their studies that A-330 Gold platinum primer improves the bonding strength [9,13,14]. This is because it contains modified polyacrylates in methylethylketone and dichloromethane and it chemically enhances the bond of silicone elastomer to acrylic resins, plastics, and metal.

In the present study, the p-value of the mean difference of PBS in Subgroup A1 & Subgroup B1 is 0.073 and that of A2 and B2 is 0.181 which was statistically not significant. The results indicate that adding nanoparticles to maxillofacial silicone, when using primer does not enhance the PBS between the given two materials. However, it has been seen in studies by Han Y et al., Cevik P et al., and Sonnahalli NK et al., that incorporation of nano-particles enhances the mechanical properties like tensile strength, tear strength, hardness and colour stability in maxillofacial silicone [6-8]. Among the nanoparticles used, nano-$TiO_2$ has a high specific surface area that reinforces contact areas and bonding, leading to significant improvements in mechanical properties [6].

It is also seen that at a concentration of more than 3%, the same nanoparticles decreased the tear strength, tensile strength, and elongation. Due to their high surface energy and chemical reactivity, nanoparticles have weak interactions with silicone elastomers, leading to some degree of agglomeration at higher concentrations, which reduces their mechanical properties [15,16].

Usually, nanoparticles can bond to polysiloxane. But, when the amount of nanoparticles increases, there may be an inadequate amount of polysiloxane to link the nanoparticles effectively. This leads to a decrease in the interfacial bonding in the nanoparticle silicone elastomer material [16,17]. Considering this fact 2% $TiO_2$ was used in the present study.

The type of bond failure in the study for every specimen were assessed visually and designated as adhesive, cohesive and mixed. Test showed that specimens of primer A-330 Gold platinum were separated in adhesive type of bond failure. A study by Rai T at el., also shows adhesive failure between acrylic and two different maxillofacial silicones when primer is used between them [18]. Whereas micromechanical undercut specimens showed cohesive and mixed type of failure. For cohesive failures, the PBS between the silicone and the denture base exceeded the intrinsic strength of the silicone material. The advantage of the peel test is that it is the only method in which failure proceeds at a controlled rate and the peel force is a direct measure of the work of detachment, cohesive peel bond test failures should be interpreted with caution and other causes like irregularity, voids must be ruled out.

### Limitation(s)

This in-vitro study was performed under controlled laboratory conditions and may not fully represent the clinical environment of maxillofacial prostheses. Peel bond strength was measured at a single time point without aging procedures. Since the extra oral prostheses are constantly exposed to heat and moisture the aging of samples may affect long-term bond durability.

The constant up-gradation of material science, has introduced newer technologies and materials in maxillofacial dentistry too. The advent of CAD CAM and 3D printing has revolutionized the industry. However, there lies scope for evaluating the bond between the advanced silicone and the newer biomaterials.

## CONCLUSION(S)

Within the limitations of this study, it can be concluded that retentive holes offer significantly greater PBS between PMMA acrylic resin and maxillofacial silicone compared to the PBS achieved using maxillofacial primer. It can also be concluded that adding nano-particles to maxillofacial silicone does not enhance the PBS between the given two materials, thus it can be suggested that in extraoral prostheses, which incorporate the use of PMMA acrylic resin and maxillofacial silicone, reinforcement of silicone should not be done.

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