

Comparative evaluation of effect of metal primer and sandblasting on the shear bond strength between heat cured acrylic denture base resin and cobalt-chromium alloy: An *in vitro* study

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Abstract

Aims: The aim of this study was to evaluate the effect of metal primers and sandblasting on the shear bond strength (SBS) of heat cured acrylic denture base resin to cobalt-chromium (Co-Cr) alloy. **Materials and Methods:** A total number of 40 disk shaped wax patterns (10 mm in diameter and 2 mm in thickness) were cast in Co-Cr alloy. Samples were divided into 4 groups depending on the surface treatment received. Group 1: No surface treatment was done and acts as control group. Group 2: Only sandblasting was done. Group 3: Only metal primer was applied. Group 4: Both metal primer and sandblasting were done. After surface treatment samples had been tested in Universal Testing Machine at crosshead speed of 0.5 mm/min in shear mode and scanning, electron microscope evaluation was done to observe the mode of failure. **Statistical Analysis:** All the observations obtained were analyzed statistically using software SPSS version 17; one-way analysis of variance (ANOVA) and *post-hoc* Tukey test were applied. **Results:** The one-way ANOVA indicated that SBS values varied according to type of surface treatment done. The SBS was highest (18.70 ± 1.2 MPa) when both sandblasting and metal primer was done when compared with no surface treatment (2.59 ± 0.32 MPa). **Conclusions:** It could be concluded that the use of metal primers along with sandblasting significantly improves the bonding of heat cured acrylic denture base resin with the Co-Cr alloy.

Keywords: Cobalt-chromium alloy, metal primer, shear bond strength

Introduction

Removable partial denture (RPD) is widely used as a treatment modality in partially edentulous patient. With the increase in the life expectancy, patients are wearing RPD for a longer period, thus requiring improvement in the overall quality of prosthesis to serve better.

Cobalt-chromium (Co-Cr) alloy is the most widely used dental alloy for fabrication of metal framework of RPD because of its rigidity and ease of fabrication.^[1] Denture base resins are used with these alloys for the fabrication of RPD where it serves to attach artificial teeth with the metal framework.

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The most commonly used heat cured acrylic denture base resin is polymethylmethacrylate.^[2]

Conventionally, denture base resins were attached to the metal framework by mechanical retention in the form of loops, mesh, beads, nail heads, undercut finish lines, and struts.^[3,4] However, other methods such as electrolytic etching,^[5] chemical etching^[6] and silica coating,^[7] Sandblasting, and metal primers can be used to improve bonding between metal and resin.

Currently, a variety of metal primers containing different functional groups like 11-methacryloyloxyundecan-1,1-decarboxylic acid (MAC10), 10-methacryloyloxydecyl dihydrogen phosphate (MDP), 6-4-vinylbenzyl-n-propyl amino-1, 3, 5-triazine 2, 4-dithione (VBATDT), and methacryloyloxyalkyl thiophosphate derivatives (MEPS) are available.

Various studies have been undertaken to investigate the effect of different metal primers on the bond strength of resins with various alloys, but it has been observed that bond strength depends on the type of resin used, the composition of the metal alloy and the metal primer used.

But little research is available to evaluate the use of metal primer that having the both functional groups (VBATDT and MDP [Kuraray Medical Inc., Japan]) on bonding between metal alloy and heat cured acrylic denture base resin. Hence, this study was designed to evaluate the shear bond strength (SBS) using this metal primer.

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Aims and objectives of the study

- To evaluate and compare the effect of no surface treatment, on the SBS of heat cured acrylic denture base resin to Co-Cr alloy
- To evaluate and compare the effect of sandblasting, on the SBS of heat cured acrylic denture base resin to Co-Cr alloy
- To evaluate and compare the effect of use of Alloy Primer (metal primer), on the SBS of heat cured acrylic denture base resin to Co-Cr alloy
- To evaluate and compare the effect of both sandblasting and use of Alloy Primer (metal primer), on the SBS of heat cured acrylic denture base resin to Co-Cr alloy
- To evaluate the bond failure between heat cured acrylic denture base resin and Co-Cr alloy using scanning electron microscope (SEM).

Materials and Methods

A total of 40 samples (calculated using statistical software version) were prepared and divided into 4 groups depending on the surface treatment received [Table 1].

Forty disk shaped wax patterns (Schuller Dental, ULM-W, Germany) were fabricated using special stainless steel mold of dimensions of 10 mm diameter and 2 mm thickness [Figure 1] which were then invested and casted in Co-Cr alloy (Wironit, Bego, Germany) by recommended technique. The castings thus obtained were finished following standard procedures. All the finished samples were put in ultrasonic cleaner.

After obtaining 40 Co-Cr alloy disks, modeling wax was added to the disk in a specific area of 5 mm diameter placed centrally using split stainless steel mold. The mold was fabricated in such a way that it accommodated the Co-Cr disk (10 mm diameter 2 mm thickness) and above the disk, the mold narrowed in the diameter up to 5 mm and extended 2 mm above the disk [Figure 2]. This created a space of 5 mm diameter and 2 mm thickness in the center of the disk where the modeling wax was applied.

These samples (Co-Cr alloy disk modeling wax assemblies) were flaked by following standard procedure for flaking in a conventional denture flask with dental plaster (KalaBhai Corp.). Totally, 10 Samples were flaked at a time. Then, the

samples were dewaxed and were cleaned using a steam cleaner (Aquaclean-3 Degussa).

Prior to the application of heat cured acrylic denture base resin, surfaces of Co-Cr alloy disks were given surface treatment according to the group.

For sandblasting, samples were subjected to airborne particle abrasion with aluminum oxide 110 µm (Hi alumina) in a sandblasting unit (Harnisch and Rieth) at 4-bar pressure. The distance between the nozzle tip and the specimen surface was maintained at 2 cm held perpendicular to the tip for 14 s.

For samples, to be treated with metal primer, a thin layer of metal primer (Alloy Primer Kuraray Medical Inc., Japan) was applied on the surface with the help of brush and then allowed to dry. For samples, to be treated with both sandblasting and metal primer, sandblasting was followed by application of metal primer.

After the surface treatment of samples, the heat cured acrylic denture base resin (Trevalon HI, Dentsply) was mixed as per manufacturer's instructions and packed over the bonding surface of the sample in the denture flask. The curing procedure was followed as per manufacturer's instruction in a curing unit. After the curing, flask was bench cooled, deflasked; bonded samples were retrieved carefully. The excess resin material was removed; the sample was finished. The finished samples were then stored in distilled water at 37°C for 24 h.

The samples were then embedded in the autopolymerizing acrylic resin (DPI) to prepare them for testing. The samples were embedded in such a way that only the Co-Cr alloy disks were embedded leaving the heat cured acrylic denture base resin exposed [Figure 3].

A custom made jig was fabricated with a semicircular knife edge to apply shear force at the interface of heat cured acrylic denture base resin and Co-Cr alloy disk sample [Figure 4]. Specimens were mounted in Universal Testing Machine with the help of the jig, and shear force was applied at the crosshead speed of 0.5 mm/min at the heat cured acrylic denture base resin and Co-Cr interface until fracture occurred. Values were observed at which fracture occurred. SBS was evaluated by the formula:

$$\text{SBS (MPa)} = \text{Load (N)/unit area (mm}^2\text{)}$$

Load: The value at which debonding of sample took place.

Area: Bonding area of the sample that was circular and 5 mm in diameter.

Area was calculated: πr^2 .

Table 1: Group distribution of samples

Group A (n=10)	No surface treatment was done on the bonding surface. This acted as a control group
Group B (n=10)	The bonding surface of alloy samples was sandblasted with 110 µm aluminum oxide particle
Group C (n=10)	The bonding surface of alloy samples was treated with metal primer (Alloy Primer, Kuraray Medical Inc., Japan)
Group D (n=10)	The bonding surface of alloy samples was sandblasted and then metal primer (Alloy Primer, Kuraray Medical Inc., Japan) was applied



Figure 1: Forty samples of wax pattern of dimension 10 mm diameter and 2 mm thickness

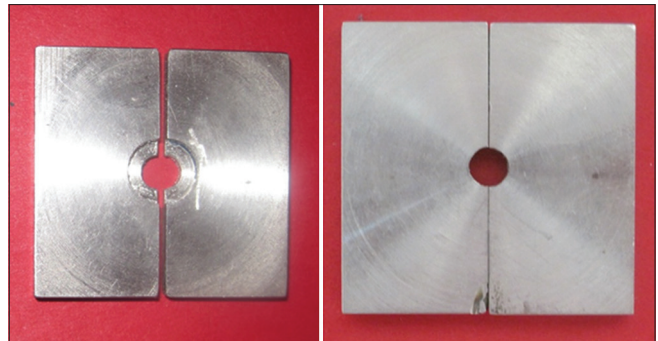


Figure 2: Split metal mold (5 mm diameter and 2 mm in thickness)



Figure 3: Forty samples mounted in autopolymerizing resin (clear acrylic)

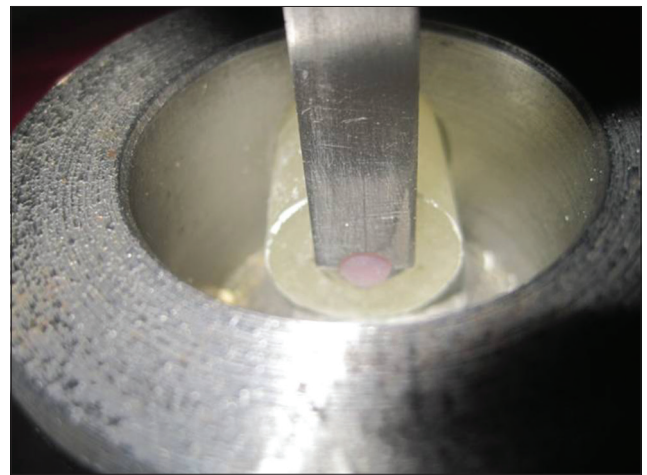


Figure 4: Samples mounted in Universal Testing Machine

Where r = radius of the disk.

After the SBS testing, SEM evaluation was done to observe the mode of failure. The type of failure was defined as adhesive, cohesive, and mixed. Samples were examined under SEM (SEM, model no 3400 Hitachi) at the magnification of $\times 500$ at the fracture site to verify the type of failure.

Results

All the observations obtained were analyzed statistically using software IBM SPSS version 17 (India); one-way analysis of variance and *post-hoc* Tukey's test were applied. $P < 0.05$ was considered as statistically significant in all the tests.

The result of the study [Table 2] showed that the SBS of the group treated with both sandblasting and metal primer (Group D) was highest among all with the SBS value of 18.70 ± 1.2 MPa and the SBS of control group (Group A) was lowest among all with SBS value of 2.59 ± 0.32 MPa.

Since there was a statistically significant difference ($P < 0.05$) between the bond strength of Group B (sandblasting only), Group C (primer only), and Group D (sandblasting and metal primer) as compared to the control Group A (no surface treatment) having bond strength of 2.59 ± 0.32 MPa, so it can be said that surface treatment of metal alloy definitely improves the bonding of the metal with resin.

Table 2: Mean SBS value of groups and failure mode evaluated by SEM

Group	SBS (mean \pm SD) (MPa)	Failure percentage		
		Adhesive	Cohesive	Mixed
A	2.59 \pm 0.32	100	00	00
B	9.65 \pm 0.58	100	00	00
C	14.49 \pm 0.96	10	00	90
D	18.70 \pm 1.12	00	00	100

SD: Standard deviation; SBS: Shear bond strength; SEM: Scanning electron microscope

The SBS of the group treated with sandblasting only (Group B) was less (9.65 MPa) than group treated with primer only (Group C) having SBS of 14.49 MPa; since there was a significant statistical difference ($P < 0.05$) between the groups, it can be said that the metal primer provides better bonding of metal with the resin as compared to the sandblasting.

The SBS of group treated with metal primer only (Group C) had statistically significant ($P < 0.05$) lower SBS (14.49 MPa) than the group treated with both sandblasting and the metal primer (Group D) having SBS of 18.70 MPa; it can be

said that the surface treatment with both sandblasting and metal primer provides better bonding of resin with metal as compared to the use of metal primer alone.

SEM evaluation of the type of failure [Table 2] shows that the Group A (no surface treatment) and Group B (sandblasting) showed 100% adhesive failure indicating that the resin completely delaminated from the metal surface and there was no chemical bonding with the metal.

Group C (metal primer only) and Group D (metal primer and sandblasting) showed mixed type of failure indicating that some part of resin remained attached to the metal surface. This might be explained by the chemical bonding of the resin with the metal because of the use of adhesive primer. SEM images of the debonded samples of each group are shown in Figure 5a-d.

Discussion

In this study, the effect of sandblasting (110 μm alumina particles) and use of metal primer (Alloy Primer, Kuraray Medical Inc., Japan) on the bond strength of heat cured acrylic denture base resin (Trevalon, Dentsply) with Co-Cr alloy (Wironit, Bego, Germany) has been evaluated, so that the appropriate surface treatment can be suggested while using this combination of material in the fabrication of the prosthesis.

To improve the bond strength, various methods have been tried in the reviewed literature like electrolytic etching,^[5] chemical etching^[6] sandblasting, etc. Every method used had its own limitations for example electrolytic etching requires cumbersome equipment whereas, sandblasting has shown to

improve the bond strength of the resins with the alloys by increasing the surface area thus aiding in the micromechanical retention; however, there is no chemical bonding between acrylic resin and metal so, the problem of microleakage remains.^[8] Chemical surface treatment with adhesive primer of metal alloys has shown to improve the bonding properties but depend on the type of alloy used, the denture base resin used and the adhesive primer composition. This chemical method is easy to use as it requires no complex equipment. But, the reliability of these metal primers or adhesive primers needs to be thoroughly investigated.

Although few studies are available with the metal primers, since it is dependent on variable factors, the study to evaluate the efficacy of one such metal primer with particular metal alloy-denture base resin combination is useful.

Many studies have been done to investigate the effect of sandblasting on the SBS of resin with the metal alloys and it has shown to have a positive effect. Ishii *et al.*^[9] reported that the alumina air abrasion enhances the bond strength of the resin with the metal and the roughness produced depend on the composition of the alloy. May *et al.*^[10] in 1997 reported that there is remarkable improvement in the SBS in the resin-alloy after sandblasting. Similar findings were reported by many authors.^[11] Since sandblasting has already proven to increase the bond strength of the resin with the metal alloy as stated in the previous studies, the values of this group were used to compare the SBS values obtained from the Group C and Group D in which the metal primer was used so as to come at a conclusive result regarding the bonding properties of adhesive primers as compared to time-tested sandblasting procedure.

The metal primer used in this study was Alloy Primer (Kuraray Medical Japan Co.) which is a VBATDT- and MDP-based metal primer. Metal primers with different composition are available like VBATDT-based, MDP-based,^[12] 4-META-based, BIS GMA-based, MAC-10, MEPS, etc. Antoniadou *et al.*^[13] stated that the composition of Alloy Primer affects the bond strength value. VBATDT, a thione-thiol tautomer was synthesized by Kojima *et al.*^[14] during late 1980s, to be used as a coupling agent between methacrylate-based monomers and noble metal alloys. This VBATDT alone was ineffective in increasing the bond strength of the Co-Cr alloy to the resin. MDP alone showed improvement in the bond strength of the resin with base metal alloys in a study conducted by Kim *et al.*^[8]

Ali^[11] conducted a study to evaluate the effect of primers on the SBS of two types of acrylic resin to Co-Cr partial denture alloy. Fifty Co-Cr ingots and fifty cast specimens were fabricated and embedded in resin. The bond strength between primed specimens improved significantly compared with the control group. It was shown that primers enhanced the bond strength of acrylic resin and cast Co-Cr alloy. Kawaguchi *et al.*^[15] conducted a study to evaluate the

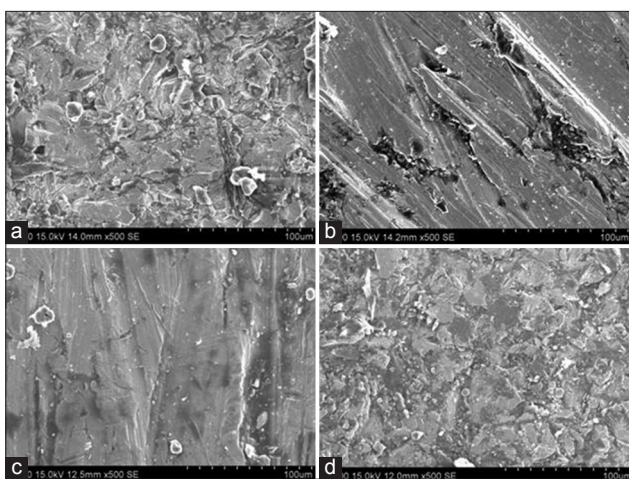


Figure 5: Scanning electron microscope image of debonded sample: (a) Group A (no surface treatment) showing adhesive failure. (b) Group B (sandblasting only) showing adhesive failure. (c) Group C (metal primer only) showing mixed failure. (d) Group D (metal primer and sandblasting) showing mixed failure

effect of surface preparation on the bond strength of heat-polymerized denture base resin to commercially pure titanium and Co-Cr alloy. The alloy specimens were divided into five groups: (1) Airborne-particle abraded with 50 μm alumina (SAND), (2) Rocatec tribochemical silica coating system (RO), (3) Air-abraded followed by application of Epricord Opaque Primer (EP), (4) Air-abraded followed by application of Super Bond C and B liquid (SB), and (5) Air-abraded followed by application of alloy primer (AL).

In this study, the primer used was Alloy Primer, which had principle ingredients: Acetone, MDP and VBATDT both, a thione-thiol tautomer. The coupling mechanism of this primer is by: (i) MDP has a phosphate ester group that presents great chemical bonding with the surface layer of oxide of chrome formed at the surface of Co-Cr alloy subsequently primary bond formation^[12,16] and (ii) copolymerization of vinyl groups with the methacrylate-based resin monomer.

Sandblasting along with primer application significantly improved the bond strength as compared to no surface treatment or sandblasting alone or metal primer alone. Also, the application of metal primer alone resulted in higher bond strength than sandblasting alone.

The bond strength values obtained in this study were comparable to the bond strength values obtained in the similar studies reviewed in the literature. Kim *et al.*^[8] evaluated the effect of two different metal primers (Alloy Primer, Kuraray and MR Bond, Tokuyama, Japan) on the SBS of Co-Cr alloy (Biosil F, Degussa). The bond strength values obtained in their study in primed group (primer and sandblasting) was 17.1 MPa, which was comparable to the value obtained in this study (Group D; 18.70 MPa).

In all the studies, it was seen that the bonding of resin improved with the application of primer irrespective of the bond strength values. The bonding with use of primer was better than the sandblasted surface in all studies. The results obtained in the present study also indicated similar findings that is the bonding was significantly improved with use of primer on both sandblasted group (Group D) and also in nonsandblasted group (Group C). So, this can be concluded from this study that metal primer along with sandblasting can be used to provide better bonding between Co-Cr alloy (Wironit) and the heat cured acrylic denture base resin (Trevalon).

The bond failures obtained in this study were observed under SEM under $\times 500$ magnification to classify the type of failure. Group A and Group B showed 100% adhesive failure whereas Group C and Group D showed mixed failure. The probable reason for adhesive failure in Group A and Group B is the absence of any chemical bond between the resin and alloy whereas in Group C and Group D where metal primer was used, mixed failures occurred because of chemical bonding

between the resin and the metal surface. This finding can be correlated with many studies that presented similar findings.^[8,17,18]

Conclusion

It could be concluded that the use of metal primers along with sandblasting significantly improves the bonding of heat cured denture base resin with the Co-Cr alloy but the value of SBS and types of failure may be different depending upon the chemical composition of metal primer.

Limitations of the study

- The dimensions of the test sample used did not represent the actual clinical condition so the difference in the geometry may affect the stress distribution and hence the SBS
- In this study, samples were immersed in distilled water. However, the oral cavity is bathed with saliva, which is not chemically similar to distilled water. Also, the effect of temperature fluctuations in the oral cavity has not been simulated in the study.

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