

Evaluation of the effect of ultraviolet light polymerized clear adhesive on shear and tensile bond strength of heat cure denture base resin to the cobalt-chromium retentive minor connector: An *in vitro* study

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Abstract

Aim: The aim of this study is to evaluate the efficacy of new material ultraviolet (UV) light polymerized clear adhesive on shear and tensile bond strength of heat cure denture base resin (Polymethylmethacrylate (PMMA)) to cobalt-chromium (Co-Cr) retentive minor connector.

Setting and Design: Comparative evaluation- In-vitro study.

Materials and Methods: Sixty samples of Co-Cr plates mimicking minor connectors were fabricated. Thirty samples were coated with new material UV light polymerized clear adhesive and cured under UV light source for 10 min. In gun-metal flask, metal plates were placed in the lower compartment over it. Heat cure acrylic resin was packed in the dough stage with the help of clamps and processed according to the manufacturer's instructions. Samples were kept in artificial saliva for 90 days. Shear and tensile bond strengths were calculated of each sample with a Universal testing machine, and results were statistically analyzed. Type of bond failure was observed for each sample under stereomicroscope.

Statistical Analysis Used: Unpaired t test.

Results: Tensile bond strength, as well as shear bond strength, showed that 0.93 N and 1.64 N respectively for without application of new adhesive was more as compared to that of samples with the application of new adhesive which is 0.75 N and 1.54 N respectively. Bond failure was found to be an adhesive failure in resin-metal interface.

Conclusions: Excellent bonding seen between the new adhesive and acrylic interface but limited effect on the metal interface. To increase bond strength between metal and resin interface, some surface treatment with the metal surface is needed to increase the bonding of the new adhesive to the metal surface.

Keywords: AcResin A250 ultraviolet, adhesive, bond failure, cast partial denture, PMMA, ultraviolet light cure

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INTRODUCTION

In removable prosthodontics, removable partial denture is given as a temporary prosthesis, followed by the definitive prosthesis, i.e., cast partial denture (CPD). CPD are commonly fabricated with acrylic resin and metal. Base metals, such as cobalt-chromium alloy (Co-Cr alloy), are commonly used for CPDs that contain metal frameworks, bars, or clasps. Heat polymerized acrylic resin is used to fabricate denture base for removable prostheses.^[1]

The minor connector of the CPD joins the metal framework to denture base resin. The minor connector helps to increase mechanical retention to the resin. The minor connector attaches to acrylic denture by either mechanically or chemically or by both. Despite these retentive elements, functional forces often result in failure of the acrylic resin at the junction to the framework.

Vermeulen *et al.* reported a fracture percentage of 17% after 5 years, increasing to 35% after 10 years.^[2] Körber *et al.* found a repair percentage of 40% after 5 years, of which 15% was caused exclusively by fractures of metal parts.^[3]

Numerous investigations have been carried out to observe the bond strength of denture base resin to the metal. In CPD, mechanical retention can be provided between denture base resin and the framework with the help of bars, mesh, and lattice, but they were not sufficient to prevent microleakage. Various methods and techniques, such as silica coating, chemical etchants, spark erosion, and tin-plating, adhesive primers, have been tried to increase bonding of the resin to the metal.^[4] Hence, a combination of both techniques increases the longevity of the prosthesis.

acResin A 250 ultraviolet (UV) (Polyacrylate), acrylic hotmelt (acrylic ester) is UV light polymerized adhesive. This material has been used in medical applications like medical tapes but has not been used in dentistry before. It has advantageous properties such as resistance to aging and heat, resistance to humidity and water, low odor, clear to clear application, no skin irritation, not cytotoxicity, and minimally allergic.

There is a need to evaluate the bond strength of this new material as no study has been carried out regarding this material till date.

This study is to evaluate the bond strength of heat cure denture base resin (PMMA) to Co-Cr retentive Minor connector with and without using a new material-UV light polymerized adhesive. The aim of this study was to

evaluate the efficacy of new material UV light polymerized clear adhesive on Shear and Tensile bond strength of heat cure denture base resin (PMMA) to Co-Cr retentive Minor connector. Objectives of this study were to compare tensile and shear bond strength of heat cure acrylic resin (PMMA) to Co-Cr retentive Minor connector with and without using UV light polymerized clear adhesive and to determine the type of bond failure between the metal plate and UV light polymerized adhesive versus bond failure between heat cure acrylic resin (PMMA) and UV light polymerized adhesive. The null hypothesis was that there is a decrease in tensile and shear bond strength between PMMA and Co-Cr alloy minor connectors in CPD on the application of UV light polymerized clear adhesive.

MATERIALS AND METHODS

Sample fabrication

Total sixty samples of Co-Cr alloy plates (20 mm × 20 mm × 1.5 mm) with the central area of the plate was incorporated with mesh (15 mm × 15 mm × 0.5 mm) and inner each square of mesh are of about 2 mm × 2 mm) [Figure 1] mimicking mesh type of minor connector were fabricated, which was made with computer-aided designing and then fabricated by using Laser printing and sintering [Figure 2].

AcResin A 250 UV adhesive (BASF, Germany) is hotmelt material [Figure 3] with high viscosity, and hence, it was melted at temperature 100°C–120°C in the heating pot, and ethyl alcohol solvent was added in a small amount for ease of application (as a thinner) which got evaporated after application. Then, it was applied in a thin layer with a metallic spatula over the thirty Co-Cr plates and was polymerized with UV-C light (Wavelength 256 nm) for 20 s. The source for UV-C light used was laminar flow [Figure 4].

Custom made Gunmetal flask was fabricated with dimensions of 36.5 mm × 30 mm × 30 mm. It was made

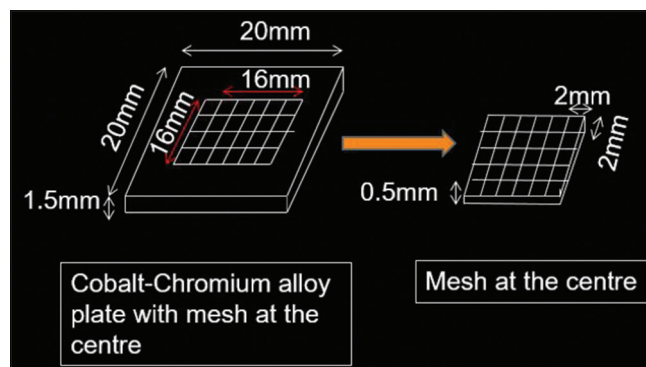


Figure 1: Schematic representation of the cobalt-chromium alloy plate with mesh

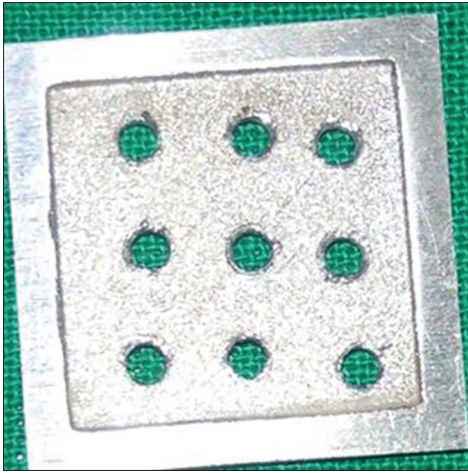


Figure 2: Cobalt-chromium metal plate with mesh

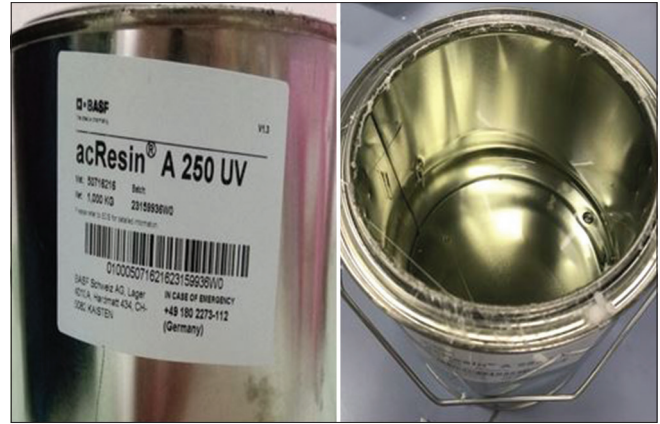


Figure 3: acResin A 250 ultraviolet adhesive, BASF, Germany

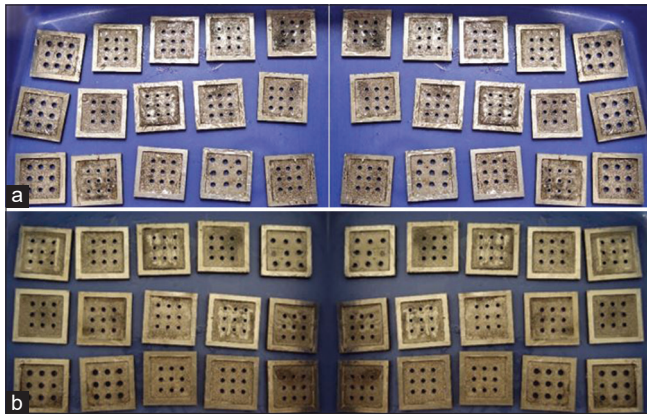


Figure 4: Samples treated with acResin A250 ultraviolet adhesive (a) Samples after application of acResin A 250 ultraviolet, (b) Samples after polymerization under ultraviolet light source)



Figure 5: Custom-made gun-metal flask

in three compartments. Lower compartment secured Co-Cr plate. In middle compartment, space was made for heat cure acrylic resin, while the upper compartment was used as a lid [Figure 5].

Co-Cr alloy plate was placed inside the lower compartment, and then custom made flask was packed with heat cure acrylic resin (DPI, India) in the dough-like stage [Figure 6]. The flask was then clamped with C clamp, and intermittent pressure was applied. After bench curing for 1 h, processing was done in the Wasserman heat curing unit according to the manufacturer's instruction. After complete polymerization and cooling of flask specimens were retrieved. After polymerization, samples were placed in artificial saliva (Salivart, India) for 90 days. All samples were later subjected to finishing and polishing [Figure 7].

Study design

Sixty samples were divided into four groups, each containing 15 samples. They were categorized as Group I,

Group II, Group III, and Group IV [Table 1] depending on with and without application of new adhesive and tests to be carried out. Thirty samples (Group III and Group IV) were tested for tensile bond strength. It was determined by loading the bonded specimens to failure on universal testing machine. Sample size were determined using following/ formula and previous studies:

$$n = \frac{(SD_1^2 + SD_2^2) \times 7.84 / 13}{(X_1 - X_2)^2}$$

Specimens were loaded at a crosshead speed of 5 mm/min. Thirty samples (Group I and Group II) were tested for shear bond strength. It was measured using a screw-driven universal testing machine at a crosshead speed of 1.0 mm/min. In both testing groups, specimens were fractured at the junction between acrylic and Co-Cr plate [Figure 8]. Means and standard deviations (SDs) were calculated for each test group. These readings were utilized as data for the statistical analysis. After testing, debonding surfaces were observed through stereomicroscope in $\times 45$ for the identification of mode of failure. Adhesive failure at the

resin-metal interface, cohesive failure within resin material & combination of adhesive and cohesive failure were analyzed. Comparisons were carried out between samples with and without application of adhesive, which were subjected to shear and tensile bond strengths.

RESULTS AND STATISTICAL ANALYSIS

Fifteen samples for testing shear bond strength with the application of adhesive is compared with another

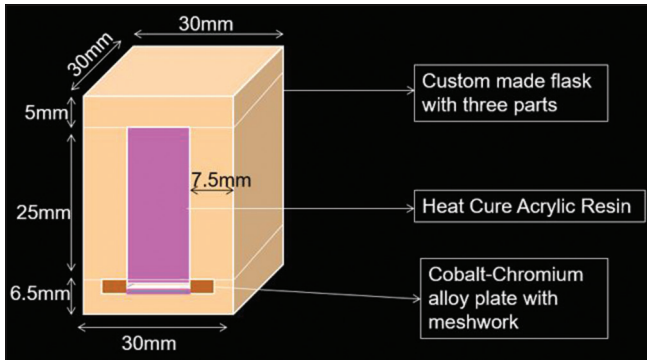


Figure 6: Schematic representation of custom made flask design with material



Figure 7: Fabrication of samples (a) Samples of study group, *n* = 30; (b) Samples of control group, *n* = 30

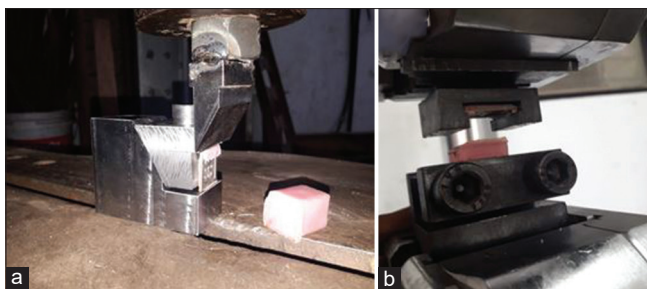


Figure 8: Separation of the cobalt-chromium plate from acrylic after testing (a) For testing shear bond strength, (b) For testing tensile bond strength)

15 samples without the application of adhesive. Similarly, 15 samples for testing tensile bond strength with the application of adhesive are compared with another 15 samples for testing tensile without the application of adhesive. Shear and tensile bond strength values for 60 samples were recorded in MPa [Table 2].

Descriptive statistics for tensile and shear bond strength were expressed as mean ± SD. Two groups (with adhesive and without adhesive) were compared by the unpaired *t*-test. In the above tests, *P* ≤ 0.05 was taken to be statistically significant. All analyses were performed using SPSS (Statistical Package for Social Sciences) software version 20 (SPSS Inc., IBM, India).

The compared data were subjected to statistical analysis at a 95% confidence level, and results were interpreted as *P* > 0.05 – Nonsignificant and *P* ≤ 0.05–Significant.

The mean difference in mean tensile strength (KN) with adhesive (0.7473 ± 0.16078) and without adhesive (0.9267 ± 0.19025) and was - 0.17933 with

Table 1: Table depicted distribution of 60 samples

Groups	Samples
Group I	15 samples for testing shear bond strength without application of UV light polymerized acResin adhesive
Group II	15 samples for testing shear bond strength with application of UV light polymerized acResin adhesive
Group III	15 samples for testing tensile bond strength without application of UV light polymerized acResin adhesive
Group IV	15 samples for testing tensile bond strength with application of UV light polymerized acResin adhesive

UV: Ultraviolet

Table 2: Tensile and shear bond strength values for both the groups, i.e., with and without application of ultraviolet light polymerized acResin adhesive and means of each group

Serial number	Group I (MPa)	Group II (MPa)	Group III (MPa)	Group IV (MPa)
1	1.4	1.4	1	1
2	1.5	1.3	0.6	0.92
3	1.6	1.6	1.29	0.57
4	2.2	1.9	1.31	0.74
5	1.5	1.3	1.06	0.41
6	1.5	1.5	0.82	0.66
7	1.5	1.4	0.91	0.81
8	1.4	1.2	0.87	0.88
9	1.7	1.6	0.82	0.76
10	1.6	1.5	0.95	0.66
11	1.7	1.7	0.94	0.55
12	1.5	1.4	0.8	0.68
13	1.8	1.8	0.83	0.89
14	1.8	1.6	0.98	0.79
15	1.9	1.9	0.72	0.89
Average	1.64	1.54	0.93	0.75

standard error (SE) = 0.06431 which was found statistically significant $P = 0.009$ ($t = -2.788$ $df = 28$) (confidence interval: -0.31108 ; -0.04759) [Tables 3 and 4].

The mean difference in mean shear bond strength (KN) with adhesive (1.5400 ± 0.21647) and without adhesive (1.6400 ± 0.21647) and was -0.1000 with $SE = 0.07904$ which was found statistically insignificant $P = 0.216$ ($t = -2.788$ $df = 28$) [Tables 5 and 6].

The type of bond failure was the adhesive type of failure when observed under stereomicroscope. It was an adhesive failure from the metal side of the sample as material found to be polymerized well with the resin portion of the sample [Figures 9 and 10].

The results and statistical analysis of all the four groups are tabulated in Table 2, and the graphical representation of the same is shown in Figure 11.

DISCUSSION

The current project is focused on bond strength between

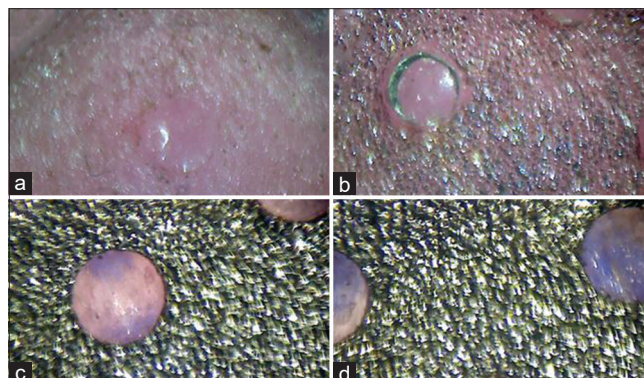


Figure 9: Stereomicroscopic images of samples after debonding. (a) Acrylic portion of sample with the application of adhesive after debonding. (b) Acrylic portion of the sample without application of adhesive after debonding. (c) Metal plate portion of the sample with the application of adhesive after debonding. (d) Metal plate portion of the sample without application of adhesive after debonding

Table 3: Descriptive statistics tensile bond strength (with and without application of adhesive)

Groups	n	Descriptive statistics		
		Minimum	Maximum	Mean±SD
With application of adhesive (KN)	15	0.41	1.00	0.7473±0.16078
Without application of adhesive (KN)	15	0.60	1.31	0.9267±0.19025

SD: Standard deviation, KN: Kilo-Newton

Table 4: Comparison tensile bond strength of heat cure acrylic resin poly (methyl methacrylate) among with and without application of adhesive by unpaired t-test

With application versus without application (mean±SD) Tensile strength (KN)	t	df	Unpaired t-test				
			Significant (two-tailed) P	Mean difference	SE difference	95% CI of the difference	
						Lower	Upper
0.7473±0.16078 0.9267±0.19025	-2.788	28	0.009*	-0.17933	0.06431	-0.31108	-0.04759

*Statistically significant. df: Degree of freedom, SE: Standard error, t: t table value, CI: Confidence interval, P: Probability value, SD: Standard deviation, KN: Kilo-Newton

Table 5: Descriptive statistics shear bond strength (with and without the application of adhesive)

Groups	n	Descriptive statistics		
		Minimum	Maximum	Mean±SD
With application of adhesive (KN)	15	1.20	1.90	1.5400±0.21647
Without application of adhesive (KN)	15	1.40	2.20	1.6400±0.21647

SD: Standard deviation, KN: Kilo-Newton

Table 6: Comparison shear bond strength of heat cure acrylic resin poly (methyl methacrylate) among with and without application of adhesive by unpaired t-test

With application versus without application (mean±SD) Shear strength (KN)	t	df	Unpaired t-test				
			Significant (two-tailed) P	Mean difference	SE difference	95% CI of the difference	
						Lower	Upper
1.5400±0.21647 1.6400±0.21647	-1.265	28	0.216	-0.10000	0.07904	-0.26191	0.06191

*Statistically significant. df: Degree of freedom, SE: Standard error, t: t table value, CI: Confidence interval, P: Probability value, SD: Standard deviation, KN: Kilo-Newton

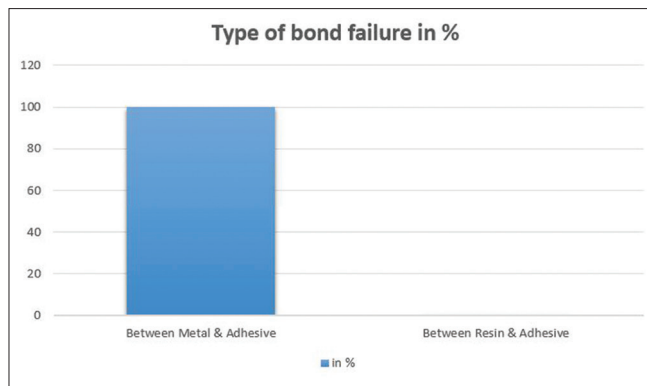


Figure 10: Analysis of the type of bond failure

the metal framework of minor connector and acrylic denture base over it. For standardization, the entire research project was handled by a single operator using a gunmetal custom made flask for polymerization under the same temperature and pressure and metal plates, which were laser printed and sintered of equal dimensions to simulate it as a mesh type of minor connector.

Even though there is a incorporation of various mechanical retentive elements, failure of the acrylic resin occurs at the junction with framework due to stress concentration within resin on application of functional forces. Along with mechanical retention, chemical bonding between metal framework and denture base resin is a necessity which can increase bonding between them.^[1]

Lack of chemical bond between denture base resin and metal framework leads to microleakage because of the different coefficient of thermal expansion of metal and resin. This causes the accumulation of oral debris and microorganisms, discoloration, and staining of margins of metal resin interface, resulting in potential adhesive failure and fracture at the finishing line and unfavorable soft-tissue response. All mechanical retention systems are insufficient to prevent microleakage. Furthermore, forces during function often result in failure of CPD at acrylic resin and metal framework interface due to stress concentration within the resin.^[1,2]

acResin A 250 UV is intended for the production of pressure-sensitive adhesives UV-reactive, solvent-free acrylic copolymer. It has an excellent adhesive property with bond strength approx. 20–30 N. When this adhesive is irradiated with UV-C light, the potentially reactive groups attached to the chains form cross-links with neighboring poly-acrylate chains to achieve desirable bonding between them. UV-C sensitive photoreactive groups are an integral part of the polymer and, they are non-volatile, so no products of potential toxicological concern are released. Hence, mainly suitable for its use intraorally.

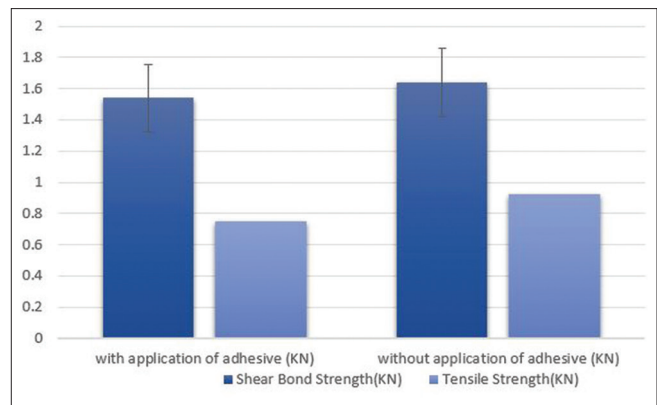


Figure 11: Comparison of tensile and shear bond strength of heat cure acrylic resin (PMMA) among with and without the application of adhesive

To mimic minor connector, in this study, Co-Cr metal plates were fabricated.^[3,4] In general, minor connectors should be elevated at least 1 mm from the tissue surface to allow the metal framework to be adequately embedded in the acrylic resin, which provides strength to support the acrylic resin.^[5]

Mesh type has multiple vents that extend over the crest of the residual ridge. It is used when multiple teeth have to be replaced. It shows more difficulty in packing acrylic resin in the dough stage because more pressure is needed against resin to force it through small holes. However, it does not provide a strong attachment for denture base.^[6] Hence, so as to overcome this, it is necessary to use chemical bond along with mechanical retention to increase bond between the metal framework and acrylic denture base.

In this study, all the samples were placed in artificial saliva for 90 days. Artificial saliva often used as an oral simulation medium because of their electrochemical properties that are similar to those of natural saliva.^[7] Furthermore, for complete polymerization of PMMA, about 90 days are required. Hence, samples were placed in artificial saliva for 90 days.

Two parameters were used, shear and tensile bond strength. Shear bond is the strength of a material or component against the type of yield or structural failure when the material or component fails in shear. While tensile strength of a material is the maximum amount of tensile stress that it can take before failure, which shows the force required to cause debonding between acrylic, adhesive, and metal framework in this case.^[8]

Results of tensile bond strength as well as shear bond strength showed that strength of samples without application of adhesive was more as compared to that of samples with the application of adhesive. Hence, null hypothesis was accepted.

For shear bond strength, mean for Group I is 1.64 MPa is more than that of the mean of Group II is 1.54 MPa. While for tensile bond strength, the mean for Group III is 0.93 MPa is more than that of the mean of Group IV is 0.75 MPa [Table 1].

The difference in tensile bond strength with or without the application of acResin adhesive was found to be statistically significant.

Tensile bond strength is mainly concerned about bonding strength of acrylic and metal plate as it is directed in the vertical direction.^[9,10] As in this study, with the application of adhesive is decreasing tensile bond strength compared to without application of adhesive, this means the application of adhesive is not able to bond properly with either acrylic or with metal plate. As in this study, with the application of adhesive tensile bond strength is decreasing compared to without application of adhesive, this means application of adhesive is not able to bond properly with acrylic as well as with metal plate.

The difference in shear bond strength with or without the application of acResin adhesive was found to be statistically insignificant.

Shear bond strength is related to horizontal forces, which are decreased due to the application of adhesive. Shear bond strength is mainly associated with a bond strength of the adhesive itself.^[8,11,12]

Bond failure was found to be an adhesive failure in resin–metal interface. On examination, under stereomicroscope, adhesive was seen to be bonded with the acrylic portion of sample. Adhesive was found to be polymerized with acrylic. Bond failure was the adhesive type of bond failure from the metal side of the sample as the material was not bonded to the metal plate [Figure 11]. It came to be 100% bond failure between metal and adhesive while, no bond failure between resin and adhesive.

Because of a lack of bonding of acResin with metal, it showed a reduction in bond strength, and hence, adhesive forming a separate layer between metal plate and acrylic, leading to a decrease in bond strength.

However, adhesive was polymerized with acrylic, the bond between them was so strong that even after application of 1.9 N of force, samples were fractured from the interface of the metal framework and adhesive.

So to increase the effect of this adhesive in CPD, any conditioning or surface treatment of the metal surface

of the framework is needed like sandblasting, etchant application, or application of silane agent. This will help to increase the bonding of this adhesive to metal.^[7, 13-15]

The bond strength of denture base resin is significantly increased by application to base metal alloys of metal conditioners containing functional monomers designed for bonding, such as 4-methacryloyloxyethyl trimellitic acid anhydride and 10-methacryloyloxydecyl dihydrogen phosphate, to the titanium alloys and Co-Cr alloy.^[8,16-18]

However as the bond between acrylic and adhesive is much good, it can be used in other treatment plans like rebonding of acrylic teeth to denture base and in denture repairs.

The need of additional retentive mechanisms is needed to increase the bond between metal and resin; that was the limitation of this study.

Halim stated that PMMA showed the highest bond strengths to both flat and beads, and bond strength of acrylic resin to titanium not pretreated with the bonding agent was gradually decreasing down from flat surface lattice-to-mesh-to-beads.^[5,19-21]

Kim and Vang stated that the use of appropriate adhesive metal primers makes it possible not only to eliminate the need for surface preparation of the metal framework before applying the heat cure resins but also reduce the need for retentive devices on the metal substructure.^[11,22-24]

Furthermore, studies can be done using this acResin UV cured adhesive resin to improve bonding between resins with a different application.

CONCLUSIONS

Within the limitations of the study, the following conclusions were drawn:

1. The new material (acResin 250 UV adhesive) is an excellent adhesive used in the Medical field but limited use in enhancing chemical bonding between metal and acrylic interface in CPD
2. There was no significant difference found in shear and tensile bond strength between samples of with and without application of adhesive resin. Hence, the null hypothesis is accepted in this study
3. There is excellent bonding between the adhesive and acrylic interface but limited effect on the metal interface
4. To increase bond strength between metal and resin interface, some surface treatment with the metal surface is needed to increase the bonding of adhesive to the metal surface

5. Further scope of the study: As acResin 250 UV showed polymerization with resin, it can be used in other applications to carry out bonding within resins like instant denture repair, rebonding of denture teeth, etc.

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Nil.

Conflicts of interest

There are no conflicts of interest.

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