

Evaluation of peel bond strength between plexiglas acrylic (pmma) and maxillofacial silicone using three different primers

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Objective: The bonding of maxillofacial silicone to acrylic resin is a critical issue when rehabilitating with implants retained maxillofacial prosthesis. The aim of this study was to evaluate 180° peel bond strength between prefabricated Plexiglas acrylic and maxillofacial silicone by using three different primers.

Material and Methods: The peel bond strength between maxillofacial silicone, A-2186 and Plexiglas acrylic resin was assessed by using three different primers (A-306, A-304 and A-330G). Specimens were divided into 3 groups according to the specific primer. All specimens were fabricated within Aluminum mold and evaluation of peel bond strength was done by Universal testing machine. One way ANOVA and Tukey's test were used to analyze data with significance level set at 0.05. Modes of bond failures were assessed by visual inspection and categorized into cohesive, mixed and adhesive failures.

Results: There was significant impact of primers on bond strength between silicone A2186 and Plexiglas acrylic ($p < 0.05$). The 180° peel bond strength ranged from 1.63 to 4.67N/mm among primers groups. The primer A-330 had the highest peel bond strength of 4.67N/mm among primers ($p < 0.05$). Cohesive failure was mainly found with primer A-330 while mixed and adhesive failures were predominantly seen in primer groups, A-306 and A-304 respectively.

Conclusion: A-330 primer provided the highest 180° peel bond strength between prefabricated Plexiglas acrylic and silicone A-2186. Plexiglas acrylic might be used as an alternative material to conventional acrylic resin during fabrication of maxillofacial prosthesis.

Keywords: Peel bond strength, Silicone A-2186, Primers, maxillofacial silicone

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Introduction:

Maxillofacial prosthetics deals with the prosthetic management of congenital and acquired defects. Generally, maxillofacial prosthesis had been retained by various methods including medical adhesives, anatomical undercuts and eyeglasses. The concept of Osseo-integration and the introduction of craniofacial implants added the mechanical advantage over conventional retentive mechanisms in terms of improved esthetics, function and life span of prosthesis. [1-4]

Craniofacial implants retained silicone facial prosthesis needs the retentive housing to secure the various attachments (bar clips or magnets). The rigid housing is usually made from auto-polymerizing acrylic resin to which the maxillofacial silicone is attached. The attachment between silicone and acrylic housing can be chemically or mechanically enhanced, however research is mainly focused towards chemical bonding between silicone and acrylics.

Maxillofacial silicone and acrylic resin are both polymer-based materials however they are different in their chemical structure i.e maxillofacial silicone is poly (dimethyl siloxane) while acrylic resin is poly-methyl-methacrylate. Therefore, silane-coupling agents are applied over rigid acrylic resin housing surface to enhance the bond strength. [5] Silane coupling agents have one organic substituent and three hydrolysable substituents that react with both silicone and acrylic materials. [6] These coupling agents prepare the surfaces by etching or promoting hydrogen bonding and covalent coupling, promoting the wettability of the substrate by allowing the polymeric ingredients to impregnate into the surface layer. [7]

During function, silicone facial prosthesis faces numerous physical and mechanical failures, including bond failures between silicone and acrylic housing, color changes, wear and tear of silicone prosthesis margins. [8, 9] Though, silicone elastomers have undergone much improvement in terms of improved physical and mechanical properties; debonding of silicone away from the retentive housing is still a consistent problem. (Fig. 1) The in-vitro studies have been carried out to test and replace the retentive acrylic resin with retentive glass-fiber framework, or the use of different bond agents including primers and adhesives. [10-12]

Surface roughness is believed to increase the surface area for bonding between silicone and acrylic resin. Various methods have been used to prepare surface of acrylic prior to application of primers, such as beading with rotating burs, sand blasting, holes and rubbing with SiC paper over acrylic substrate. Polyzois and Frangou [11] found that the surface preparation with 80 grit SiC paper had enhanced the bond strength as compared to 240, 260, 600 or 1000-grit SiC paper. Frangou et al. [12] further claimed that bond strength had improved after resin substrate surface preparation using 80 Grit SiC paper in polishing machine. The best technique for surface preparation of bonding surface is still unclear; therefore current study was done using 80 Grit SiC paper to determine peel bond strength between Plexiglas acrylic and maxillofacial silicone elastomer.

Many studies have been done so far in order to find the best possible bond strength between acrylic and silicone based materials, which can withstand the functional peeling and shearing forces. The bond strength tests have been evaluated according to tensile, sheer and peel forces acting on functional prosthesis. Shear tests

concentrate the stress on edges and are affected by the selected deformation ratio, the material under investigation, and the test methodology itself. Shear and 180-degree peel tests simulate clinical situation for testing the bonding strength of maxillofacial silicone elastomers to the substructure materials. However, the results are dependent on type of material, its rigidity and thickness. In contrast, the tensile tests signifies the strength of materials instead of bond strength determination.[13]

The Plexiglas acrylic resin was introduced by, German chemist, Otto Rohm in 1933. Plexiglas acrylic is also known as organic glass. Although it does not belong to silica family but like other thermoplastics, it is often technically classified as type of glass. Plexiglas acrylic sheets are produced by solution polymerization, emulsion polymerization and bulk polymerization. Generally, polymerization proceeds from radical initiation, however anionic polymerization can also be performed as an alternative way. The forming temperature proceeds from glass transition temperature (Tg), at which the Plexiglas acrylic can be molded. Different molding techniques can be used during processing

stages i,e injection molding, compression molding and extrusion. Plexiglas acrylic has been used in medical fields, Ophthalmic, Orthopedics and Cosmetic surgeries [14-16] while its application in maxillofacial prosthesis has never been tested previously. This study was aimed to determine the bond strength between Plexiglas acrylic and commonly used maxillofacial silicone elastomer A-2186 by using different platinum primers to assess its possible application in maxillofacial prosthetics. The mode of failures were examined visually and categorized into cohesive, mixed and adhesive failures.

Materials and Methods

Materials

The prefabricated Plexiglas acrylic strips were used as substrate for primers application. Three different platinum primers (A-330, A-306 and A-304, Factor II, USA) were utilized to treat the surface of Plexiglas acrylic. One maxillofacial silicone elastomer A-2186 (Factor-II, USA) was used. It is an addition cure silicone, supplied as base and catalyst. (Table 1)

Table 1 Description of materials used in this study

Material	Brand Name	Manufacturer	Batch Number
Silicone	A-2186 Platinum silicone elastomer	Factor II, Inc., Lakeside, AZ, USA	R-62846
Primers	A-330 G Platinum Primer	Factor II, Inc., Lakeside, AZ, USA	R4409753
	A-304 Platinum Primer	Factor II, Inc., Lakeside, AZ, USA	S69842-1
	A-306 Platinum Primer	Factor II, Inc., Lakeside, AZ, USA	F72751
Acrylic	Plexiglas Acrylic	Thai Poly-Acrylic Public Limited	

Study Design

The peel bond test was designed according to previous studies. [17-19] The specimens were divided into 3 groups with 10 specimens in each group. Prefabricated Plexiglas acrylic resin strips

were treated with different primers (A-306, A304 and A-330) after surface preparation. Metal mold was designed to accommodate the specimens for heat vulcanization. Universal testing machine was used to perform 180-degree peel bond test.

Mold Fabrication

Three piece Aluminum mold was designed to standardize the fabrication of specimens. Upper and lower lids of mold were used to support and exert force during silicone packing while the middle lid of mold was designed to accommodate 5 sets of specimens (75mm x 10mm x 6mm) for each curing cycle. (Figure 1)

Specimens Fabrication

Pre-fabricated Plexiglas acrylic was precisely laser cut into strips (75mm x 10mm x 3mm). (Figure 2) The bonding area of Plexiglas acrylic strips (25mm x 10mm) was prepared by using 80 grit, SiC paper (Figure 3) while, the un-bonded area (50mm x 10mm) was covered with Adhesive tape (3M scotch). Plexiglas acrylic strips were cleaned with acetone after surface preparation and left to dry for 15 minutes. The uniform layer of primer was applied over Plexiglas strip and left to dry for 30 minutes.

The acrylic strips were aligned into the aluminum mold. Silicone elastomer A-2186 was mixed according to manufacturer's recommendations and placed in vacuum chamber to eliminate air-entrapped bubbles.

The silicone was injected into the mold with the help of syringe. Curing of silicone was done in dry heat oven at 70°C for 2 hours. The specimens were dry stored for 1 week at 23 ± 1 °C room temperature. The cured specimen thickness was 6mm (3mm of Plexiglas acrylic and 3mm of cured silicone elastomer). The silicone strip was bonded to Plexiglas acrylic at one end (25mm x 10mm x 3mm) and free at the other end (50mm x 10mm x 3mm).

Specimens Testing

The specimens were tested with universal testing machine by gripping the rigid Plexiglas acrylic strip in lower clamp while the unbounded silicone was turned back at 180° and gripped in upper clamp. (Figure 4) The crosshead speed was adjusted at 10 mm/min. The specimens were pulled at 180° to peel the silicone from Plexiglas acrylic resin. The force required to induce failure and the modes of bond failure were registered. Peel strength (PS) (N/mm) was calculated according to Eq. [20, 21] F

$$PS = \frac{F}{W} \left(\frac{1+\lambda}{2} + 1 \right)$$

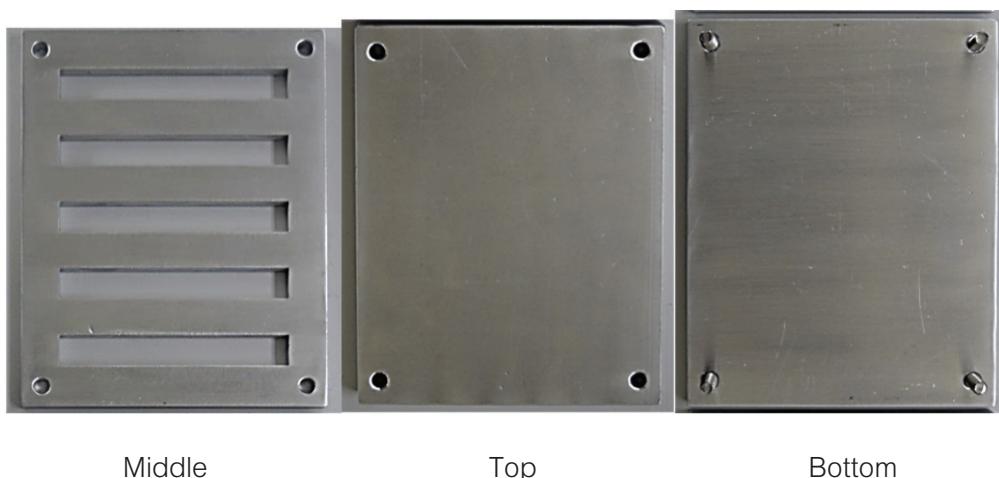


Figure 1 Three piece Aluminum mold for fabrication of specimens

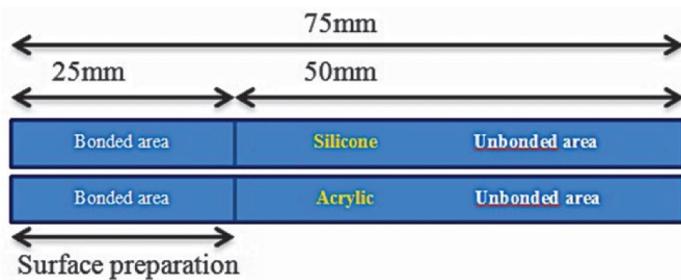


Figure 2 Dimensions of Plexiglas acrylic specimen



Figure 3 Surface preparation of plexiglas acrylic strips

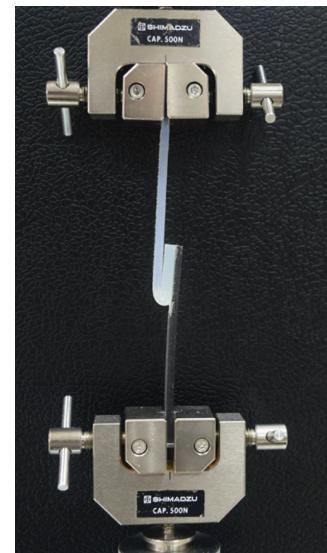


Figure 4 Specimens position in Universal testing machine during peel bond test.

Where F indicates the maximum force (N), W is the width of individual specimen (mm), and λ is the extension ratio (the ratio of stretched to unstretched length) of silicone elastomer. The surface of the Plexiglas acrylic interface was visually assessed, and modes of failures were categorized as adhesive (peel), mixed and cohesive (tearing of silicone elastomer). [22] (Figure 5)

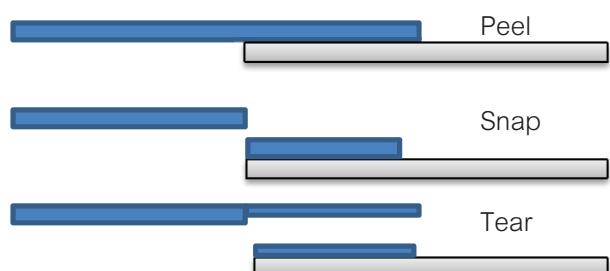


Figure 5 Types of bond failures seen after peel bond test.

One way ANOVA (release 16, SPSS Inc., Chicago, IL) ($p < 0.05$) was used to analyze the significant differences in peel bond strengths among different primers. All data was passed through Levene's test of homogeneity of variance ($\alpha = 0.05$), following the assumption of equal variances. Equal variances were speculated ($p > 0.05$) and Bonferroni *post hoc* test were used to detect statistical significances in bond strength among primers.

Results

There was a significant influence of primers on bond strength between Plexiglas acrylic and maxillofacial silicone A-2186 ($p < 0.05$). Among primers, A-330 G primer had the highest

mean peel bond strength (4.00 N/mm) followed by A-306 primer (2.81 N/mm) and A-304 primer (1.88 N/mm) respectively.

One-way ANOVA test was used to determine statistical significances in peel bond strengths among different primer groups. Peel bond strength among primers was statistically different ($P < 0.001$). Tukey post Hoc test further confirmed, all primers bond strength significantly different from each other. (Table 2)

Mode of failures were visually analyzed and grouped into cohesive, mixed and adhesive failures. The cohesive failure (70%) was found with specimens treated with primer A-330 and adhesive failure (80%) was recorded with primer A-304 while the mixed failure (60%) was predominantly seen with primer A-306 respectively. (Table 3)

Table 2 Descriptive values after peel bond test.

Groups	Specimens N	Mean	Standard deviation
		N/mm	SD
A-306	10	2.81	.29182
A-304	10	1.88	.21582
A-330	10	3.82	.11509

Table 3 Mode of bond failures among primer groups

Mode of Failure	Primer A-306	Primer A-304	Primer A-330
Cohesive	1 (10%)	0 (0%)	7 (70%)
Adhesive	3 (30%)	8 (80%)	0 (0%)
Mixed	6 (60%)	2 (20%)	3 (30%)

Discussion

Bonding of maxillofacial silicone elastomers to the acrylic resin is a critical factor that prolongs the functional life of maxillofacial silicone prostheses. Generally, patients remove implants retained facial prostheses by gripping a part of the prosthesis or peeling it away from the skin. Different types of forces act on prosthesis margins and interface between silicone and acrylic housing, which can lead to tearing of silicone from margins and dislodgement of acrylic housing from silicone prosthesis due to bond failure. The maxillofacial silicone elastomer and primers, selected for this in-vitro study are commonly used in maxillofacial prosthetics, and their physical and mechanical properties have been studied previously. [17, 18, 23, 24] The testing method chosen in the study gave insight knowledge of bonding force, necessary to keep intimate relationship between flexible silicone and rigid Plexiglas acrylic. The 180° peel force during removal of prosthesis can stimulate the horizontal component of de-bonding forces, leading to bond failures.

The differences in terms of bond strength within each group are due to variations in primers and adhesives compositions and their chemical affinity with silicone A-2186 and Plexiglas acrylic resin.. The peel bond strength of specimens treated with primers, A-306, A-304 and A330 ranged 2.81, 1.88 and 3.82 N/mm respectively while for the specimens treated with adhesives, Silastic adhesive type-A and A-564, ranged 1.34 and 0.53 N/mm respectively. The bond strengths for primer and adhesive combinations, A-306 & silastic adhesive type and A-306 & A564, ranged 1.74 and 1.47 N/mm respectively.

The primers consist of an organic solvent and an adhesive agent that is believed to react with both, silicone elastomer and acrylic resin materials. [25] It acts as an intermediate layer

composed of hydrophilic and hydrophobic groups, which react with functional groups of silicone elastomer. [26] Additionally, primers activate the surface of resins by etching or promoting covalent coupling and hydrogen bonding, enhancing the surface energy and wettability of the resin substrate, and penetration of the polymeric ingredients into the surface layer. [7]

The peel-testing, generated stresses in horizontal plane and the debonding proceeds through a line of junction, whereas in case of shear testing, interfacial area is stressed, with tear resistance of the silicone elastomer plays a critical role to prevent bond failures. [21] In present study peel bond strength was computed considering both the elastic deformation (λ) of prepared silicone, and the applied adhesive bonding. This formula calculates the amount absorbed energy required for deformation of the silicone and the energy used to peel the silicone away from acrylic resin. Therefore, the absorbed energy is affected by the hardness and dimensions (thickness and width) of the specimen; whereas the energy used to peel the silicone from acrylic, is affected by the interfacial thickness of adhesive primer and the area of bonding. Thus, if peeling of flexible silicone occurs with a minimum strain, the elastic energy present in the unattached tab can only be neglected. [27] In Previous studies, peel bond strength was calculated (the highest peel force was considered per unit of width), where the extension ratio was not taken into account. [22, 28] Therefore their results should be interpreted accordingly.

The types of bond failures were analyzed visually and recorded as adhesive, cohesive, or mixed failures. Among primers, the cohesive failure was predominantly found with primer A-330, mixed failure was predominant with primer A-306 and adhesive failure was more common with primer A-304 respectively (Table 3). Cohesive failures were assessed when the interfacial bond strength between silicone and Plexiglas acrylic

was higher than the silicone material strength leading to tearing of silicone. Adhesive failures were noticed when no silicone residues were found over Plexiglas acrylic after testing which represents the weak interfacial bond strength. Mixed failure was recorded when silicone residues were seen over the Plexiglas acrylic after peel testing. The Peel bond strength test has the benefit of being the only method in which bond failure proceeds at the constant controlled rate and simulates the methodology of prosthesis in service.

In case of extra-oral maxillofacial prosthesis, optimum bond strength for serviceable prosthesis is yet to be experimentally proven, however, for intra-oral prostheses, bond strength of 0.44MPa was reported sufficient for bonding acrylic denture base resin to silicone soft liners. [29] Biting forces in the posterior molar area were reported in the range of 847N and 597N for healthy young men and women respectively. [30] The normal masticatory forces were calculated about 40% of the biting forces. [31] It must be understood that Intra-oral forces are much higher than extra-oral forces; hence, the bond strength of primers and adhesives should be sufficient for functioning prosthesis.

The peel bond strength was influenced by the type of primer used in each group and the mode of surface preparation. Various studies have been done to modify the surface preparation technique to attain higher bond strength. Amin et al. [32] had reported that the bond strength between silicone and acrylic had decreased after sandblasting. Similarly, Miami et al. [25] found that surface preparation of the denture resin surface was not effective in providing long term bond strength when treated with air abrasion method. In contrast, Polyzois and Frangou [11, 12] claimed that, 80 Grit, SiC provided higher bond strengths as compared to succeeding grits of SiC paper. Therefore, in current study, Primers (A-306, A-304 and A-330)

were applied after surface preparation with 80 Grit SiC paper to increase the surface area of rigid Plexiglas acrylic. The surface treated specimens bonded with primer A-330 exhibited the highest peel bond strength 4.05N/mm while for specimens primed with A-304 and A-306 had low peel bond strength between 1.63-3.18N/mm. The SiC paper becomes less coarse with increasing number of grits, hence the 80 Grit SiC paper was coarser and deemed suitable for surface preparation of high impact Plexiglas acrylic.

Plexiglas acrylic has been used in medical field [14, 15] however its application in maxillofacial prosthetics is still unknown. Plexiglas acrylic is known for its high flexural and impact strength, minimum porosity level due to heat curing cycle and its ability to be disinfected with chemicals without affecting its physical properties. Currently, conventional denture base acrylic resin is the only acrylic type frequently used in dentistry. In this study, Plexiglas acrylic was used instead of denture base resin to assess bond strength and it has shown comparable bond strength as compared to previous studies.

Conclusions

The following conclusions can be extracted from this in-vitro study:

1. The primer A-330 provided the highest peel bond strength as compared to primers A-306 and A-304 respectively.
2. Cohesive mode of bond failure was found with primer A-330 while adhesive and mixed failures were seen with primers A-304 and A-306 respectively.
3. Plexiglas acrylic provided the comparable peel bond strength to previous studies, therefore, it might be used as alternative acrylic to auto-polymerizing acrylic for maxillofacial prosthetics.

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