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RESEARCH ARTICLE

EFFECT OF SURFACE CHARACTERIZATION AND COLOUR PIGMENT ON THE BOND STRENGTH BETWEEN MAXILLO-FACIAL SILICONE AND ACRYLIC RESIN USING CYANOACRYLATE AS BONDING AGENT: AN IN-VITRO STUDY

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ABSTRACT

Aim: To evaluate the effect of 3 surface characterizations and incorporation of color pigment into maxillo-facial RTV silicone elastomer on the bond strength with heat-cured acrylic resin using cyanoacrylate as bonding agent. **Materials and Method:** Five groups of 10 specimens of heat polymerized acrylic resin were fabricated with dimensions of 75 × 10 × 3 mm. These acrylic specimens were bonded to RTV maxillo-facial silicone using cyanoacrylate as a bonding agent. In the first group, silicone without any color pigment or characterization was bonded onto cured PMMA denture base blanks using cyanoacrylate as bonding agent; this was the control group. In the second group, silicone with added color pigment but no surface characterizations was bonded onto the acrylic blanks. In the remaining 3 groups, silicone was bonded onto acrylic blanks whose surface had been roughened with an acrylic bur in the form of retentive holes of 1 x 1.5 mm, 1.5 x 1.5 mm and 2 x 1.5 mm in dimension. The 'overlap-joint' model was used to evaluate the bond strength and the specimens were subjected to 1800peel strength test on universal testing machine according to ASTM D-903 specifications with a cross head speed of 10 mm/min until bond failure occurred. One-way ANOVA and Scheffe multiple post hoc test were carried out to detect statistical significance (p < 0.05). **Result:** Maximum bond strength was seen in the samples with surface characterization with retentive holes of 2 x 1.5 mm. The control group showed the minimum bond strength. Surface characterization in the form of retentive holes increased bond strength considerably as compared to color pigment and samples without any surface characterization. **Conclusion:** Within the limitations of the study, it was concluded that Cyanoacrylate formed a significant strong bond between maxillofacial silicone and acrylic resin. Retentive holes made on acrylic surfaces increased the bond strength considerably than those without any surface characterization and silicones with color pigment.

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INTRODUCTION

Maxillo-facial prosthetics is defined as "The art and science of anatomical, functional or cosmetic reconstruction by artificial substitutes of those regions in the maxilla, mandible and face that are missing or defective because of surgical intervention, trauma, pathology or developmental or congenital malformation".¹ Surgical reconstruction of such defects is often limited by insufficient residual soft and hard tissue and vascular compromise. Thus, a facial prosthesis presents the only attractive and practical alternative when esthetic and functional demands cannot be surgically fulfilled.^{2,3}

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Extra-oral facial prostheses used in conjunction with implants require a retentive matrix to hold the bar clips or magnets. The retentive matrix is commonly made from acrylic resin (heat-polymerizing, auto-polymerizing or light-cured materials) to which the facial silicone elastomer is attached. Hence sufficient bond strength is vital to ensure a serviceable and functional prosthesis.¹ Nowadays, the use of implants^{4,5} to retain maxillo-facial prosthesis⁶ provides excellent clinical outcomes. A retentive matrix to hold clips and magnets are mandatory when using implant retained extra-oral prosthesis. This retentive matrix is made using acrylic resin to which the elastomer of extra-oral prosthesis is processed. This demands acrylic resin matrix to be securely bonded to flexible soft material of prosthesis. That means that denture resins may be used as a rigid base into which retention components are embedded, while the facial surface supports

the silicone component of facial prosthesis.^{1,7} Maxillo-facial silicone elastomers are Dimethyl siloxane polymers and have different chemical structure to PMMA denture base resin. An adhesive primer is required to aid their bonding to the denture base.⁸ It is likely that these adhesive primers have an organic solvent and an adhesive agent that reacts with both silicone and resin materials. They activate the surfaces via etching or promoting hydrogen bonding and covalent coupling, increasing the wet ability of the substrate and by impregnating the surface layer with polymeric ingredients.⁹ The primers available commercially do serve their purpose well, but are often expensive which increases the working cost of fabricating the prosthesis; this may be a significant factor preventing patients from seeking prosthetic rehabilitation. In an in-vitro study conducted by Shetty US et al¹⁰, even though the author found cyanoacrylate resin adhesive having less bond strength compared to the bond strength of the two primers (G-611, A-330G) used in his study and also he explained different variables which may affect the bond strength (surface characterization, effect of outdoor weathering, colour pigment added to silicone) he concluded that cyanoacrylate resin has satisfactory bond strength. Further, use of cyanoacrylate resin in place of primer brings a tremendous decrease in the cost of prosthesis (a primer bottle of 10g costs around Rs.3,500/-- in Indian market, where-as a 500mg pack of cyanoacrylate resin costs only Rs.5/--). Cyanoacrylate resin (instead of regularly available primers) will be used in this study as adhesive between maxillo-facial silicone and acrylic substructure.¹⁰ Amin *et al.* reported that sandblasting the acrylic resin base weakened the bond between the resin and the silicone.⁸ Similar results were reported by Miami *et al.* who proved that roughening of the denture surface with air-particle abrasion was not effective for enhancing failure load and maintaining longevity of the silicone and acrylic resin.⁹ The best way to prepare a denture resin base in order to get maximum bond strength while attaching it with silicone is unclear. Achieving a proper color match is very important for any prosthesis to be acceptable to the patient. So it is mandatory to mix colors while fabricating prosthesis. Studies documenting the effect of incorporating color in silicone over the bond strength with the acrylic are scarce in literature. The aim of this study was to evaluate the effect of 3 surface characterization and incorporating color pigment into silicone elastomer over the bond strength using cyanoacrylate resin as bonding agent with the null hypothesis that no difference would exist in the bond strength between them.

MATERIALS AND METHODS

The bond strength between heat polymerizing acrylic resin (Dentsply India Pvt. Ltd., Gurgaon, Haryana, India) and silicone elastomer (M P Sai Enterprise, Thane, India) was evaluated using cyanoacrylate resin (Fevikwik; Pidilite Industries, Mumbai, India.) were used as bonding agents. The bond strength between silicone elastomer and heat polymerized acrylic resin specimens were tested using an acrylic resin blank as a test specimen with the dimension of 75 mm × 10 mm × 3 mm. Silicone elastomer was bonded to a part of specified area on the acrylic resin blank. A total of 50 test specimens were fabricated, which were then divided into 3 main groups (A, B, and C). Group B was further divided into subgroups B1, B2, and B3 with 10 specimens each. Specimens of group A and C had no surface characterization (plain) on the acrylic resin blank, Specimens of subgroup B1, B2 and B3 had surface characterization with retentive holes of 1 mm, 1.5 mm and 2 mm deep respectively and 1.5 mm in diameter.

Preparation of the specimens: Fifty specimens of heat polymerized acrylic resin (10 specimens for each of above mentioned three experimental groups) were fabricated using a custom made 3 piece stainless steel metal mold. This stainless steel mold consisted of 3 components: upper member, lower member and middle member. These were assembled together with the help of 4 lock screws present in the lower member. The middle member had 4 rectangular cavities of the following dimensions: 75mm length, 10mm width and 3mm thickness into which heat cure acrylic resin were packed to obtain the acrylic specimens.¹⁰(Fig- 1). In each of the 4 cavities, at the length of 25 mm, V shaped notches were incorporated so as to obtain V

shaped horizontal projections in the acrylic specimens. (Fig- 1) Heat polymerized acrylic resin was mixed and packed into the custom made mold (Fig 2). This entire assembly was placed under the hydraulic bench press at 1.25 tons of pressure for 10 minutes (Fig 2), after bench curing for 30 minutes, the mold was placed in a digital acrylizer and cured according to manufacturer's instructions. After bench cooling for 12 hours, the metallic mold was dis-assembled and the plain acrylic resin blanks without any surface characterization were retrieved carefully from the middle member for the group A and C. They were finished and polished using sandpaper.(Fig 3)

Fabrication of acrylic resin blank for the subgroup B1, B2 and B3: Plain acrylic blanks were fabricated as mentioned before for the group A and C. After the acrylic blanks were finished and polished a line was drawn with the help of metallic scale and pencil corresponding to the V shaped projections. This line demarcated the acrylic blank into 2 parts, A and B; Part A with a length of 25 mm, and Part B with a length of 50 mm. An adhesive tape was applied onto Part-B of the acrylic blank so that part-A remained exposed to be subjected for subsequent bonding with the maxillo-facial silicone. Horizontal lines were marked in this area at a distance of 3 mm and vertical lines were marked at a distance of 2.5 mm. At the intersection of these lines, 24 holes were made with depth of 1 mm, 1.5 mm and 2 mm respectively for subgroup B1, B2 and B3 using a round tungsten carbide bur of diameter 1.5 mm. (Fig- 4). On the surface of Part-B, two horizontal lines and two vertical lines were drawn 3 mm and 5 mm from the borders respectively. At the intersection of these lines, 4 orientation indentations were made with diameter of 1.5 mm and depth of 1.0 mm using a round tungsten carbide bur. These indentations helped in re-orienting the silicone strips in the same original position over the acrylic blanks after application of cyanoacrylate bonding agent.

Preparation of mold for Silicone samples: On the acrylic resin blanks fabricated above (with and without surface modifications), another acrylic blank of the same dimension (75 mm × 10 mm × 3 mm in length, width and thickness respectively) was overlapped and the borders were sealed neatly with wax to seal the gap between the two blanks. The combined thickness of both the blanks was 6 mm. The fused acrylic blanks were flaked with the first pour covering till the junction of the two blanks. Once the dental plaster was set, petroleum jelly was applied all over followed by the second pour with dental stone and the flask was clamped. After 1 hour, the flask was opened, dewaxed and the overlapped acrylic blank from the upper member of the flask was removed from the mold and discarded. The lower member of the flask contained the acrylic blank with the required surface modification and the upper member of the flask contained the mold in which silicone was packed without cyanoacrylate and cured. (Fig 5) The dental flask was clamped once again and bench cured for 72 hours for the complete polymerization of silicone material to occur.

After 72 hours, the dental flask was opened to retrieve the silicone strips and acrylic blanks from the mold. (Fig 6). For group A, the silicones were weighed using precision digital scale (Shimadzu, Shimadzu Corporation, Shimadzu Philippines Manufacturing INC. (SPM) The pigment (yellow Artist's oil color) weight was equivalent to 0.2 %¹² of the weight of the silicone. Pigment was mixed with the silicone on a glass slab with the help of a stainless steel spatula until a homogenous mixture was obtained. The silicone was then packed in the master mold and the excess was removed with a spatula to maintain a regular thickness. After 72 hours, the silicone strips and acrylic blanks were retrieved from the mold.

Preparation done prior to bonding of silicone strips to the acrylic blanks: All the acrylic blanks with and without surface modifications were initially cleaned with water. The uncovered area of acrylic blank was cleaned with acetone for 10 seconds and then left to air dry for 5 minutes. Cyanoacrylate resin was applied on to Part-A of acrylic resin blank to be bonded to silicone and the cured silicone strip was placed immediately in its correct position using the four orientation grooves on Part-B of the acrylic resin blank.



Fig.1. Custom made stainless Steel mold (lower, middle and upper member), V shaped notch

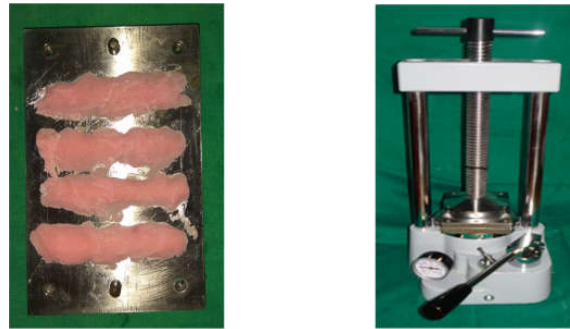


Fig.2. Acrylic resin in dough stage packed and placed under hydraulic bench press

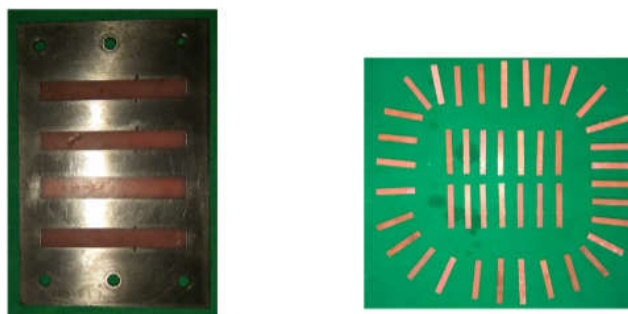


Fig. 3. Retrieval of acrylic resin blanks and finished



Fig. 4. Surface characteristics of acrylic substrate



Thickness of Group B1 sample 1 mm holes

Thickness of Group B2 sample 1.5 mm holes

Fig. 5. Fabrication of mold for silicone

A weight of 1 kg was placed on the specimens for 15 minutes. (Fig 7) The procedures mentioned above were carried out with all the 50 specimens. All the test groups were subjected to a 180° peel strength test on Hounsfield universal testing machine. The test was carried out according to the ASTM D-903 specification. In each specimens, the silicone strip was bonded to acrylic denture base at one end (25 mm×10 mm×3 mm) and left free at the other (50 mm×10 mm×3 mm). The free end of the strip was turned back at 180° so that the hard acrylic base was clamped in the lower clamp and the soft free silicone strip was gripped in the upper clamp (Fig 8). The force needed to cause bond failures was recorded. Peel strength (N/mm)¹⁰ was determined using the formula

$$\text{Peel strength} = F / W (1 + \lambda / 3)$$

Where F = maximum force recorded (N)

W = Width of specimens (10 mm)

λ = Extension ratio of silicone elastomer

(The ratio of stretched to primary length)

The results obtained were then subjected to statistical analysis using ANOVA. Pair wise comparison with respect to bond strength was analyzed using Scheffe multiple comparison test procedures. Statistical Package for Social Sciences (SPSS 21) was used to analyze the data.

RESULTS

Analysis for comparison of all groups and surfaces with respect to peel strength revealed that there was statistically significant difference in the peel strengths between the groups (P= .000). The mean peel strength was highest among B3 group (4.26 ± 1.1) and least among C group (1.33 ± 0.2) as shown in Table 1. Further analysis of pair wise comparison among various groups with respect to peel strength using post hoc analysis revealed that statistically significant difference between B3 group and control group (P= .000) and between group B2 and control group (P=.025) respectively as shown in Table 2.

DISCUSSION

Maxillofacial materials are used to replace facial structures lost through disease or trauma. The prosthesis is commonly fabricated with heat-polymerizing, auto-polymerizing, or VLC resin to which the silicone facial elastomer is attached.⁴ The bond of silicone elastomer to the acrylic resin component must be sufficiently tenacious to withstand the substantial forces acting upon the bond interface, not only during placement and removal of the prosthesis, but also during mold opening and deflasking procedures as this is the weakest link in the restoration.

Table 1. Comparison of various groups considered in the study

GROUP	N	MEAN	SD	MINIMUM	MAXIMUM	P
A	10	1.50	.84327	.50	3.10	0.000*
B1	10	1.99	.62279	1.37	2.96	
B2	10	2.52	.69459	1.55	3.83	
B3	10	4.26	1.10040	2.55	5.90	
C	10	1.33	.28756	.89	1.81	
TOTAL	50	11.6	3.548661	6.86	17.6	

Table 2. Pair wise comparisons of various groups with respect to peel strength by Scheffe multiple post hoc analysis

(I) group	(J) group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
A	B1	-.49000	.33912	.720	-1.5791	.5991
	B2	-1.02100	.33912	.077	-2.1101	.0681
	B3	-2.76100*	.33912	.000	-3.8501	-1.6719
	C	.17000	.33912	.992	-.9191	1.2591
B1	A	.49000	.33912	.720	-.5991	1.5791
	B2	-.53100	.33912	.655	-1.6201	.5581
	B3	-2.27100*	.33912	.000	-3.3601	-1.1819
	C	.66000	.33912	.446	-.4291	1.7491
B2	A	1.02100	.33912	.077	-.0681	2.1101
	B1	.53100	.33912	.655	-.5581	1.6201
	B3	-1.74000*	.33912	.000	-2.8291	-.6509
	C	1.19100*	.33912	.025	.1019	2.2801
B3	A	2.76100*	.33912	.000	1.6719	3.8501
	B1	2.27100*	.33912	.000	1.1819	3.3601
	B2	1.74000*	.33912	.000	.6509	2.8291
	C	2.93100*	.33912	.000	1.8419	4.0201
C	A	-.17000	.33912	.992	-1.2591	.9191
	B1	-.66000	.33912	.446	-1.7491	.4291
	B2	-1.19100*	.33912	.025	-2.2801	-.1019
	B3	-2.93100*	.33912	.000	-4.0201	-1.8419



Fig. 6. Preparation of silicone specimens

The chemical structure of maxillofacial silicone elastomers (dimethyl siloxane polymers) and PMMA denture base resin is different, exhibiting poor bond characteristics. Hence, primers are provided to increase the bond strength between silicone elastomer and acrylic resin.¹⁰ The primer molecules collectively serve as a chemical intermediate of the silicone and resin substrate by swelling the surface and improving wettability of the substrate via organic solvents, promoting hydrogen bonding, covalent coupling, and the formation of an interpenetrating network (IPN) at the boundary interphase.¹³ Thereby preventing delamination of silicone and enhancing the longevity of the prosthesis. The primers available commercially do serve their purpose well, but are often expensive which increases the working cost of fabricating the prosthesis; this may be a significant factor preventing patients from seeking prosthetic rehabilitation. This study was done to find out the effect of different surface characterization and incorporating color pigment into silicone over the bond strength using cyanoacrylate as bonding agent. A study showed cyanoacrylate produced significant bond strength over the primer¹.

Bond strength can be measured and evaluated by tensile test, shear test and peel test.¹⁴ A horizontal component of detaching forces are generated when the patient removes the craniofacial implant retained prosthesis. This type of force is well simulated in the peel test.^{3,4} For this reason, 180° peel test was used in this study to evaluate the bond strength. In the present study, the type of bond failure was assessed visually and designated as adhesive, cohesive or mixed. Test showed that the experimental groups B1, B2 and B3 failed cohesively while the control group (C) and group A showed mixed bond failure. For cohesive failures, the peel bond strength between the silicone and the denture base was stronger than the strength of the silicone material. Although the peel test has the advantage of being 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. It is likely that the cohesive failures were initiated by small imperfections or voids in the silicone mixture. Specimens of group B3 and B2 showed good bond strength with mean peel strength of 4.26 N/mm and 2.52 N/mm, respectively.

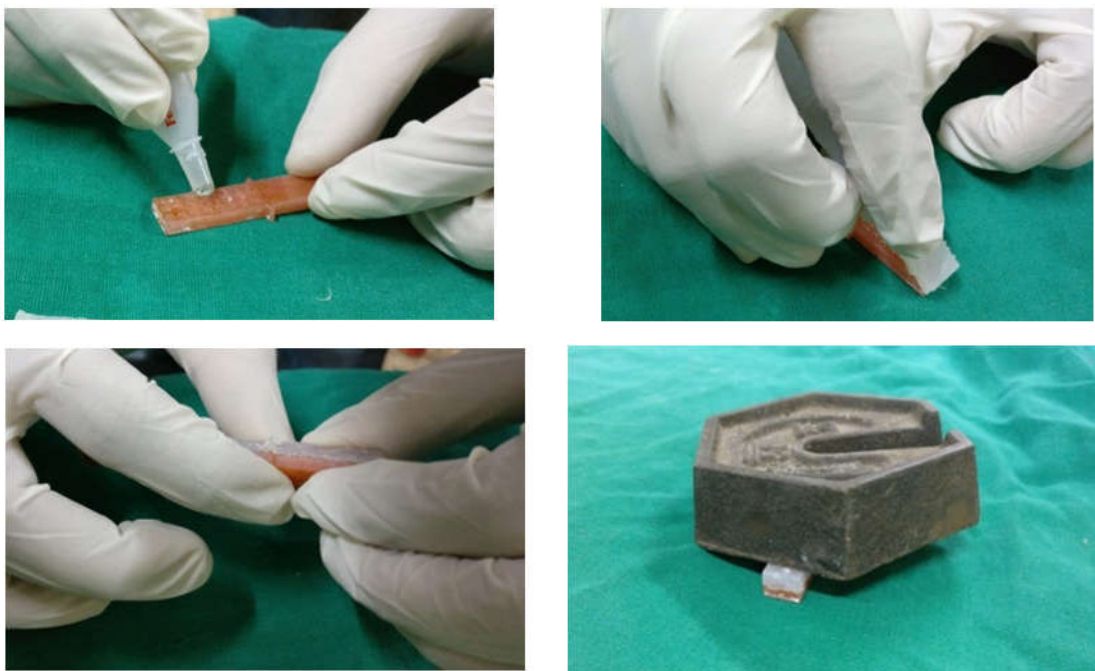


Fig.7. Bonding of silicone strips

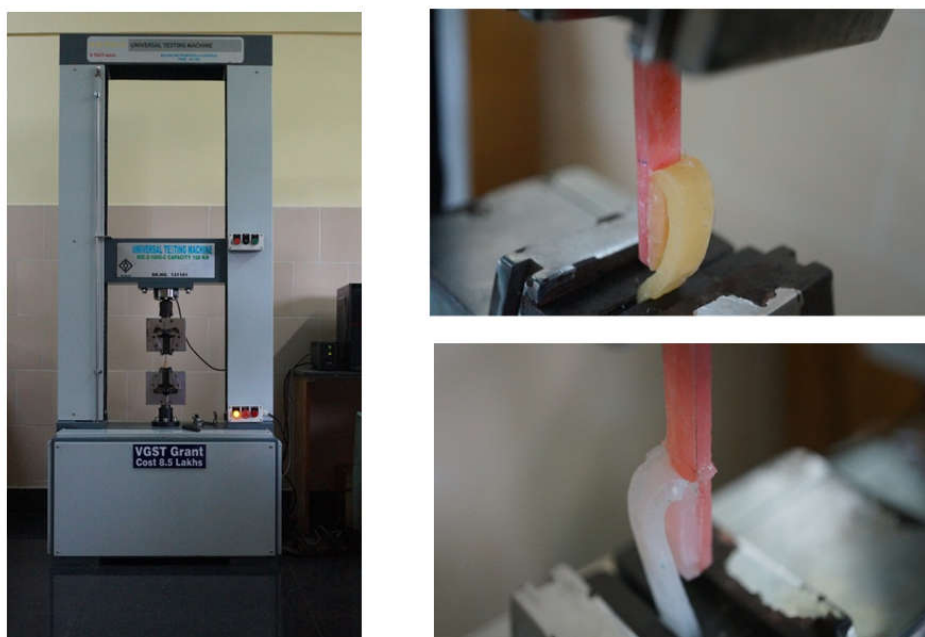


Fig. 8. Silicone peeled off from the acrylic resin substrate during 180° Peel test under UTM

Although previous study conducted by Shetty US et al¹⁰, showed lower bond strength of 2.65 N/mm which was significantly less than the bond strength produced by cyanoacrylate. Findings of the study conducted by Hatamleh and Watts¹, showed lower bond strength of 1.30 N/mm and 2.36 N/mm using the primer A-330G, which was in accordance with our study. The increased bond strength of cyanoacrylate could be due to the mechanical interlocking of peg like extension of silicone material into the holes made on acrylic substrate, also the provision of holes provided increased surface area for silicone elastomer to bond with acrylic resin substrate. Craig and Gibbons¹⁵ advocated a roughened surface to improve the adhesive bond. They reported that adhesive values obtained by roughening were approximately double those of smooth surfaces because of a slightly irregular surface provided mechanical locking for the soft material. On contrary, Jagger et al.¹⁶ 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. However, if the surface roughening is done in a definite pattern, with adequate intervals of plain surface and roughness, then the possibility of stress concentration and weakening of the acrylic resin substrate may be reduced. In the present study specimens showed some variations in the bond strength with surface characteristics which could be due to the method of sample fabrication, where silicone is peeled from the acrylic substrate from the bonding area, bonding agent is applied and the silicone elastomer had to be placed back in the same original position so that the irregularities, elevations and depressions of the two materials will correspond to each other, to overcome this, four orientation grooves were made on the non- bonding area of the acrylic substrate to orient the silicone in the same original position to avoid area of voids and spaces between the acrylic and silicone elastomer, but it is difficult to eliminate it completely, producing a weak bond between the two which could be a limitation of this study.

Conclusion

Within the limitations of this study, the following conclusions were drawn.

- Cyanoacrylate formed a statistically significantly stronger bond ($p=0.001$).
- The use of retentive holes significantly increased the bond strength in comparison to specimens with no surface characteristics.
- Among all the groups tested, group B3 Constituted acrylic blanks with surface characterization in the form of retentive holes of 2.0 mm produced the highest bond strength.
- The effect of color pigment and surface characterization in the form of retentive holes of 1.0 mm had no significant effect on bond strength.

Hence cyanoacrylate resin can be used as a bonding agent with surface characterization to achieve significantly stronger bond between resin and silicone for maxillofacial prosthesis. Further research is also required to evaluate the long term effect of cyanoacrylate on both acrylic resin and silicone elastomer along with its bond strength.

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