Evaluation of the effects of three pretreatment conditioners and a surface preparation system on the bonding durability of composite resin adhesive to a gold alloy

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This study aimed to evaluate the effect of three pretreatment conditioners and surface preparations on a composite resin adhesive for a gold alloy. Cast disk specimens were made and bonded with RelyX Unicem luting agent under six surface conditions: 1) polished with No. 600 carbide paper, 2) air-abraded with alumina, 3) Alloy Primer metal conditioner was applied after alumina-abrasion, 4) Monobond Plus multipurpose conditioner was applied after alumina-abrasion, 5) M. L. Primer metal conditioner was applied after alumina-abrasion, and 6) Rocatec multipurpose silica-coating system was applied. The bond strengths were determined before and after thermocycling (50,000 cycles). The bond strengths of the alumina-abrasion group were significantly decreased after thermocycling. The Rocatec and M. L. Primer exhibited higher bond strengths than other treatments. The application of metal conditioners and multipurpose surface preparations was recommended for improved bonding between the evaluated adhesive resin and gold alloy.

Keywords: Gold alloy, Bonding, Functional monomer, Silica-coating, Resin adhesive

INTRODUCTION

The strong bonding of luting agents to metal frameworks is an essential component in the clinical success of ceramic or composite-veneered metallic restorations and fixed prostheses. Improved resin luting materials and metal-resin bonding systems have been developed over the past few decades to create stronger and more durable chemical bonding.

Improved chemical bonding to metal/alloys has been achieved using pyrochemical or tribochemical surface modification techniques that create a silicated layer in combination with silane coupling agents or by the direct use of a functional monomer on metal surfaces1-3. Previous studies have reported the effects of the tribochemical coating system on the bond strength between several combinations of metal/ alloy or ceramic materials and luting agents4-7. It is important to consider the relationship between the type of metal and functional monomer when selecting a metal conditioner. For base metal alloys, carboxylic, phosphoric, and phosphonic acid derivative monomers result in the strongest bonds8-11, whereas thione and disulfide monomers are most effective for bonding to noble metal alloys12-16. In recent years, metal conditioners have been developed that are reported to exploit the combined effect of both acidic and sulfur-containing monomers on adhesion to noble and base metal/alloys17-19. In addition, a multipurpose conditioner designed for noble base metals and ceramic materials is also commercially available20,21.

Another alternative is the use of a composite adhesive that contains functional monomers in the luting base, commonly called self-etch or self-adhesive luting agents. These materials were designed to simplify the seating procedure as well as the conventional luting agents (zinc phosphate, carboxylic, or glass-ionomer luting materials) while achieving superior adhesion to tooth structures, metals, or porcelains22-24. There have also been several improvements in the chemical compositions of monomers and initiators to enhance their clinical performance25,26.

Although adhesive resin luting agents and resin-metal bonding systems have evolved, little information about their effects on adhesion to noble alloys used for prostheses is available27,28. The purpose of the study was to evaluate the effects of surface treatments on bonding between a gold alloy and composite adhesive luting agents. A conditioner and silica-coating system for metals and ceramic materials bonding pretreatment, as well as two metal conditioners effective for use with noble and base metals, were evaluated. The null hypotheses were as follows: 1) surface pretreatments to gold alloy would not improve the bond strength, and 2) there would be no influence of thermal stress on bond strength.

MATERIALS AND METHODS

A gold alloy designed for low-fusing ceramic applications such as restorations and denture frameworks (Pontol LFC, Metolor Dental, Oensingen, Switzerland) was used as the adherend. Three conditioners (Alloy Primer, Kuraray Medical, Tokyo, Japan; M. L. Primer, Shofu, Kyoto, Japan; Monobond Plus, Ivoclar Vivadent, Schaan, Switzerland) were evaluated. The bond strengths were determined before and after thermocycling (50,000 cycles).

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Liechtenstein) and a one-surface coating system (Rocatec system, 3M Oral Care, St. Paul, MN, USA; ROC) were assessed. The conditioners included a single-liquid sulfur-containing monomer promoting improved bonding to noble metals and an acidic functional monomer most effective for the bonding of base metals. In addition, trimethoxysilane-methacrylate was dissolved in the Monobond Plus conditioner as a silane coupling agent. The adhesive luting agent, the RelyX Unicem clicker (3M Oral Care), was also assessed. Information on the materials used in this study is presented in Table 1.

### Specimen preparation

A total of 72 disk specimens measuring 10 mm in diameter by 2.5 mm in thickness were cast from Pontol LFC. All disks were sanded with 600-grit silicon-carbide abrasive paper. After cleaning with air at a pressure of 0.4 MPa for 10 s, the disks were divided into six sets of 12 specimens. Five of the sets were divided into groups according to the type of surface treatment they received: 1) air-abraded with 50–70-µm particle alumina (AA), 2) application of Alloy Primer conditioner after the AA treatment (ALP), 3) application of M. L. Primer conditioner after the AA treatment (MLP), 4) application of Monobond Plus conditioner after the AA treatment (MBP), and 5) air-abraded with 110-µm alumina and silica-coated alumina (ROC-abrasion), followed by application of a silane coupling agent (ROC). The remaining 12 specimens cleaned with air for 10 s after No.600 sanded were used as controls (CONT). After air-abrasion of alumina or silica-coated alumina, the disks were cleaned with air at a pressure of 0.4 MPa for 10 s following the conditioners or the silane coupling agent applied to the metal surfaces using sponge pellets. The ALP, MLP, and MBP specimens were left to dry for 60 s, and the ROC specimens were dried for 300 s.

A piece of double-coated tape with a hole measuring 5-mm in diameter was placed on each specimen to define the bonding area, and a brass ring (6-mm inside diameter, 2-mm length, and 1-mm wall thickness) was placed to surround the opening. After the mixing of the luting paste, the ring was filled with the adhesive luting agent, whereas a 5 N load was applied to the filled ring using an aluminum disk (7-mm diameter and 1-mm thickness) for 10 s. The excess resin that extruded from the circumferential rings was irradiated using a conventional 75W quartz-tungsten-halogen-lump light-curing unit (JETLITE 1000, J Morita Mfg., Kyoto, Japan) for 20 s to make it easy to remove. The polymerized excess resin was carefully removed using a dental explorer.

### Shear testing and surface observation

Thirty minutes after preparation, the specimens were immersed in water at 37°C for 24 h. This state was defined as thermocycle 0 and defined as the baseline. Half of the total number of specimens were then tested for shear bond strength. The remaining specimens were

| Materials                        | Manufacturer/lot no.                              | Composition                                           |
|----------------------------------|--------------------------------------------------|-------------------------------------------------------|
| **Casting alloy**                |                                                  |                                                       |
| Pontol LFC                       | Metalor Dental, Oensingen, Switzerland/1830999    | Au 69.4, Ag 11.8, Pt 9.4, Cu 6.0, Zn 1.8, In 1.5, Ir <1.0 wt% |
| **Conditioner**                  |                                                  |                                                       |
| Alloy Primer                     | Kuraray Medical, Tokyo, Japan/00398A              | MDP¹ and VTD² in acetone                              |
| Monobond Plus                    | Ivoclar vivadent, Schaan, Liechtenstein/P00147    | Phosphoric acid-methacrylate, disulfide-methacrylate, Trimethoxysilane-methacrylate in ethanol |
| M. L. Primer                     | Shofu, Kyoto, Japan/061154                        | MDDT³ and MHPA⁴ in acetone                            |
| **Surface coating system**       |                                                  |                                                       |
| Espe Sil                         | 3M Oral Care, St Paul, MIN, USA/431877            | MPTS⁵ in ethanol                                      |
| Rocatec Pre                      | 3M Oral Care/279500                               | 110-µm alumina particle                              |
| Rocatec Plus                     | 3M Oral Care/306303                               | 110-µm silica-coated alumina particle                 |
| **Resin adhesive**               |                                                  |                                                       |
| RelyX Unicem clicker             | 3M Oral Care/447236                               | Glass, silica, phosphate-methacrylate, Bis-GMA⁶, TEGDMA⁷, Sodium persulfate |

¹10-methacryloyloxydecyl dihydrogen phosphate, ²6-(4-vinylbenzyl-n-propyl) amino 1,3,5-triazine-2,4-dithiol, dithione tautomer, ³10-methacryloyloxydecyl 6,8-dithiooctanoate, ⁴6-methacryloyloxhexyl phosphonoacetate, ⁵3-methacryloyxypropyl trimethoxysilane, ⁶bisphenol A-glycidyl methacrylate, ⁷triethyleneglycol dimethacrylate.
placed in the thermocycling apparatus (Thermocycling machine, Rika-Kogyo, Tokyo, Japan) and cycled in water (50,000 cycles) between 5°C and 55°C with a dwell time of 1 min. Each specimen was embedded into an acrylic resin mold and seated in an ISO/TR 11405 shear-testing jig. A universal testing machine (AGS-5kNG, Shimadzu, Kyoto, Japan) was used to determine the shear bond strength at a cross-head speed of 0.5 mm/min. After shear testing, the specimen surfaces were observed using an optical microscope (SMZ-10, Nikon, Tokyo, Japan) under ×20 magnification.

Six specimens, after being abraded with No. 1000 silicon-carbide paper and undergoing the application of ROC-abrasion or AA, were examined for each treatment using a scanning confocal laser microscope (VK-8500, Keyence, Osaka, Japan). To evaluate surface morphological changes with the air-abrasion especially, sanded with No. 1000 silicon-carbide paper group was used as control. The two-dimensional surface profile was measured, and the parameters were calculated. The calculated parameters included the mean width of the profile element (RSm) —a spacing parameter— and the maximum height of the assessed profile (Rz) values —an amplitude parameter. The examined area was comprised of three randomly selected surface points, with a total of 18 points evaluated for each preparation.

The surface composition of AA, a ROC-abrasion specimen, was observed using electron probe X-ray microanalysis (EPMA; JXA-8230, JEOL, Tokyo, Japan) with an acceleration voltage of 15 KeV and probe current of 0.1 nA, after spattering with a carbon layer.

**Statistical analysis**

The mean and standard deviation were calculated from the shear-testing results and measurements of surface characteristics. All data were primarily analyzed using the Shapiro-Wilk test to assess the normality of their distributions. If the data were normally distributed, Tukey’s honestly significant difference (HSD) test was also performed. If the primary test did not show normality, the median and interquartile range were calculated and determined using the Steel-Dwass test. In addition, the differences in the application of thermocycling were compared using Mann-Whitney’s U test. The threshold for statistical significance was set at p<0.05. All statistical analyses were performed using JMP 10.0 software for Macintosh (SAS Institute Japan, Tokyo, Japan).

**RESULTS**

The data were analyzed using the Steel-Dwass test for thermocycle 0 and Tukey’s HSD test for 50,000 cycles. The shear-testing data, statistical groupings, and results of Mann-Whitney’s U tests are presented in Table 2. The ROC system exhibited the highest bond strengths, followed by the MLP and ALP groups before thermocycling, and there were no statistically significant differences between the ALP, MBP, and AA groups. Tukey’s HSD test indicated that ROC and MLP produced higher bond strengths than MBP, ALP, or AA. As shown in Table 2, the bond strength after thermocycling was significantly lower than the strength observed before thermocycling only in the AA group. After shear bond testing, the failure mode of all CONT specimens showed adhesive failures, whereas the other specimens demonstrated a combination of cohesive and adhesive failure before and after thermocycling.

The results from statistical analyses of the Rz and RSm values are presented in Table 3. Tukey’s HSD test was performed on the Rz data for each surface treatment, and the results were divided into two groups.

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**Table 2 Shear testing results (in MPa)**

| Surface condition | Thermocycle 0 median (IQR) | Statistical grouping | 50,000 Cycles mean (SD) | Statistical grouping | Mann-Whitney’s U test |
|-------------------|-----------------------------|----------------------|------------------------|----------------------|----------------------|
| AA                | 7.8 (2.1)                   | C                    | 4.7 (1.6)              | b                    |                      |
| ALP               | 9.9 (2.0)                   | B, C                 | 8.8 (2.9)              | b                    |                      |
| CONT              | 3.9 (0.7)                   | D                    | 0.1 (0.1)              | c                    |                      |
| MBP               | 8.4 (1.5)                   | C                    | 8.8 (2.2)              | b                    |                      |
| MLP               | 14.8 (3.0)                  | B                    | 14.1 (2.7)             | a                    |                      |
| ROC               | 17.0 (4.1)                  | A                    | 15.5 (3.7)             | a                    |                      |

AA: air abrasion with 50–70-µm particle alumina only.
ALP: air abrasion with 50–70-µm particle alumina+Alloy Primer.
CONT: sanded with 600-grit silicon-carbide abrasive paper.
MBP: air abrasion with 50–70-µm particle alumina+Monobond Plus.
MLP: air abrasion with 50–70-µm particle alumina+M.L. Primer.
ROC: application of Rocatec tribochemical silica-coating system.

* Steel-Dwass grouping and b Tukey’s HSD grouping. There is no significant difference between identical letters (p>0.05).

Mann-Whitney U test: the asterisk indicates the absence of a significant difference (p>0.05).
Table 3 Surface characteristics, parameters (RSm and Rz), values (in µm), and statistical groupings

| Surface condition | RSm Median (IQR) | Grouping | Rz Mean (SD) | Grouping |
|-------------------|------------------|----------|--------------|----------|
| No.1000           | 8.1 (4.5)        | a        | 10.4 (0.6)   | A        |
| ROC-abrasion      | 7.6 (0.6)        | a        | 13.5 (0.7)   | A        |
| AA                | 6.4 (0.1)        | a        | 13.0 (0.8)   | B        |

No. 1000: abraded with No. 1000 silicon-carbide paper, ROC-abrasion: application of air-abrasion with Rocatec Pre and Plus, AA: application of air-abrasion with 50–70-µm particle alumina.

*Steel-Dwass grouping and *Tukey’s HSD grouping. Identical letters indicate no significant difference (p>0.05).

The present study evaluated the bond strength of a composite resin adhesive material jointed to gold alloy with surface pretreatments. The air-abrasion of metals using alumina particles was recommended for most adhesive resin luting agents before bonding. Alumina-abrasion to metals creates high surface energy, promotes micro-retention, and increases the wettability of the surface\(^3,29\). Although the RelyX Unicem luting material contained phosphate-methacrylate as the adhesive functional monomer, the alumina-abrasion group had the lowest bond strength both before and after thermocycling, with a significant reduction in bond strength resulting from the thermal stress. Previous studies have reported that acidic functional monomers have weaker chemical reactions with noble alloys because they lack a surface oxide layer\(^19,22\). These findings and results indicate a need for additional chemical modification after alumina-abrasion.

The shear-testing results showed that ROC effectively enhanced bonding. The bonding efficiency using the ROC system and Pontol LFC alloy agrees with the results of a previous study, in which data indicated comparable bonding with different composite resin materials after the application of 20,000 cycles of thermal stress on the ceramic-to-metal bond\(^30\). The ROC tribochemical system introduced the silica-coating method with alumina-abrasion, followed by impact silica-coated alumina onto adherend materials. The types of alloys and alumina particle sizes influenced the number of particles retained on the abraded surface\(^31,32\), which were suspected to affect the Rz and RSm values. With respect to the surface characteristics, the comparison of Rz values showed that the No. 1000 treatment produced lower values than the others, whereas there were no significant differences between the RSm values regardless of the treatment protocol used. Moreover, the measurement of the chemical composition of the noble alloy (Au-Ag-Cu) using X-ray energy dispersive spectroscopy determined that 1.5 wt% of residual alumina remained on the alloy surface after alumina-abrasion using 110-µm alumina particles, whereas 16.4 wt% of silica and 14.3 wt% of alumina remained after the ROC treatment\(^3\). Although a direct comparison with the current study is not possible because different noble alloys were used, it was considered that residual particles of alumina and silica also remained on the surface of the Pontol LFC alloy. The EPMA images revealed that the element Si or Al was observed on the surface of Pontol LFC alloy after ROC-abrasion and/or AA preparations (Figs. 1A–C). The ROC system requires the application of an ESPE Sil silane coupling agent to the silicated layer (2 vol% 3-methacryloyloxypropyltrimethoxysilane [MPTS] in ethanol) to form siloxane bonds with the silicated layer\(^5,30\), which may also form partial chemical bonds with the oxide layer of the remaining alumina particles.

**DISCUSSION**

The present study evaluated the bond strength of a composite resin adhesive material jointed to gold alloy with surface pretreatments. The air-abrasion of metals using alumina particles was recommended for most adhesive resin luting agents before bonding. Alumina-abrasion to metals creates high surface energy, promotes micro-retention, and increases the wettability of the surface\(^3,29\). Although the RelyX Unicem luting material contained phosphate-methacrylate as the adhesive functional monomer, the alumina-abrasion group had the lowest bond strength both before and after thermocycling, with a significant reduction in bond strength resulting from the thermal stress. Previous studies have reported that acidic functional monomers have weaker chemical reactions with noble alloys because they lack a surface oxide layer\(^19,22\). These findings and results indicate a need for additional chemical modification after alumina-abrasion.

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The MLP metal conditioner indicated comparable post-thermocycling bond strength to the ROC treatment, whereas the ALP and MBP conditioners resulted in similar bonding values to the alumina-abrasion. The three conditioners represented two different types of functional monomers, dissolved in the solvent, that are effective for the bonding of noble or base metals. In addition, the MBP conditioner also includes trimethoxysilane-methacrylate as a silane coupling agent. The 10-methacryloyloxydecyl 6,8-dithiooctanoate (MDDT) monomer in the MLP and the disulfide-methacrylate in the MBB have cyclic disulfide groups, whereas the 6-(4-vinylbenzyl-n-propyl) amino 1,3,5-triazine-2,4-dithiol, dithione tautomer (VTD) in the ALP conditioner has thione groups on the terminal monomer structure. The effectiveness of the sulfur-based functional monomers for the bonding of noble metals has been reported. Shear testing of the noble alloy bonded using the MBP conditioner with different composite luting materials showed a bonding strength of 16.5 MPa after thermocycling (37,500 cycles), similar to the bonding strength achieved using the ALP conditioner. In a study by Okuya et al., the MDDT monomer was reported to be effective for the bonding of pure gold or high-gold content (78 wt%) alloys with acrylic resin adhesive after 2,000 thermocycles, whereas the VTD monomer was reported as most effective in bonding with pure silver, copper, and silver-palladium-copper alloys. Similarly, the VTD monomer was reported to exhibit high bonding durability when joined to type IV gold or silver-palladium-copper alloys, which was speculated to result primarily from interactions between the thione or thiol groups and copper elements contained in both alloys. Previous studies have reported that the bonding mechanism of the VTD monomer to noble metal alloys involves an ionic interaction between mercapto groups in the VTD and metal ions in the alloys, especially gold, silver, or copper. With respect to the bonding mechanism of the MDDT monomer with pure gold, Ikemura et al. suggested that the cyclic disulfide group is cleaved at the disulfide bond or ring-opening and interacts with the gold atoms to form a sulfur-gold bond at the metal surface. As shown in Table 1, the adherend alloys evaluated contained primarily gold but also included silver, platinum, copper, and others. The lower rates of silver and copper in the metal alloy compositions may have affected the bonding results of the VTD monomer, whereas the MDDT monomer was actually more effective because of the higher rate of gold (69.4 wt%) alloy used in this study. In the EPMA mapping images, a large ratio of Au was observed with oxide-coated surfaces. The interactions between these monomers and base metals occurred simultaneously with interactions between sulfur-based monomers and noble alloys; however, these interactions did not interfere with each other in terms of their respective adhesion behaviors.

Although the MBP conditioner contains cyclic disulfide-methacrylate, phosphoric acid-methacrylate, and trimethoxysilane-methacrylate with similar structures to the functional groups found in the MDDT, MDp, or MPTS monomers, respectively, it exhibited a significantly different bonding strength from that exhibited by the member of the MLP group. This could be attributed to a difference in monomer concentration or chemical composition, particularly with respect to monomer structures. Further investigation is needed to better define differences in bonding behavior.

The two null hypotheses were rejected because the ROC and MLP groups exhibited greater bonding durability than others, whereas the bond strength of the AA was significantly reduced by the application of thermal stress. The results of the present study suggest that air-abrasion with 50–70 μm alumina particles, followed by the application of M. L. Primer metal conditioner or a multipurpose tribochemical silica-coating system are both effective for achieving durable bonding between a high-gold alloy and composite resin adhesive. This might result from an effective chemical interaction between the functional groups of adhesive monomers and target elements on the alloy surface. Additional research is necessary to clarify details of the bonding mechanisms and provide additional experimental evidence in support of these results.

CONCLUSIONS

Within the limitations of this study, the following conclusions are drawn:

1. Additional chemical treatment is needed after air-abrasion of alumina.
2. The metal conditioner contained the cyclic disulfide functional monomer, as well as the multipurpose silica-coating system, is effective for bonding to an evaluated high-gold alloy.

CONFLICTS OF INTEREST

None declared.

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