Research & Reviews

ISSN: 2320-7949

Research and Reviews: Journal of Dental Sciences

# A Comparative Study of the Effectiveness of Metal Surface Treatment in Controlling Microleakage of Two Different Metal and Acrylic Resin Interface.

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## Research Article

Received: 26/03/2013
Revised: 05/04/2013
Accepted: 17/04/2013

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**Keywords:** Nickel-Chromium, Cobalt-Chromium; Heat Cure Acrylic Resin; Surface treatment, Silane coupling agent and Tin plating.

**ABSTRACT**

Microleakage at the junction between the metal alloy and acrylic resin in a removable partial denture may result in discoloration, fluid percolation, and acrylic resin deterioration and Failure of a removable partial denture. Dye penetration test was conducted on eighty specimens each of Nickel-Chromium/Cobalt-Chromium metal-Heat cure acrylic resin. Samples were randomly divided into eight groups (n=10) & adapted with base plate wax and they were invested. Groups were Sandblasted, Tin plated and silane coated individually and in their various combinations and were then packed with clear heat cure acrylic resin and processed over the surface treated side of the metal. All specimens were immersed in distilled water for 24 hours followed by thermocycling for 3000 cycles before immersion in dye for 24hrs. Counting of the grids which exhibited dye penetration allowed the assessment of extent of microleakage. The mean values of the grids that exhibited dye penetration in this group for Nickel-Chromium and Cobalt-Chromium alloys are 3.3000±2.584 and 3.6000±2.6331. microleakage values for both the metals were different for different combinations.

## INTRODUCTION

Removable partial denture is subjected to variation in temperature in the oral cavity which can lead to microleakage and cause weakening of the interface between the resin and metal, finally lead to deterioration of the contact between the metal and acrylic resin [1]. A stable contact should exist between the resin-metal to prevent microleakage between the interfaces. Microleakage is caused because of absence chemical bonding and difference in coefficient of thermal expansion between metal and resin [2].

Earlier frame work designs needed certain elements to provide mechanical retention between the resin and the metal. Despite the incorporation of various mechanical retentive elements, functional forces often result in the failure of the acrylic resin at its junction with framework [3]. The absence of chemical bond directly affects the metal-resin interface as a space exists between the metal framework and the resin denture base microscopically.

Various bonding methods and techniques for base metal alloys have been developed to enhance the bond between metal-resin interfaces, such as Electrolytic etching, Sand blasting, Electroplating and Chemical bonding of resin to dental alloys E.g. Silane coupling agent [4]. Therefore this study was under taken to evaluate and compare the effectiveness of Sandblasting, Tin coating, Silination and various combinations of these processes in controlling microleakage between polymethyl methacrylate resin and Nickel-Chromium and Cobalt-Chromium alloy after thermocycling.

### Objectives of the Study

- To evaluate and compare the effect of only Sandblasting, Tin plating and Siliane primer individually on controlling microleakage between heat-cure acrylic resin to cast Nickel-Chromium and Cobalt-Chromium alloy after water immersion and thermocycling.
- To evaluate and compare the effect of combining Sandblasting and Tin coating, Sandblasting and Siliane primer, Tin coating and Siliane primer on controlling microleakage between heat-cure acrylic resin to cast Nickel-Chromium and Cobalt-Chromium alloy after water immersion and thermocycling.

- To evaluate and compare the effect of combining Sandblasting, Tin plating and Siliane primer on controlling microleakage between heat-cure acrylic resin to cast Nickel-Chromium and Cobalt-Chromium alloy after water immersion and thermocycling.

## METHODOLOGY

Present study was conducted in the department of Prosthodontics, SDM Dental College & Hospital Dharwar Karnataka. Dye penetration test was planned on each samples of nickel-chromium-heat cure acrylic resin and cobalt-chromium-heat cured acrylic resin which are subject to different surface treatment.

### Specimen Fabrication

Rectangular specimens of dimension 15x6x1mm with grid pattern were cast of Nickel-Chromium and Cobalt-Chromium alloy. Eighty eight wax patterns each for Nickel-Chromium and Cobalt-Chromium alloy were prepared using inlay casting wax (Schuller-Dental U LM- W. Germany). Fabric mesh was then attached on to these patterns to get the grid pattern on the cast specimens (Colour plate-1). Ten rectangular wax patterns were attached at a time to the runner bar. It was then fixed to a 3x size crucible former (Gusskuvetten, Deugu Dent, Germany), and the wax patterns were sprayed with a surfactant liquid to reduce the surface tension (Aurofilm, Bego, Germany) and blow dried.

The 3x casting ring (Gusskuvetten, Deugu Dent, Germany) was lined with an asbestos free liner (Kera Vlies, Dentaurm,Germany) such that it was 4mm short at either ends of the ring (Colour plate-3). The casting ring with the moistened liner was attached to a Crucible former and the wax patterns were vacuum invested (Multivac 4-Degussa, Germany) using 120gms of phosphate bonded Investment material (Bellasun, Bego, Germany) and 35ml mixing liquid (Begosol, Bego, Germany) as per manufacturer's instructions. After setting of the investment, the crucible former was separated and the ring was placed inside the Wax burnout furnace (Type 5640- KAVO EWL, Germany).

The invested casting ring was heated in this furnace according to the following schedule - (1) 55° C/ min increase in temperature until it reached 150° C, with a 90min dwell (holding time); (2) 5°C/min until it reached 250°C, with a 90min dwell (holding time): (3)5°C/min until it reached 950°C, with a 60- min dwell time. After reaching the maximum temperature of 950°C the specimens were casted immediately in a centrifugal casting machine (Colour plate-6) (Degutron, Degussa, Germany) according to manufacturer's instructions. After casting, the rings were allowed to cool down to room temperature and were devested from the investment. The casting were sandblasted with aluminum oxide powder to remove the investment material and the specimens with the individual sprue at the attachment to the runner bar, were separated using carborundum discs (Dentorium, New York, U.S.A). The samples were then trimmed and polished.

Modelling wax of dimension 15x6x1mm, (Hindustan Dental Products, India) was adapted to all the specimens. The specimens with the wax patterns were invested in conventional denture flasks (Nikhil, India) with dental plaster (kalabhai, India). Eight specimens were invested in each flask (Colour plate-10). Likewise all the samples were invested and dewaxing was done. The samples were then numbered and carefully retrieved from the flask.

### Surface treatment of the specimens

Before packing the samples with heat cured clear acrylic, the samples of nickel-chromium and cobalt-chromium alloy were randomly divided into eight groups and labeled based on the type of surface treatment they received. Each group had ten samples. Different combinations of the following surface treatments were carried out on each group.

### Sandblasting procedure

Samples were abraded with aluminum oxide (ll0μm) using a Sandblasting unit (Colour plate-11) (type 5417- KAVO EWL Germany), at 4-bar pressure. During sandblasting, the distance between the nozzle tip and the specimen surfaces was maintained at 2cm and perpendicular to the tip, Sandblasting was carried out for 15 seconds. These samples were then ultrasonically cleaned in Soniclean ultrasonic cleaner (80 T, Transtek systems, Australia) and air dried to ensure the removal of all residual particles and contaminants.

### Silane coupling agent

On the clean surface of the specimens a thin layer of silane coupling agent (Calibra, Dentsply) was applied to the surface of each of the titanium specimens with the brush following the manufacturer's instructions and allowed to air dry.

**Tin electroplating**

The lead from the tin-plating unit was attached to the samples. A cotton pellet saturated with tin-plating solution was moved over the sample in a brushing motion to get a uniform thickness of tin on the sample. The samples were then rinsed in distilled water and dried under compressed air.

**Group I**: Sandblasting
**Group II**: Tin coating
**Group III**: Silane coupling agent
**Group IV**: Tin coating and Sandblasting
**Group V**: Sandblasting and Silane coupling agent
**Group VI**: Tin coating and Silane coupling agent
**Group VII**: Tin coating, sandblasting and silane coupling agent
**Group VIII**: No surface treatment was carried-control

**Heat Cure Acrylic Resin application**

After the samples were surface treated according to above mentioned procedure, they were returned to the original place in the flask with care to avoid contamination of the treated surface. Heat cure acrylic resin (Trevalon, Dentsply, India) was mixed, packed and processed according to manufacturer's instructions. On recovery of the specimens, the edges were refined to expose the acrylic-metal interface and cleaned in ultrasonic cleaner (Soniclean 80T, Transtek). The samples were then stored in separate labeled box in distilled water for 48hrs before subjecting the samples for thermocycling.

**Thermocycling**

The specimens were wrapped in a sterile gauge piece and placed in a beaker with ice cubes which was maintained at a temperature of $5^0C \pm 3^0C$ for one minute and immediately the specimens were placed in water of $37^0C$ for one minute, followed by water maintained at $60^0C$ for one minute. This procedure was repeated for 3000 cycles.

**Dye penetration test**

The thermocycled specimens were then immersed in fluorescene dye (100mg/1000mL) for 24hrs (Colour plate-16). The samples were then cleaned in an ultrasonic cleaner to remove any surface stain. The dye penetration from the metal-acrylic interface was assessed under magnification (8x) using a Speckfinder (Speckz18-250, Dazor, U.S.A) (Colour plate-17). The penetrated dye could be easily visualized through the clear acrylic resin. The grids that showed evidence of dye penetration was recorded as positive (Colour plate-18, 19). The total number of grids containing the dye was recorded as the basic data (Annexure 1&2)

The number of grids showing the presence of dye penetration in specimen from each group of surface treatment was subjected to statistical analysis by ANOVA to compare the means and to test for interaction between each group; the values were subjected to Newman-Keuls post hoc multiple comparative test and student-t test.

## RESULTS

The Table-I shows the mean and standard deviation of microleakage in Nickel- Chromium alloy for different groups. Group V showed the least microleakage at 3.3000±2.5841. On comparison of groups by ANOVA, eight groups showed a significant difference in the level microleakage between Nickel-Chromium and heat cure resin interface (18.4349) at 1% level of significance (Table II).

**Table I: Mean and Standard deviation values for microleakage in different groups of heat cure acrylic resin-nickel-chromium interface after different surface treatment. (n=10)**

| GROUPS | MEANS | S.D | C.V | MINIMUM | MAXIMUM |
|---|---|---|---|---|---|
| I | 6.3000 | 2.4060 | 38.1907 | 2 | 10 |
| II | 8.9000 | 4.2282 | 47.5080 | 2 | 16 |
| III | 6.4000 | 3.0623 | 47.8487 | 2 | 13 |
| IV | 10.8000 | 3.0840 | 28.5556 | 7 | 16 |
| V | 3.3000 | 2.5841 | 78.3073 | 0 | 8 |
| VI | 10.9000 | 2.7264 | 25.0130 | 7 | 15 |
| VII | 12.6000 | 3.0258 | 24.0144 | 7 | 17 |
| VIII | 17.3000 | 4.0565 | 23.4482 | 10 | 24 |

**Table II: Comparison of groups by ANOVA for microleakage in heat cure acrylic resin- Nickel-chromium interface after different surface treatment.**

| Source of variation | DF | SS | MSS | F-value | P-value | Signi. |
|---|---|---|---|---|---|---|
| Between Groups | 7 | 1327.1875 | 189.5982 | 18.4349 | 0.0000* | HS |
| Within Groups | 72 | 740.5000 | 10.2847 | | | |
| Total | 79 | 2067.6875 | | | | |

*Significant at 1% level of significance ($p<0.01$)

The Table-III show the mean and standard deviation of microleakage in Cobalt- Chromium alloy for different groups. Group V showed least microleakage as 3.6000±2.6331. On comparison of groups by ANOVA, eight groups showed a significant difference in the level microleakage between Nickel-Chromium and heat cure resin interface (19.5474) at 1% level of significance (Table IV).

**Table III: Newman-Keuls post hoc multiple comparative test for microleakage in different groups of Heat cure acrylic resin-Nickel-Chromium interface after different surface treatment.**

| Group | I | II | III | IV | V | VI | VII | VII |
|---|---|---|---|---|---|---|---|---|
| Means | 6.3000 | 8.9000 | 6.4000 | 10.8000 | 3.3000 | 10.9000 | 12.6000 | 17.3000 |
| I | | | | | | | | |
| II | 0.1727 | | | | | | | |
| III | 0.9447 | 0.0857 | | | | | | |
| IV | 0.0130** | 0.1895 | 0.0285 | | | | | |
| V | 0.0401** | 0.0013* | 0.0185** | 0.0001* | | | | |
| VI | 0.0167** | 0.3492 | 0.0130** | 0.9447 | 0.0001* | | | |
| VII | 0.0006* | 0.0566 | 0.0006* | 0.4252 | 0.0001* | 0.2399 | | |
| VIII | 0.0001* | 0.0001* | 0.0001* | 0.0003* | 0.0001* | 0.0002* | 0.0017* | |

*Significant at 1% level of significance ($p<0.01$)
**Significant at 5% level of significance ($p<0.05$)

To know the significant difference for different surface treatment, analyses were done by the Newman-Keuls Post Hoc Multiple Comparative tests individually for both Nickel- Chromium alloy and Cobalt- Chromium alloy. Test results showed that the microleakage values of Group V differed significantly from that of Group I, Group II, Group III, Group IV, Group VI, Group VII and Group VIII with their microleakage values at 5% level of significance for both Nickel- Chromium alloy and Cobalt- Chromium alloy (Table V & VI). This analysis indicates superiority of the surface treatment given to group V specimens who were treated by sandblasting and application of silane coupling agent for both Nickel- Chromium alloy and Cobalt- Chromium alloy.

Statistical Pair wise comparison by Student Paired "t" test of difference in mean of microleakage values of the eight groups between Heat cure acrylic resin - Nickel-Chromium and Heat cure acrylic resin- Cobalt-Chromium. The results from the above test showed, no significant difference among the different surface treatment methods employed in controlling microleakage between both the alloys (Table VII).

**Table IV: Mean and Standard deviation heat cure acrylic resin- Cobalt-Chromium interface after different surface treatment.**

| GROUPS | MEANS | S.D | C.V | MINIMUM | MAXIMUM |
|---|---|---|---|---|---|
| I | 7.1000 | 2.6013 | 36.6378 | 3 | 11 |
| II | 9.7000 | 3.9172 | 40.3835 | 3 | 15 |
| III | 7.7000 | 2.8694 | 37.2647 | 4 | 14 |
| IV | 11.3000 | 2.3118 | 20.4585 | 8 | 15 |
| V | 3.6000 | 2.6331 | 73.1423 | 0 | 7 |
| VI | 11.9000 | 2.8848 | 24.2422 | 8 | 17 |
| VII | 13.4000 | 2.9136 | 21.7431 | 8 | 17 |
| VIII | 17.7000 | 4.0838 | 23.0726 | 10 | 23 |

**Table V: Comparison of groups by ANOVA for microleakage in heat cure acrylic resin-Cobalt-chromium interface after different surface treatment.**

| Source of variation | DF | SS | MSS | F-value | P-value | Signi. |
|---|---|---|---|---|---|---|
| Between groups | 7 | 1301.8000 | 185.9714 | 19.5474 | 0.0000* | HS |
| Within groups | 72 | 685.0000 | 9.5139 | | | |
| Total | 79 | 1986.8000 | | | | |

*Significant at 1% level of significance ($p<0.01$)

**Table VI: Newman-Keuls post hoc multiple comparative test for microleakage in different groups of Heat cure acrylic resin- Cobalt-Chromium interface after different surface treatment.**

| Group | I | II | III | IV | V | VI | VII | VIII |
|---|---|---|---|---|---|---|---|---|
| Means | 7.1000 | 9.7000 | 7.7000 | 11.3000 | 3.6000 | 11.9000 | 13.4000 | 17.7000 |
| I | | | | | | | | |
| II | 0.1505 | | | | | | | |
| III | 0.6650 | 0.1515 | | | | | | |
| IV | 0.0169** | 0.2500 | 0.0294** | | | | | |
| V | 0.0134** | 0.0003* | 0.0112** | 0.0001 | | | | |
| VI | 0.0075* | 0.2545 | 0.0169** | 0.6650 | 0.0001 | | | |
| VII | 0.0004 | 0.0440* | 0.0010* | 0.2866 | 0.0001* | 0.2806 | | |
| VIII | 0.0001* | 0.0001* | 0.0001* | 0.0002* | 0.0001* | 0.0003* | 0.0028* | |

*Significant at 1% level of significance ($p<0.01$)

**Significant at 5% level of significance ($p<0.05$)

**Table VII: Statistical Pair wise comparison by Student Paired "t" test of difference in mean of microleakage values of the eight groups between Heat cure acrylic resin - Nickel-Chromium and Heat cure acrylic resin- Cobalt-Chromium.**

| | Cobalt-Chromium | | Nickel-Chromium | | | | |
|---|---|---|---|---|---|---|---|
| Group | Mean | Std.Dev. | Mean | Std.Dev. | t-value | p-value | Signi. |
| I | 7.1000 | 2.6013 | 6.3000 | 2.4060 | 0.7140 | 0.4844 | NS |
| II | 9.7000 | 3.9172 | 8.9000 | 4.2282 | 0.4389 | 0.6659 | NS |
| III | 7.7000 | 2.8694 | 6.4000 | 3.0623 | 0.9796 | 0.3403 | NS |
| IV | 11.3000 | 2.3118 | 10.8000 | 3.0840 | 0.4102 | 0.6865 | NS |
| V | 3.6000 | 2.6331 | 3.3000 | 2.5841 | 0.2571 | 0.8000 | NS |
| VI | 11.9000 | 2.8848 | 10.9000 | 2.7264 | 0.7967 | 0.4360 | NS |
| VII | 13.4000 | 2.9136 | 12.6000 | 3.0258 | 0.6023 | 0.5545 | NS |
| VIII | 17.7000 | 4.0838 | 17.3000 | 4.0565 | 0.2197 | 0.8285 | NS |

## DISCUSSION

Denture base resins are commonly used with base metals alloys for the fabrication of removable partial dentures. The bonding is mostly achieved by mechanical retentive methods or by surface treatment of the alloys. However there is absence of chemical bonding of resin which affects the metal-resin interface. Microscopically a space is present, between the metal framework and resin part of denture.

The purpose of sandblasting the bonding surface was to increase the surface area for bonding by micromechanical retention and to decreasing the surface tension[5]. Silane coupling agent was used to enhance the bond between metal and acrylic. They possess both organic and inorganic properties; hence these chemicals react with polymer and minerals components forming covalent bonds across the interface, improving adhesion and durability.

Tin plating was initially introduced to improve the bond strength between Nobel and high Nobel metal alloys to resin cements. The effectiveness of tin plating on the bond strength of resins cements to metal shows a lot of variation. There are conflicting results as to whether; the positive effect of tin plating is dependent or independent of tin plating.

It was observed for both the metals that the microleakage values are in increasing magnitude as Group V< Group I < Group III< Group II< Group IV< Group VI< Group VII< Group VIII. Group VIII which did not receive any surface treatment had the maximum amount of microleakage. This can be attributed to lack of micromechanical retention, surface contamination and reduced wettability of the alloy surface [6,7].

The decreased level of microleakage in Group V for both the alloys can be attributed to the combined effect of sand blasting and use of the silane coupling agent, which resulted better bonding there by reducing microleakage. Mukai et al [8] reported that silane when used in combination with sand blasting improves wettability and enhances the chemical bonding between metal and acrylic.

The results from the study indicate that Tin plating did not cause any improvement in the samples when used alone or when used in different combination in fact it deteriorated the bonding of heat cure acrylic resin with the metal even though the surface were treated by sandblasting and or silane coupling agent (Group II, VI, VII) . The results were not consistent or as effective as Group V. The effectiveness of tin plating in improving bond strength for noble metal has been reported in many studies [9,10]. A different result has been reported in a study by Imberry et al [11], it was reported as to whether positive effect of Tin plating of Noble metal alloys on bond strength was dependent on tin plating or not. Other studies have shown lower bond strength values of Tin plated alloys when used with Nickel-Chromium-Beryllium alloys [12].

Students Paired T-test was performed to compare the mean values of the measured microleakage of the eight groups between Heat cure acrylic resin - Nickel-Chromium and Heat cure acrylic resin- Cobalt-Chromium interface. The p-value showed no significance at 5% level of significance ($p<0.05$). The results from the test showed there is no significant difference among the different surface treatment methods employed in controlling microleakage between both the alloys.

## CONCLUSION

Based on the findings of the study, the following pre-treatment is recommended in order to reduce the amount of microleakage between Heat cure acrylic and base metal alloys.

- Finishing of the metal surface using burs.
- Sandblasting by controlled application of 110μm alumina air abrasive to metal surface and then cleaning it in ultrasonic bath for 10min.
- Application of silane coupling agent uniformly on sandblasted surface of metal before packing.

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