Original Article

An *in vitro* study to compare the influence of two different primers on the peel bond strength between a maxillofacial silicone material and an acrylic resin material versus a composite resin material

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Abstract

Aim: The aim of this study was to evaluate and compare the peel bond strength of an autopolymerizing acrylic resin and a fiberreinforced composite (FRC) resin to a heat temperature vulcanizing maxillofacial silicone (M511) using two different primers.

Settings and Design: *In vitro* **-** comparative study.

Materials and Methods: Autopolymerizing acrylic resin and FRC resin specimens with a dimension of 75 mm (length) ×10 mm (width) × 3 mm (height) were fabricated. A total of 60 samples were split into six categories based on the substructure material and primers (A330G primer and Sofreliner tough primer) used to bond the maxillofacial silicone to the FRC and acrylic resin specimens. In a universal testing machine, the peel bond strength was conducted at a 10 mm/min crosshead speed until bonding failure occurred.

Statistical Analysis Used: The t-test, one-way analysis of variance, and the Tukey's honest significant difference (post hoc test) tests were used to statistically assess the values.

Results: The Sofreliner tough primer produced the greatest peel bond strength in both the acrylic resin (0.89690 N/mm) and the FRC resin groups (3.19860 N/mm). Adhesive failures predominated in the acrylic resin group regardless of the primer used. The FRC group showed predominantly cohesive failures with both the A330G primer and Sofreliner tough primer.

Conclusion: This study suggests that FRC resin combined with Sofreliner tough primer can significantly enhance the peel bond strength.

Keywords: Acrylic resin, fiber-reinforced composite resin, maxillofacial silicone, primers

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INTRODUCTION

Maxillofacial extraoral prostheses have been utilized to rehabilitate individuals who have experienced substantial hard and soft tissue loss in the maxillofacial region.[1] Silicone elastomers are utilized extensively for manufacturing such prostheses since they are biocompatible, durable, chemically inert and are capable of matching skin color by addition of pigments.[2,3]

Various means of auxiliary retention include adhesives, magnets, eyeglasses, implants, and combination of the above.[1,4] With the development of craniofacial implants, improved prostheses retention and stability is possible.[5] In implant-retained prostheses, a retentive matrix is necessary to keep the bars, clips, or magnets in place. This retentive matrix is constructed of either acrylic resin (heat-polymerizing, auto-polymerizing, or visible light-curing acrylic resin) or fiber-reinforced composite (FRC) which has enough strength to hold the attachments in place and serve as a substructure.

Maxillofacial silicone elastomers are chemically distinct polymers of dimethylsiloxane compared to acrylic resin or FRC resulting in a low bond strength between the two.[6] With the use of silane-containing primers, the bond between maxillofacial silicone and the substructure can be enhanced. In addition to the facial silicone primer, silicone soft liner primer can also be used as it is cost effective and gives desirable clinical outcome. Multiple studies have investigated the influence of various primers on the peel bond strength of a room temperature vulcanizing maxillofacial silicone to substructure materials. However, there is no study investigating the influence of a silicone soft liner primer on the peel bond strength of a heat temperature vulcanizing maxillofacial silicone (M511 platinum silicone) to substructure materials. Furthermore, none of the research assessed and compared the peel bond strength of maxillofacial silicone to two substructure materials, namely autopolymerizing acrylic resin and a FRC resin. Hence, the goal of this study was to assess and compare the peel bond strength of a heat temperature vulcanizing maxillofacial silicone to an autopolymerizing acrylic resin and a FRC resin utilizing a facial silicone primer and a silicone soft liner primer.

The null hypothesis was that after treatment with a facial silicone primer and a dental silicone soft liner primer, the peel bond strength of a heat temperature vulcanizing maxillofacial silicone to an autopolymerizing acrylic resin and a FRC resin would be comparable.

MATERIALS AND METHODS

The study was approved by the institutional review board (Goa Dental College/IEC/PG/Dated 9/04/2018). The M511 platinum silicone elastomer is a low viscosity heat vulcanizing silicone available in two parts and mixed in a ratio of 10:1 by weight. Sixty specimens were fabricated in total for the study. The following formula was used to derive the sample size:

$$N = \frac{2\sigma^2 \times (Z\alpha + Z\beta)^2}{\Delta^2}$$

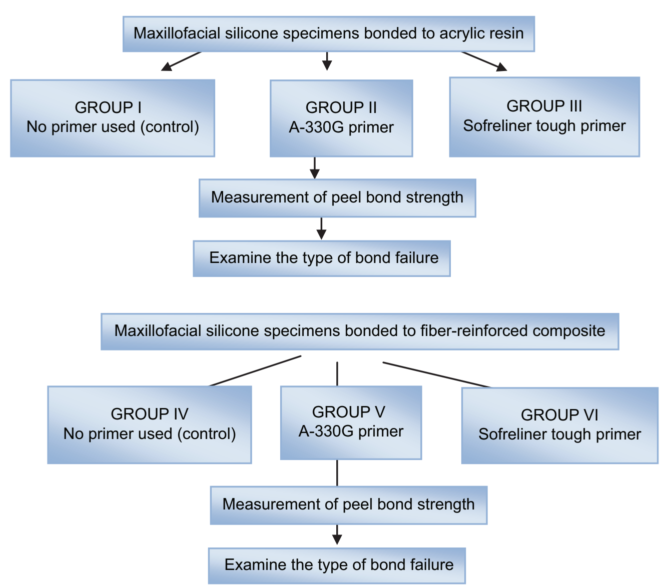
σ⟶ Standard deviation

Δ⟶ Difference of mean

A sample size of 10 per group was determined using the aforementioned formula and taking values from the key article. The methodology is depicted in Flowchart 1.

Distribution of specimens

- Group I – Ten specimens of acrylic resin bonded to maxillofacial silicone without the use of a primer (control)
- Group II – Ten specimens of acrylic resin bonded with the A-330G primer to maxillofacial silicone
- Group III – Ten specimens of acrylic resin bonded with the Sofreliner tough primer to maxillofacial silicone
- Group IV – Ten specimens of FRC attached without the application of a primer to maxillofacial silicone (control)
- Group V – Ten specimens of FRC attached with A-330G primer to maxillofacial silicone
- Group VI – Ten specimens of FRC attached with Sofreliner tough primer to maxillofacial silicone.



Flow Chart 1: Flowchart of the methodology

Description of specimen

Each test specimen of acrylic resin and FRC of dimensions 75 mm (length) × 10 mm (width) × 3 mm (height) was made. Twenty-five millimeter length of each specimen was bonded to the maxillofacial silicone elastomer using two different primers and the rest 50 mm was left unbonded.

Fabrication of acrylic resin bars (*n* = 30)

Wax patterns with dimensions of 75 mm (long), 10 mm (width), and 3 mm (height) were fabricated. Using the traditional compression molding procedure, these were invested and dewaxed [Figure 1]. Self-polymerizing acrylic resin (RR Cold Cure; DPI) was manipulated in a ratio of 3:1 (polymer: monomer) and packed into the mold cavity on reaching the dough stage. Following polymerization at room temperature, the specimens were retrieved and polished with silicon carbide paper no. 220.

Fabrication of fiber-reinforced composite bars (*n* = 30)

Five additional acrylic resin bars were fabricated. These bars were attached with cyanoacrylate resin to a smooth glass surface, and an impression of the bars with putty addition silicone was produced to obtain a mold for fabricating FRC bars. The FRC (everX Posteriors; GC) was packed into the silicone mold and light polymerized for 120 [Figure 2]. Thirty specimens were made in total.

Figure 1: Wax patterns of 75 mm (length) × 10 mm (width) × 3 mm (height) invested in dental stone

Figure 3: Two layers of the modeling wax placed on the specimens

Bonding of maxillofacial silicone elastomer to the fiber-reinforced composite resin and acrylic resin specimens

For 50 mm length, all the specimens were covered with a 0.027-mm Teflon strip. The remainder of the 25 mm length was utilized to bond the silicone elastomer to the acrylic resin and the FRC resin bars. Two layers of modeling wax (3 mm) were placed on the specimens and aligned with the substructure material edges [Figure 3]. All of the specimens were conventionally invested and dewaxed.

Every specimen surface was cleaned for 30 s with acetone, then left to air dry. The specimens were categorized into 6 groups. In Groups II and V, a thin coating of A-330G primer was applied to the uncovered area of the specimens with a camel hair brush and left to dry for 30 min as directed by the manufacturer [Figure 4a]. A thin coating of Sofreliner tough primer was applied to the specimens in Groups III and IV and left to dry as per the manufacturer's instructions [Figure 4b].

The M511 platinum silicone elastomer (Technovent Ltd) consists of a base (Part A) and a catalyst (Part B) which are to be manipulated in a ratio of 10:1 by volume or weight. A digital precision weighing scale was used to measure the base and catalyst using a plastic spoon to maintain 10:1 (5.650 g base: 0.5 g catalyst) by weight ratio. The two components were mixed using a stainless steel spatula on a white ceramic tile until a homogeneous mix was obtained. To eliminate small air bubbles, the mixture was placed in a

Figure 2: Fiber-reinforced composite resin dispensed into putty addition silicone mold

Figure 4: (a) Application of A330-G primer. (b) Application of Sofreliner primer

dappen dish and vacuum deaeration was done for 5 min at 0.9 bars. This mix was packed into the mold using a spatula and polymerized for 1 h at 100°C in a preheated hot air oven as directed by the manufacturer.

The specimens were recovered and a scissor was used to cut out the flash. The Teflon tape was removed from the specimens, resulting in a 50 mm long unbonded area that could be placed in the universal testing machine [Figure 5].

Bond strength test (180° peel test)

A universal testing machine (Star Testing Systems, India. Model No. STS 248) was utilized to perform the 180° peel bond strength test at 10 mm/min crosshead speed. The test was conducted as per the American Standards for Testing and Materials (ASTM) D-903. The silicone elastomer was attached to the substructure material on one end (25 mm to 10 mm to 3 mm) whereas the other end was left free (50 mm to 10 mm to 3 mm). The silicone elastomer was connected on the other end. The silicone strip's free end was twisted back at 180°, allowing the substructure material to be clamped in the bottom clamp and the free end of the silicone strip to be grasped in the higher clamp [Figure 6]. The maximum force required for bond failure was noted. The peel bond strength for each specimen was calculated using the given formula:"

PG = F/W

PG = Peel bond strength (N/mm), F = Greatest force at the starting point of failure (N), W = Sample width (mm).

The failure mechanisms were categorized into adhesive, cohesive, or mixed by visual examination of the interface between the substructure and silicone. 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 of the test readings was conducted.

OBSERVATIONS AND RESULTS

The peel bond strength values were analyzed by:

- Independent *t*-test was performed to evaluate whether there was a difference between the mean peel bond strength in the autopolymerizing acrylic resin and the FRC resin groups with respect to a particular primer
- One-way analysis of variance was used to compare the mean peel bond strength among the different groups in the autopolymerizing acrylic resin and the FRC resin groups, respectively
- Tukey's honest significant difference (HSD) (*post hoc* test) was carried out to compare the mean peel bond strength of the autopolymerizing acrylic resin and the FRC resin groups, two at a time (pair wise) to determine where a significant difference exists.

Table 1 shows the mean peel bond strength values. The peel bond strength values were found to be the maximum in Group IV (3.19860) and the least in Group I (.13870). The control group shows significantly higher peel bond strength for the FRC group (0.87310) as compared to the acrylic resin group (0.13870) ($P < 0.05$). The A-330G primer group shows significantly higher peel bond strength for the FRC Group (1.82580) compared to the acrylic resin group (0.32740) ($P < 0.05$). The Sofreliner primer group shows significantly higher peel bond strength for the FRC group (3.19860) in comparison to the acrylic resin group (0.89690) ($P < 0.05$).

Tukey's HSD test showed a statistically significant difference in the peel bond strength values at 0.00

Figure 5: Fiber-reinforced composite resin and acrylic resin specimens bonded to maxillofacial silicone elastomer

Figure 6: 180° peel bond strength test

Table 1: Descriptive statistics for the peel bond strength for different groups

	Mean	SD	Minimum	Maximum
Bond strength of acrylic to maxillofacial silicone (N/mm)				
Control (I)	0.13870	0.001947	0.136	0.142
A-330G primer (II)	0.32740	0.001776	0.325	0.330
Sofreliner primer (III)	0.89690	0.004228	0.891	0.903
Total	0.45433	0.327806	0.136	0.903
Bond strength of FRC to maxillofacial silicone (N/mm)				
Control (IV)	0.87310	0.003900	0.868	0.880
A-330G primer (V)	1.82580	0.002530	1.821	1.830
Sofreliner primer (VI)	3.19860	0.002633	3.195	3.203
Total	1.96583	0.970854	0.868	3.203

SD: Standard deviation, FRC: Fiber-reinforced composite

Table 2: Comparison of the peel bond strength based on groups

Dependent variable	Mean difference (I–J)	SE	Significant	95% CI (lower bound-upper bound)
Bond strength of acrylic to maxillofacial silicone (N/mm)				
Control (I)				
A-330G primer (II)	−0.188700	0.001286	0.000	−0.19189-−0.18551
Control (I)				
Sofreliner primer (III)	−0.758200	0.001286	0.000	−0.76139-−0.75501
A-330G (II)				
Sofreliner primer (III)	−0.569500	0.001286	0.000	−0.57269-−0.56631
Bond strength of FRC to maxillofacial silicone (N/mm)				
Control (IV)				
A-330G primer (V)	−0.952700	0.001379	0.000	−0.95612-−0.94928
Control (IV)				
Sofreliner primer (VI)	−2.325500	0.001379	0.000	−2.32892-−2.32208
A-330G (V)				
Sofreliner primer (VI)	−1.372800	0.001379	0.000	−1.37622-−1.36938

CI: Confidence interval, SE: Standard error, FRC: Fiber-reinforced composite

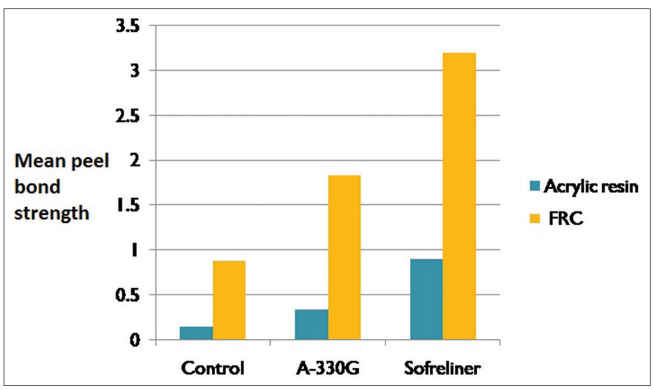


Figure 7: Comparison of the mean peel bond strength in various groups

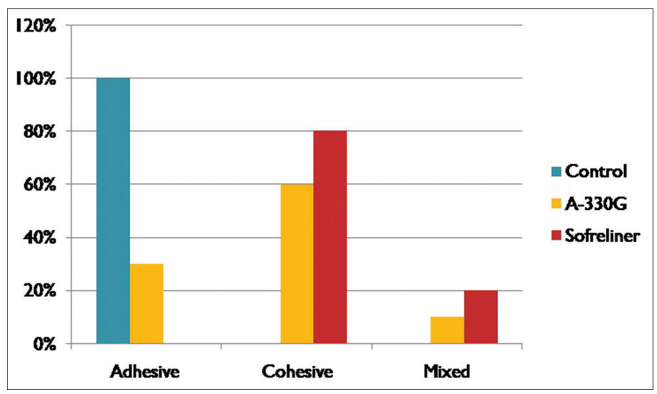


Figure 8: Mode of failures in the fiber-reinforced composite group

level ($P < 0.05$) [Table 2]. In general, the Sofreliner primer groups presented the highest bond strength values and the control groups presented the lowest bond strength values.

Figure 7 shows the comparison of the mean peel bond strength in various groups. Figures 8 and 9 show the mode of failures in the FRC and acrylic resin groups, respectively.

DISCUSSION

As per the results of this *in vitro* study, the null hypothesis was rejected as there was statistically significant difference between the peel bond strength of a heat temperature vulcanizing maxillofacial silicone to an autopolymerizing acrylic resin and a FRC resin using a facial silicone primer and a soft liner primer.

In maxillofacial prostheses, acrylic resins have been used as a framework material to hold the clips, bars, or magnets. Kurunmäki H and Kantola[7] introduced the use of FRC as a substructure material for restoring lateral midfacial defects. FRC contains polymers (the resin matrix) and inorganic glass fibers and fillers. The structure of FRC utilized in dentistry consists of an interpenetrating polymer network (IPN), which is a network-like combination of two

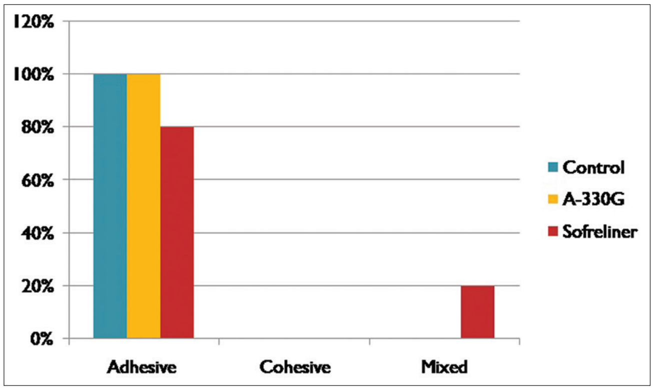


Figure 9: Mode of failures in the acrylic resin group

or more polymers.[8] Both the polymer matrix and the fillers are involved in the bonding process. The polymer must be dissolved using a solvent (monomer) when bonding an IPN. The OH groups found in the silica and glass fibers can also be silanized using methacrylate silanes.

Silanes usually consist of two parts: an organic group that bonds to the resin and inorganic components that attach to the inorganic substrate. The adhesive bond between the polymer matrix and the filler is enhanced when a new resin or a composite resin is bonded to FRC. Mechanical interlocking is the other important adhesion mechanism between FRC and another polymer. The exposure of the fibers on the bonding surface improves their adhesive properties. Furthermore, the monomers diffusing from the resin to be bonded may adhere to the noncross linked phases of the polymer within the FRC matrix.[8,9]

A good bonding between silicone and substrate material which hold the clips and magnets is necessary for the success of implant retained extraoral prostheses. This bond must be strong enough to withstand the forces exerted during prostheses removal as well as during deflasking of the mold. The different chemical nature of these materials frequently results in loss of bonding. Primers are therefore necessary to improve the bonding strength of the maxillofacial silicone elastomer to the substructure, thereby avoiding silicone delamination.

In the present study, the peel bond strength of a heat temperature vulcanizing maxillofacial silicone to an autopolymerizing acrylic resin and a FRC resin using a facial silicone primer and a soft liner primer were evaluated. Furthermore, the mode of bond failure was evaluated visually. Tensile,[10,11] shear,[12,13] and peel[12,14,15] tests can be used to determine bond strength. During removal, implant-retained maxillofacial prostheses are subjected to a variety of stresses and detaching forces. These are often peeled away from the skin by the patient during removal. The peel test effectively simulates this type of force. The 180º peel test was thus utilized to evaluate the bond strength between maxillofacial silicone elastomer and acrylic resin/FRC. The test was performed as per the ASTM D-903 specifications.

Maxillofacial silicone elastomers are chemically different from that of FRC or acrylic resin resulting in a low bond strength between the two materials. The M511 platinum silicone elastomer (Technovent Ltd.) is a two-component system that uses an addition reaction to cross-link. The base component of silicone (Part A) consists of polydimethylsiloxanes, a platinum complex as catalyst and surface treated silica fillers.[16] The cross-linker (Part B) contains a hydrogen siloxane polymer. A hydrogen siloxane polymer is included in the cross-linker (Part B). For bonding platinum-cured silicone elastomers, the A-330-G primer is recommended by Factor II. The polyacrylates in dichloromethane and methyl ethyl ketone solvents chemically improve the bonding between maxillofacial silicone elastomers and acrylic resin or FRC.[11] In this study, the A-330G primer resulted in greater peel bond strength values of 0.32740 and 1.82580 for the acrylic resin and FRC groups, respectively, in comparison to the control groups (0.13870 and 0.87310).

Due to differences in primer composition, differences in peel bond strengths were detected. The Sofreliner primer is a dental silicone soft liner primer that improves the bond strength between acrylic resin and soft liner. The type of priming solvent has a significant impact on bonding strength. When the primers were compared, the Sofreliner primer produced the strongest peel bond strength for both the acrylic resin (0.89690) and FRC (3.19860) groups. The high peel bond strength exhibited by the Sofreliner primer can be attributed to the presence of ethyl acetate solvent, polymethylmethacrylate (PMMA), and polyorganisiloxane as active ingredients. The FRC polymer matrix includes a network of cross-linking diacrylates and a linear network of PMMA polymers. Ethyl acetate is known to be a good solvent for PMMA that dissolves the polymer matrix of the FRC resulting in high bond strength. The potential bonding mechanism is that PMMA in the primer may link to acrylic resin and polyorganisiloxane may bond to a facial silicone substrate.

The results obtained in this study are in accordance with that of Haddad *et al*.[17] who found the maximum peel bond strength between MDX4-4210 silicone and acrylic resin when a Sofreliner primer was used in comparison to the bond strength obtained with mechanical retention (scratches on the acrylic resin surface) and Dow Corning 1205 Primer. Similar findings have been reported by Chang *et al*.[18] who assessed the peel bond strength between facial silicone and polyurethanes and found a higher bond strength for the Sofreliner primer. There are currently no worldwide criteria governing the clinically acceptable bonding strength of maxillofacial materials to diverse surfaces.

The bond failure types were divided into adhesive, cohesive, or mixed. All specimens exhibited adhesive failure when no primer was used (Groups I and IV) which could be due to a lack of affinity between acrylic resin or FRC and maxillofacial silicone. In the acrylic resin group, the control and A-330G primer groups presented 100% adhesive failures whereas the Sofreliner primer group showed 80% adhesive failures and 20% mixed failures. Similar results were obtained by Sanohkan *et al*.[11] The Sofreliner primer showed 80% cohesive failures whereas the A-330G primer produced 60% cohesive failures in the FRC group indicating molecular adhesion. The findings are consistent with those of Haddad *et al*.[17] and Chang *et al*.[18]

Although the study was carried out according to the established methods, there are certain constraints. This study has been conducted *in vitro*, and hence, clinical conditions were not replicated. Bond strength tests only apply forces in one direction, whereas a facial prosthesis is subjected to a variety of stresses. Hence, specimens in bonding tests do not characterize the actual facial prostheses. The evaluation was carried out for a single type of unpigmented maxillofacial silicone elastomer. Further studies with various other maxillofacial silicone materials with the addition of pigments are indicated. The effect of aging was not assessed. Since the patient will be using the extraoral prosthesis for long periods, additional research at various stages of accelerated aging is warranted.

In this *in vitro* study, FRC demonstrated high peel bond strength when bonded to heat temperature vulcanizing maxillofacial silicone regardless of the primer used and hence should be considered as an alternative to substructure materials to prolong the life of the prosthesis. Having demonstrated greater peel bond strength for the Sofreliner tough primer irrespective of the substructure material, its use should be considered for bonding to heat temperature vulcanizing maxillofacial silicones as it is cost effective and gives desirable clinical outcome.

CONCLUSION

Within the limits of this research, the following conclusions were drawn:

1. The variation in the peel bond strength among the different groups was statistically significant
2. Results from the 180° peel test have shown that the self-polymerizing acrylic resin groups were statistically lower when compared to the FRC attaching to the silicone elastomer ($P < 0.05$)
3. Comparing the primers, the Sofreliner tough primer produced the greatest peel bond strength in both the acrylic resin and the FRC groups
4. Adhesive failures predominated in the acrylic resin group regardless of the primer used. The FRC group showed predominantly cohesive failures with both the A-330G primer and Sofreliner tough primer.

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Conflicts of interest
There are no conflicts of interest.

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