

## 실란과 알칼리 처리가 두 코발트-크롬 합금에 대한 아크릴 레진의 전단결합강도에 미치는 영향

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## Effect of silane and alkali treatment on the shear bond strength of acrylic resin to two Co-Cr alloys

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가철성 국소의치에서 주조 합금의 골격과 의치상용 레진 사이에 결합을 향상시키기 위하여, 본 연구에서는 코발트-크롬 합금과 아크릴 레진 사이의 결합에서 실란 및 알칼리 처리가 화학적 결합을 향상시켜 최종적으로 금속레진 전단결합강도를 향상시키는지를 조사하였다. 전단 결합강도용 시편은 직경 3.5 mm 및 높이 10 mm의 최종 크기로 준비하였고, 접촉각 및 표면 거칠기 측정용 시편은 폭 10 mm, 길이 10 mm 및 두께 1 mm로 만들었다. 3-MPTMS 실란을 사용하여 실란 용액을 만들었다. 시편을 실란 및 알칼리 처리하여 미처리(UT)군, 실란 처리(ST)군, 알칼리 처리(AT)군, 알칼리 및 실란 처리(AST)군과 같이 4개의 군으로 하였다. 또한 표면에 대하여 접촉각과 표면 거칠기를 측정하였다. 합금 시편에 혼합된 아크릴 레진을 결합시킨 다음 레진을 중합시켜 결합강도 시편을 만들었고, 전단결합강도를 측정한 후 파절편을 주사 전자현미경으로 관찰하였다. 모든 군에 대한 결과는 이원 분산분석을 이용하여 분석한 다음 0.05의 유의 수준에서 Tukey 다중 비교 테스트를 수행하였다. 통계 분석 결과 모든 군에 대하여 Ra 값에 유의한 차이는 없었고(p>0.05) 접촉각에 있어서도 큰 차이가 없었다. 두 합금 모두에서 ST군과 AST군이 가장 높은 전단 결합강도를 보였고(p<0.05), UT군과 AT군은 두 합금 모두에서 낮은 전단 결합강도를 나타내었다. UT군과 AT군의 경우 파절된 합금의 표면에서 완전한 접착성 파괴를 나타내었다. 본 연구에서 Co-Cr 합금에 대한 실란 처리는 전단 결합강도를 향상시키는데 효과적이었으나, 알칼리 처리는 뚜렷한 효과를 보이지 않았다.

**색인단어 :** 알칼리, 아크릴 레진, 코발트-크롬 합금, 전단 결합강도, 실란

## Introduction

A strong and stable bond between denture base resin and removable partial denture (RPD) metal framework requires for the esthetic appearance and clinical longevity of a restoration, which should endure a combination between mechanical, chemical and thermal stresses (1, 2). Therefore, it would be advantageous to find an effective method of achieving a strong and stable bond between the denture base resin and metal framework.

Conventional methods of attaching denture base resin to metal framework of a RPD include macromechanical retention. Previous studies (3-5) have reported the chemical etching methods to create an understructure of micromechanical retention to improve resin bond strength to alloy. Other studies (6-8) have described the sand-blasting treatment of the alloy surface to enhance the bond strength between resin and alloy. However, their surface treatment method has depended on mechanical bond of resin and alloy surface, which was more affected by thermal changes than chemical bond systems (8, 9). *In vitro* studies have also indicated that mechanical bond systems could form a relative weak metal-resin bond. Although these are currently available for chemical bond

between resin and metal frameworks, they have lower bond strength when compared with porcelain (10).

The use of silane coupling agents in enhancing the bond of organic matter to inorganic matter is well accepted in the dental literature (11). In the bonding system of resin-porcelain using a silane coupling agent, reliable bonds were achieved (12). To change the filler surface and bond the filler to the resin matrix of the composite, silanes are also applied as surface treatment agents (13). In these silanes, organic functional groups can co-polymerize with the resin matrix. The silanes also have three alkoxy ready to hydrolyze and then to react with surface hydroxyl groups on an inorganic substrate such as metals/alloys.

Chemically suitable hydroxyl groups are found on the surfaces of titanium, chromium and molybdenum, which form a thin, passive oxide layer. They all contain hydroxyl groups on their outermost surface under standard temperature and alkali treatment (14). Hydroxyl groups on the surface alloy surface play an important role in alloy-resin bond. This is the reason why active hydroxyl groups are the initiation sites for chemical reactions during silane bonds with alloy. Other studies on hydroxyl groups (15) also showed the effects of surface oxidation on the

resin bonding to titanium. Therefore, it can be concluded that hydroxyl groups are suitable for the bonding between metal and resins. Extending these concepts, our work was undertaken to provide chemical bonding between examine shear bond strength between cobalt-chromium (Co-Cr) RPD alloys and PMMA resin respectively, using silane and alkali treatments.

## Materials and Methods

### 1. Preparation of cast RPD alloys

Cylinder-shaped wax patterns (3.5 mm in diameter and 45 mm in height) for shear bond strength test and wax patterns (10 mm in width, 15 mm in length and 1 mm thickness) for water contact angle and surface roughness test were formed in a silicone molds. All wax patterns were invested under vacuum with phosphate-bonded investment (BC 700, Bukwang Inc., Busan, Korea). A Co-Cr alloys designed for RPD casting was used. The chemical compositions of the alloys are presented in Table 1. After burnout at the temperatures specified by each manufacturer, the specimens were centrifugally cast by melting using a gas-oxygen multi-orifice torch. All molds were bench-cooled after casting. The remaining parts of the investment were removed by sandblasting with 110  $\mu\text{m}$   $\text{Al}_2\text{O}_3$ . The alloy strips were cut with a disc to the final dimensions of 3.5 mm in diameter and 10 mm in

height for shear bond test and  $10 \times 10 \times 1$  mm for water contact angle and surface roughness measurements. These were visually inspected for surface defects and defective samples were replaced. All specimens were wet-ground successively with 150 and 2,000 grit silicon carbide paper, rinsed with distilled water, ultrasonically cleaned in acetone for 10 min and finally dried in a stream of air at room temperature.

### 2. Surface treatment

3-methacryloxypropyltrimethoxysilane (MPS, Sigma-Aldrich Chemie GmbH, Steinheim, Germany, Lot No. 107k0006) that contains three inorganic reactive groups on silicon was used. A silane solution was prepared by the addition of 2 ml MPS in solvent solution containing 10 ml distilled water and 90 ml methanol. The pH was adjusted with 1 M acetic acid to 3.6. It was allowed to be stabilize for 24 h at room temperature. Ten specimens were dispersed in 25 ml of silane solution. The specimens were stirred using a magnetic bar at 600 rpm at room temperature for 5 h. For removing additional silane solution from the surface, the specimens were washed three times in distilled water and then dried at 100°C for 2 h. The samples were treated in 20 ml of 10 M sodium hydroxide (NaOH) at a temperature of 60°C for 24 h followed by gentle washing using distilled water and drying for 3 h at 60°C. Some samples were subjected to the silane treatment after the alkaline pre-treatment.

**Table 1.** Composition of the two Co-Cr alloys used

Brand name	Manufacturer	Lot No.	Composition (wt%)*
Biosil-F	Degussa Dental GmbH & Co. KG, Hanau, Germany	Not available	Co (64.8), Cr (28.5), Mo (5.3), Mn (0.5), Si (0.5), C (0.4)
Vera PDS-Hard	Albadent Co., Corderlia, ND, USA	0604062	Co (63.5), Cr (27.0), Ni (1.0), Mo (5.5), Fe (2.0), others (1.0)

\*According to the information provided by the manufacturers.

The four groups were named as: untreated (UT), silane treated (ST), alkali treated (AT), and alkali treated followed by silane treatment (AST).

### 3. Surface characterization

All specimen surfaces were evaluated for wettability by measuring contact angles using distilled water. Computerized contact angle system (OCA 15 Plus, Dataphysics Instruments, Filderstadt, Germany) was used in combination with SCA 20 software (Dataphysics Instruments) for image analysis and contact angle calculation. Five specimens were measured in each group. The contact angles, determined with drops of ultrapure distilled water ( $0.6 \mu\text{l}$ ) were measured at room temperature.

Surface roughness was measured by profilometer (Surftest SV-400, Mitutoyo Instrument, Tokyo, Japan) by traversing over a length of 5 mm for three sites of five specimens of each group. Results are displayed as the arithmetic average peak-to-valley value (Ra).

### 4. Shear bond test

In this experiment, heat-cured PMMA resin (Vertex Rapid Simplified, Vertex Dental B.V., Zeist, The Netherlands) was used. The cylinder-shaped alloys were placed in cylinder mold (Figure 1 (a)). A silicone cap placed over the materials minimized air inclusions. The mold was then sandwiched and clamped between flasks. The mold with flask was treated for 30 min in water bath maintained at  $100^\circ\text{C}$ . The PMMA resin undergoes polymerization during this treatment. The mold after polymerization was cooled in air for 1 h. After air cooling, the mold was opened and the specimens were removed.

Shear bond strengths of RPD alloys to PMMA resin were determined using a universal testing machine (Model 3343, Instron Co., Canton, MT, USA) at a crosshead speed of 1.0 mm/min. By knowing the fracture load (N) and the bonded area the shear bonding strength is calculated in terms of MPa. The fractured surfaces were examined to assess alloy-resin bonding fracture mode. The fractured specimen interface was inspected under a field emission scanning electron microscope (FE-SEM) (JSM-6700, JEOL,

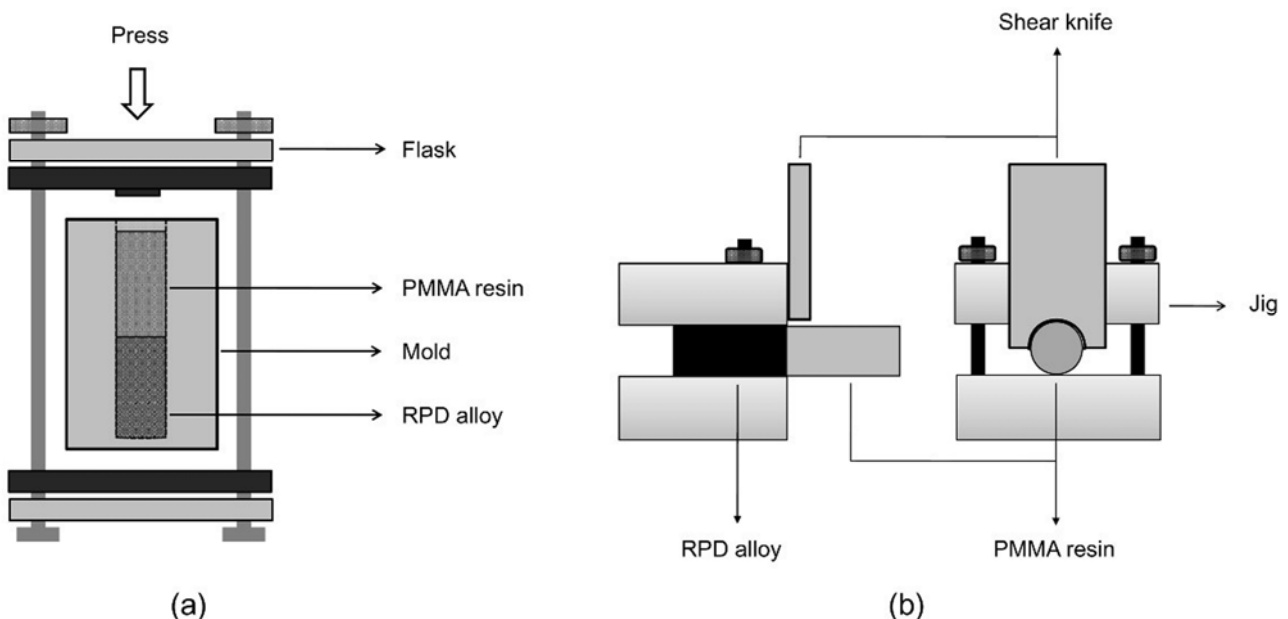


Figure 1. Apparatus for (a) polymerization, (b) shear bond strength test.

Tokyo, Japan) at 25X magnification. In addition, selected specimens of all the groups were examined in an energy dispersive X-ray spectroscopy (EDS) attachment of the SEM, to analyze the elementary composition of characteristic sites of the fractured surfaces.

## 5. Statistical analysis

All the statistical analyses were performed using SPSS 17.0 for Windows (SPSS, Chicago, IL, USA). The results for all groups were statistically analyzed using two-way analysis of variance (ANOVA) followed by Tukey's multiple comparison tests at a significance level of 0,05. The two factors analyzed were RPD alloy and surface treatment.

## Results

The results of the surface roughness for the all groups are presented in Table 2. Two-way ANOVA showed that there were no significant differences of the surface roughness (Ra) values for all groups ( $p > 0,05$ ). The contact angle results are summarized in Table 3. No significant differences were found between surface treatment groups within each Co-Cr alloy.

Table 4 shows the shear bonding strength results. One-way ANOVA showed that there were statistically significant differences between surface treatment groups within each RPD alloys ( $p < 0,001$ ). No significant difference was found in the shear bond strength between ST group and AST group, within each Co-Cr alloys. The

**Table 2.** Results (mean  $\pm$  SD) of surface roughness (Ra in nm)

Group	Biosil-F	Vera PDS- Hard	Group comparison*
Untreated (UT)	14 $\pm$ 5,5	18 $\pm$ 8,4	a
Silane-treated (ST)	16 $\pm$ 8,9	16 $\pm$ 8,9	a
Alkali-treated (AT)	16 $\pm$ 5,5	16 $\pm$ 8,9	a
Alkali/silane-treated (AST)	18 $\pm$ 8,4	18 $\pm$ 8,4	a
Group comparison**	A	A	

\*Same lowercase letter indicates no significant differences ( $p > 0,05$ ). \*\*Same uppercase letter indicates no significant differences ( $p > 0,05$ ).

**Table 3.** Results (mean  $\pm$  SD) of contact angle (  $^{\circ}$  ) measurements

Group	Biosil-F	Vera PDS- Hard	Group comparison*
Untreated (UT)	65,8 $\pm$ 7,2	66,7 $\pm$ 7,5	a
Silane-treated (ST)	59,6 $\pm$ 4,8	63,8 $\pm$ 4,1	a
Alkali-treated (AT)	57,2 $\pm$ 5,8	57,2 $\pm$ 6,4	a
Alkali/silane-treated (AST)	63,6 $\pm$ 6,4	63,2 $\pm$ 2,4	a
Group comparison**	A	A	

\* Different superscript numbers indicate significant differences between surface treatment groups within each alloy ( $p < 0,05$ ).

\*\* Different superscript lowercase letters indicate significant difference between alloys within each surface treatment ( $p < 0,05$ ).

**Table 4.** Results (mean  $\pm$  SD) of shear bond strength test

Group	Biosil-F	Vera PDS- Hard	Failure mode
Untreated (UT)	0,3 $\pm$ 0,2 <sup>1,a**</sup>	0,4 $\pm$ 0,2 <sup>1,a</sup>	Adhesive
Silane-treated (ST)	5,3 $\pm$ 1,0 <sup>2,b</sup>	7,1 $\pm$ 1,8 <sup>2,ab</sup>	Combination
Alkali-treated (AT)	0,2 $\pm$ 0,3 <sup>1,b</sup>	0,2 $\pm$ 0,1 <sup>1,b</sup>	Adhesive
Alkali/silane-treated (AST)	5,5 $\pm$ 1,0 <sup>2,a</sup>	7,1 $\pm$ 1,3 <sup>2,a</sup>	Combination

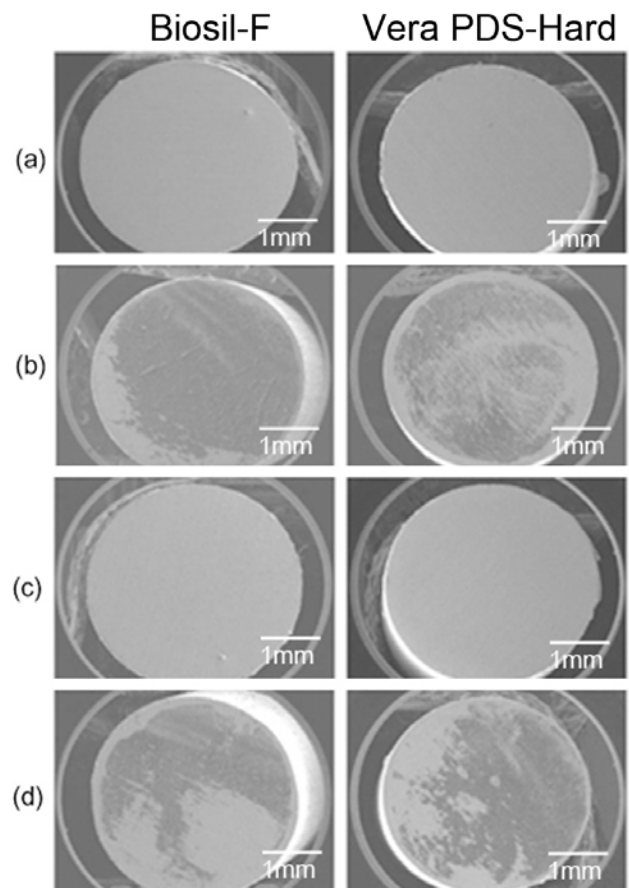
\* Different superscript number indicates significant differences between surface treatment within each alloy ( $p < .05$ ).

\*\* Different superscript lowercase letter indicates significant difference between alloys within each surface treatment ( $p < .05$ )

shear bond strengths of the UT group and AT group were lowest among the surface treatment groups in each RPD alloy.

In UT groups and AT groups the mode of fracture observed by SEM (Figure 2(a) and Figure 2(c)) were complete adhesive fracture between the alloys surface and the resin. The fracture mode in ST groups and AST groups revealed that in all specimens a combination (adhesive/cohesive fracture) type of fracture had occurred (Figure 2(b) and Figure 2(d)). The effect of silane is to change the fracture mode from adhesive to combination type.

Figure 3 shows the higher magnification SEM images of the fracture surfaces and the EDS of specific sites. The discriminated areas of a combination fracture specimens, revealed the presence of PMMA resin (sites A and B in Figure 3). For EDS analysis selected sites in SEM image are debonded PMMA resin surfaces (area marked as A, the upper part; area marked as B, the lower part) and RPD alloys surface (area marked as C). EDS area analysis indicate that, areas A and B contain higher amounts of carbon and oxygen than the metallic C area. Among them, the A area showed a higher carbon and oxygen amounts than the B area. However, no differences of silicon amounts were found between the areas A and B. The SEM and EDS analyses showed consistent results,



**Figure 2.** SEM images of the alloy surfaces after debonding: (a) UT, (b) ST, (c) AT, and (d) AST groups.

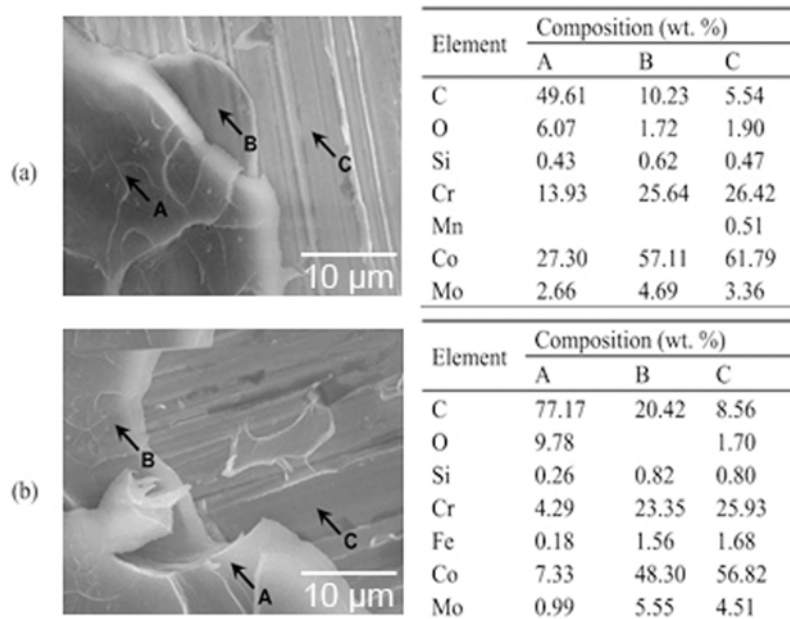


Figure 3. SEM images of the alloys of the AST group after debonding and corresponding EDS results: (a) Biosil-F and (b) Vera PDS-Hard.

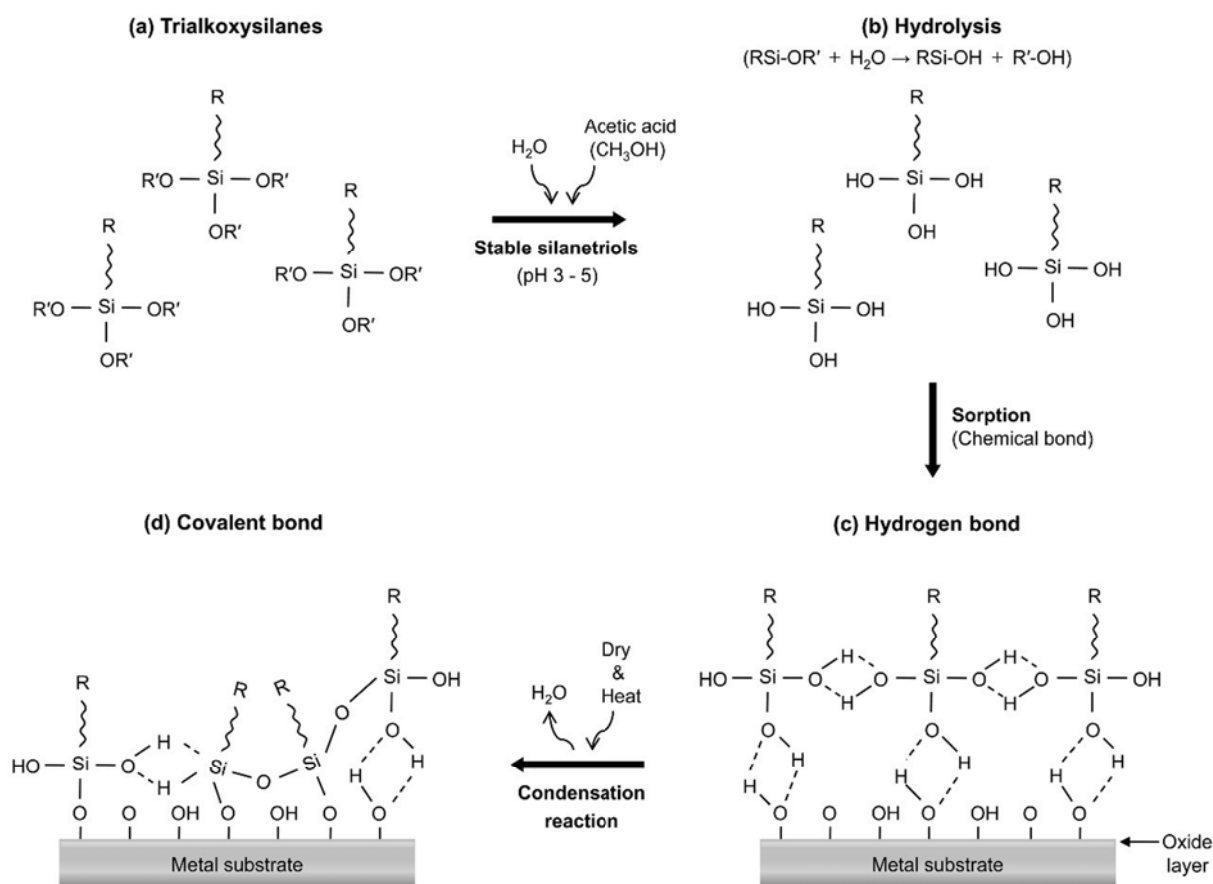


Figure 4. Schematic images of the chemical reactions of organotrialkoxysilane (R; Functional group, R' Alkoxy group).

## Discussion

The current study evaluated the effect of chemical surface treatments on the shear bond strengths between Co-Cr RPD alloys and PMMA resin. No significant differences in surface roughness were found between untreated group and surface treatment groups. The surface roughness for all the groups were very low. In previous studies (3-8, 16), mechanical roughening of metal surface increased the metal-resin bond strength. In our work, all the treatments provided on chemical bonding and there is no mechanical bonding. And so, the bond strengths were generally low. If the surface becomes rougher, it may be possible that bond strengths are higher than the present values.

In restorative dentistry, silanes are used either as a composite filler surface modification agent or as a coupling agent to bond the composite resin with ceramics/metals (17). A schematic image of the chemical reaction of MPS with the surface oxide layer is shown in Figure 4. The alkoxy groups react with acid in aqueous alcohol solution to form intermediate acidic silanol groups ( $-\text{Si}-\text{OH}$ ) at the first stage (Figure 4(b)). In the next fast step (Figure 4(c)), silane molecules are adsorbed onto the metal surface through hydrogen bonds formed between silanol groups and OH groups of the metal hydroxides. After condensation upon drying (Figure 4(d)), both  $\text{Si}-\text{O}-\text{M}$  ( $\text{M} = \text{metal}$ ) and  $\text{Si}-\text{O}-\text{Si}$  covalent bonds are formed at the interface, giving an excellent adhesion to the metal substrate, as well as a hydrophobic interface (18).

The surface modifications by silanes create a silicified layer on the metal. Therefore, a silica-coated surface will be silanized, typically with an MPS solution, and a dimethacrylate veneering resin or other prosthetic acrylic materials are cured onto it (19). However, the procedures are often complicated and basically require a custom apparatus. There is also the possibility of third type, where, a primer is applied on the metal (20). In chair-side clinical

applications when silanes are used, the curing is expected to happen at room temperature in the space of a few minutes. Therefore, the primer type is simple and can be easily used in clinical practice. However, metal primers are not always available for all types of dental metal alloys. And because application time of metal primer is short, stable bond between dental metal alloy and dental resin may not be achieved (21). If dental prostheses are made in the dental laboratory, prolonged application times can be used.

In this study, the RPD alloys were treated in silane solution (in ST and AST groups) for long hours to form stable bond between RPD alloys surface and silane. In addition alkali treatment created a lot of hydroxyl groups on the surface of RPD alloys. However, this effect was not seen the Co-Cr alloys. Co-Cr alloys showed strong corrosion resistance in alkaline solution and did not form sufficient bonding hydroxyl groups and so, the bonding and wettability was similar in both ST and AST conditions. Therefore, it seems that there was no effect of alkali pre-treatment over silane treatment in the Co-Cr alloys.

When the concentration of hydroxyl groups increased, the numbers of MPS molecular units bonding with hydroxyl groups are found to increase (22). Therefore, the shear bond strength of metal/polymer interface increased with increase in the concentration of hydroxyl groups on the surface oxide film (22). If drying temperature or time is not high or long enough after silane treatment, stable covalent bond may not form (Figure 3 (c) and (d)). If sufficient covalent bonds are not formed, bond strength of the silane treated surface will be low. Therefore, further research should be conducted in order to improve the bond durability between RPD alloys and PMMA resin, in the AST condition.

SEM images showed that, when untreated groups and alkali treated groups were employed in bonding RPD alloys to PMMA resin the mode of fracture of RPD alloys surface was adhesive fracture (Figure 2(a) and Figure



2(c)) with relative low bond strength values. And combination failure (Figure 2(b) and Figure 2(d)) indicates that, a stronger bond between RPD alloys and PMMA resin was established through the application of silane. Therefore, there is interaction between shear bond strength values and silane treatment. Using area analysis with the aid of EDS, silicon of silane was found on each fractured resin (Figure 3, sites A and B) surface after shear bonding test. Confirmation of fractured resin is derived from the almost exclusive presence of carbon and oxygen in this area. The elemental analysis performed at the RPD alloys surface (Figure 3, sites C) revealed decreased concentration of carbon and oxygen and increased concentration of each alloys composition, signifying the absence of silane on the alloy surfaces (Si element of Cr-Co alloys surface (Figure 3(c) and Figure 3(d)) is the trace element and not from the silane). However, on the fractured resin, EDS analysis revealed complete presence of silane, supported by the amounts of silicon element. This means that fracture of bond happens from bond layer of silane and resin. Therefore, in addition to the bond between silane and metal being important factor to improve the overall bond strength, the binding between silane and resin is also an important contributor this.

## Conclusion

Within the limitations of the present study, the following conclusions are drawn.

1. The untreated groups (UT) and alkali treated (AT) groups did not show any significant effect of on the RPD alloys-PMMA resin bond strength.
2. The silane treated (ST) groups and alkali followed by silane treatment (AST) groups showed similar shear bond strengths.
4. For silane treated (ST) groups and alkali followed by silane treatment (AST) groups, the mode of fracture were combination fracture between the PMMA resin and the RPD alloy surface.
5. With respect to bond strength, the use of any of the silane treatment in this study apparently would be acceptable for achieving clinically durable bonds of RPD alloys to PMMA resin.

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## Effect of silane and alkali treatment on the shear bond strength of acrylic resin to two Co–Cr alloys

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In this study, we investigated whether the silane and alkali treatments improve the chemical bonding between two chromium-chromium alloys and acrylic resin and thus the shear bond strength. A silane solution was prepared using 3-MPTMS. The alloy specimens were treated with silane and alkali, prepared as four experimental groups: untreated (UT), silane treated (ST), alkali treated (AT), and alkali/silane treated (AST) groups. In addition, the contact angles and surface roughness of the alloy surfaces of the four groups were measured. The acrylic resin was bonded to the alloy specimens, and then the resin was polymerized to form a bonded specimen. After measuring the shear bond strength, the failure modes were examined using scanning electron microscopy. Results for all groups were analyzed using two-way ANOVA followed by Tukey's multiple comparison test at a significance level of 0.05. The statistical analysis showed no significant differences in Ra value for all groups ( $p > 0.05$ ). In both alloys, the ST and AST groups showed relatively higher shear bond strengths ( $p < 0.05$ ) than the UT and AT groups. The UT and AT groups showed complete adhesive failure at the fractured surfaces. In this study, the silane treatment was shown to be effective to improve the shear bond strength, but the alkali treatment did not show any significant effect.

**Key Words** : Alkali, Acrylic resin, Co-Cr alloy, Shear bond strength, Silane

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