Effect of thione and phosphate priming agents on the bonding to a gold alloy of three composite luting agents

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Purpose: The purpose of this study was to evaluate the effect of metal priming agents on the bond strength between a gold alloy and composite luting agents.
Materials and Methods: Disk specimens were cast from a gold alloy (Degudent U) and air-abraded with alumina. Four priming agents (Alloy Primer, Estenia Opaque Primer, Metaltite, and V-Primer) were applied separately to the abraded surface. Unprimed specimens served as the control. Three composite luting agents (Esthetic Cement, Panavia F2.0, and SA Luting) were inserted into the brass ring on the cast disks and were chemically-polymerized. The specimens were stored in 37˚C water for 24 hours and subsequently thermocycled (5˚C-55˚C, 1-1 minute, 20,000 cycles). The shear bond strengths were determined, and the results were analyzed using the Steel-Dwass multiple comparison test.
Results: The application of Metaltite enhanced the bond strength of three luting agents. The Estenia Opaque Primer, V-Primer, and Alloy Primer were effective in bonding the gold alloy and Esthetic Cement, whereas the bond strength of the SA Luting did not improve with use of the Estenia Opaque Primer, V-Primer, or Alloy Primer.
Conclusion: Thione-based priming agents (Alloy Primer, Metaltite, and V-Primer) were substantially effective for bonding the gold alloy with specific composite luting agents. **(Asian Pac J Dent 2011; 11: 55-60.)**
Key Words: bonding, composite, gold alloy, luting agent, primer

Introduction

The application of composite luting agents for seating metallic restorations has increased substantially.[1-13] This trend is likely attributed to development of priming agents compatible with both base and noble metal alloys. Gold alloys have been used as coping materials in the porcelain-fused-to-metal (PFM) restorative systems. It is desirable that metallic material and dentin can be bonded durably.

Many articles reported the usefulness of various luting agents for bonding noble metal alloys including gold alloys for PFM systems.[14-19] Currently available luting agents are based on a hydrophobic phosphate monomer for the enhancement of bond strength to tooth structure,[2] base metal alloys,[3,4] and zirconia.[20]

Although varoious adhesive systems are being introduced for bonding tooth structure and PFM restorations, limited information is available about bonding characteristics of dual-activated composite luting agents, especially as they relate to the bonding of noble metal alloys.[13] The purpose of the present study was to evaluate the effect of priming agents on the bond strength and durability of three luting agents joined to a gold alloy.

Materials and Methods

The materials used in the current project are summarized in Table 1. A gold alloy designed for a metal ceramic restoration (Degudent U, DeguDent, Hanau, Germany) was used as the adherend material. Four priming agents were tested: Alloy Primer (Kuraray Medical Inc., Tokyo, Japan), Estenia Opaque Primer (Kuraray Medical Inc.), Metaltite (Tokuyama Dental Corp., Tokyo, Japan), and V-Primer (Sun Medical Co., Ltd., Moriyama, Japan). The Alloy Primer contains two functional monomers; 10-methacryloyloxydecyl dihydrogen phosphate (MDP) and 6-(4-vinylbenzyl-*n*-propyl)amino-1,3,5-triazine-2,4-dithione (VTD). The Estenia Opaque

Primer contains MDP. Metaltite contains 6-methacryloyloxyhexyl 2-thiouracil-5-carboxylate (MTU-6), and the V-Primer contains VTD. Three luting agents, Esthetic Cement, Panavia F2.0, and SA Luting (Kuraray Medical Inc.) were tested. Each of the luting agents consists of two pastes and is dual-cure, i.e., cured by redox reaction and visible light. The Panavia F2.0 and SA Luting agents contain MDP.

Table 1. Materials assessed

Material (Manufacturer)	Lot number	Composition
Gold alloy		
Degudent U (DeguDent)	10011271	Au 77.3, Pt 9.8, Pd 8.9, Ag 1.2, Cu 0.3, Others 2.5 (mass%)
Metal priming agents		
Alloy Primer (Kuraray Medical)	00321A	MDP, VTD, Acetone
Estenia Opaque Primer (Kuraray Medical)	00168A	MDP, Monomer solvent
Metaltite (Tokuyama Dental)	025	MTU-6, Alcohol
V-Primer (Sun Medical)	VG1	VTD, Acetone
Luting agents		
SA Luting (Kuraray Medical)	Paste A 00122A	MDP, Bis-GMA, TEGDMA, BPO, Hydrophobic aromatic dimethacrylate, Silanated barium glass, Silanated colloidal silica, *dl*-Camphorquinone, initiator
	Paste B (Universal) 00122A	Bis-GMA, Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic dimethacrylate, Silanated barium glass, Silanated colloidal silica, Surface treated sodium fluoride, Accelerators, Pigments
Panavia F2.0 (Kuraray Medical)	Paste A 004103A	MDP, Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic dimethacrylate, Hydrophilic aliphatic dimethacrylate, Silanated silica, Silanated colloidal silica, *dl*-Camphorquinone, Initiators
	Paste B (Light) 00073B	Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic dimethacrylate, Hydrophilic aliphatic dimethacrylate, Silanated barium glass, Initiators, Accelerators, Pigments
Esthetic Cement (Kuraray Medical)	Paste A 00163C	Bis-GMA, TEGDMA, Hydrophobic aromatic dimethacrylate, Silanated barium glass filler, Colloidal silica, Accelerators
	Paste B (Universal) 00163C	Bis-GMA, TEGDMA, BPO, Hydrophobic aromatic dimethacrylate, Hydrophilic aliphatic dimethacrylate, Silanated silica, Silanated barium glass, Colloidal silica, *dl*-Camphorquinone, Initiators, Accelerators, Pigments

MDP, 10-methacryloyloxydecyl dihydrogen phosphate; VTD, 6-(4-vinylbenzyl-*n*-propyl) amino-1,3,5-triazine-2,4-dithione; MTU-6, 6-methacryloxyhexyl 2-thiouracil-5-carboxylate; Bis-GMA, bisphenol A diglycidylmethacrylate; TEGDMA, triethyleneglycol dimethacrylate; BPO, benzoyl peroxide

A total of 165 disks (10 mm in diameter and 2.5 mm in thickness) were cast from the gold alloy. The disk surfaces were ground with a #1,500-grit silicon carbide abrasive paper and air-abraded with 50 µm alumina at an air pressure of 0.2 MPa for 10 s at a distance of 20 mm. A piece of tape with a 5.0-mm-diameter circular hole was then positioned on the surfaces of the disks to define the bonding area. The disks were divided into 15 test groups consisting of three luting agents and five surface conditions, i.e., four priming agents and an unprimed control. Eleven specimens were prepared for each group. For the primed groups, one of the priming agents was applied to the bond areas and then air-dried. A brass ring (6.0 mm in inner diameter and 2.0 mm in height) was positioned on the metal surfaces surrounding the 5-mm diameter area. Each luting agent was mixed according to the manufacturer's instructions and incrementally inserted into the brass ring. Specimens were stored in the dark box for 30 minutes and then stored in distilled water at 37˚C for 24 hours. All specimens were thermocycled between 5˚C and 55˚C water for 20,000 cycles with a 60-s dwell time per bath (Thermal Shock Tester TTS-1 LM, Thomas Kagaku Co., Ltd., Tokyo, Japan). The shear bond strength was determined with a mechanical testing device (Type 5567, Instron Corp., Canton, MA, USA) at a crosshead speed of 0.5 mm/minute.

Statistical analyses were performed using two statistical software packages (SPSS version 15.0, SPSS Inc., Chicago, IL, USA; Kyplot 5.0, KyensLab Inc., Tokyo, Japan). The distributions of the results and the equality of the variances were analyzed by the Kolmogorov-Smirnov test and Levene test, respectively (SPSS version 15.0). If a normal distribution or an equality of variance was not found in some groups, the Steel-Dwass multiple comparison test was performed (Kyplot 5.0). A significance level of 5% was used for each analysis.

The debonded surfaces were observed through an optical microscope (SZX9, Olympus Corp., Tokyo, Japan) at 8x magnification to perform the failure mode analysis. The failure modes of the tested specimens were classified as follows: A, adhesive failure at the luting agent-alloy interface; C, cohesive failure within the luting agent; and AC, a combination of adhesive and cohesive failures.

Selected specimens were cross-sectioned into slabs with a low-speed diamond saw with water coolant. After an osmium coating was applied with a sputter coater (HPC-1S, Vacuum Device, Mito, Japan) for 30 s, the specimens were observed with a scanning electron microscope (SEM, S-4300, Hitachi High- Technologies Corp., Tokyo, Japan) with an accelerating voltage of 15 kV.

Results

The Levene test run on the bond strength results revealed inequality of variance in several groups. The Steel-Dwass test therefore was applied to the shear testing results. The shear bond strengths, statistical categories, and failure modes are summarized in Tables 2 and 3.

Table 2 presents the effect of two thione primers on the bond strengths between the Degudent U alloy and three luting agents. The shear bond strength ranged from 11.8 to 23.3 MPa for SA Luting, 15.3 to 24.2 MPa for Panavia F2.0, and 5.4 to 8.2 MPa for Esthetic Cement. The application of Metaltite improved the bond strength of three luting agents, whereas the application of the V-Primer was effective for the Panavia F2.0 and Esthetic Cement agents ($p<0.05$). Among the results in Table 2, the bond strength of Esthetic Cement was considerably lower than those of Panavia F2.0 and SA Luting.

Table 2. Effect of two thione primers on the bond strength to gold alloy of three luting agents

Luting agents	Priming agents	Post-thermocycling shear bond strength (MPa)				Failure mode		
		Median	Mean	SD	Category	A	AC	C
SA Luting	Unprimed	11.8	12.4	5.8	b	11	0	0
	V-Primer	15.7	15.6	2.0	b	10	1	0
	Metaltite	23.3	23.3	3.4	a	11	0	0
Panavia F2.0	Unprimed	15.3	14.3	4.3	d	10	1	0
	V-Primer	20.2	20.3	2.4	c	11	0	0
	Metaltite	24.2	23.6	3.8	c	11	0	0
Esthetic Cement	Unprimed	5.4	5.1	1.3	f	11	0	0
	V-Primer	7.6	7.6	1.1	e	9	2	0
	Metaltite	8.2	8.0	1.7	e	10	1	0

Thermocycling, 20,000 cycles; Identical letters indicate no significant difference (Steel-Dwass test, $p>0.05$). A, Adhesive failure at the luting agent-alloy interface; AC, Combination of adhesive and cohesive failures; C, Cohesive failure within the luting agent

Table 3 shows the effects of the three primers on the bond strength to the gold alloy of three luting agents. The shear bond strength ranged from 11.8 to 15.7 MPa for SA Luting, 14.7 to 20.2 MPa for Panavia F2.0, and 5.4 to 11.1 MPa for Esthetic Cement. The application of the three primers improved the bond strength of Esthetic Cement, whereas the V-Primer was effective for Panavia F2.0 ($p<0.05$). Additionally, it is important to note that considerable amount of specimens primed with the Alloy Primer exhibited the CA-type failure.

Table 3. Effect of thione and phosphate primers on the bond strength to the Dugudent U gold alloy

Luting agents	Priming agents	Post-thermocycling shear bond strength (MPa)				Failure mode		
		Median	Mean	SD	Category	A	AC	C
SA Luting	Unprimed	11.8	12.4	5.8	A	11	0	0
	Estenia Opaque Primer	13.3	13.7	1.6	A	11	0	0
	V-Primer	15.7	15.6	2.0	A	10	1	0
	Alloy Primer	15.3	13.6	3.5	A	11	0	0
Panavia F2.0	Unprimed	15.3	14.3	4.3	C	10	1	0
	Estenia Opaque Primer	14.7	15.0	1.4	C	9	2	0
	V-Primer	20.2	20.3	2.4	B	11	0	0
	Alloy Primer	16.6	17.3	2.9	C	3	8	0
Esthetic Cement	Unprimed	5.4	5.1	1.3	F	11	0	0
	Estenia Opaque Primer	8.1	8.1	1.2	D E	9	2	0
	V-Primer	7.6	7.6	1.1	E	9	2	0
	Alloy Primer	11.1	12.3	5.3	D	6	5	0

Thermocycling, 20,000 cycles; Identical letters indicate no significant difference (Steel-Dwass test, p>0.05). A, Adhesive failure at the luting agent-alloy interface; AC, Combination of adhesive and cohesive failures; C, Cohesive failure within the luting agent, Estenia Opaque Primer, phosphate (MDP); V-Primer, thione (VTD); Alloy Primer, both phosphate (MDP) and thione (VTD)

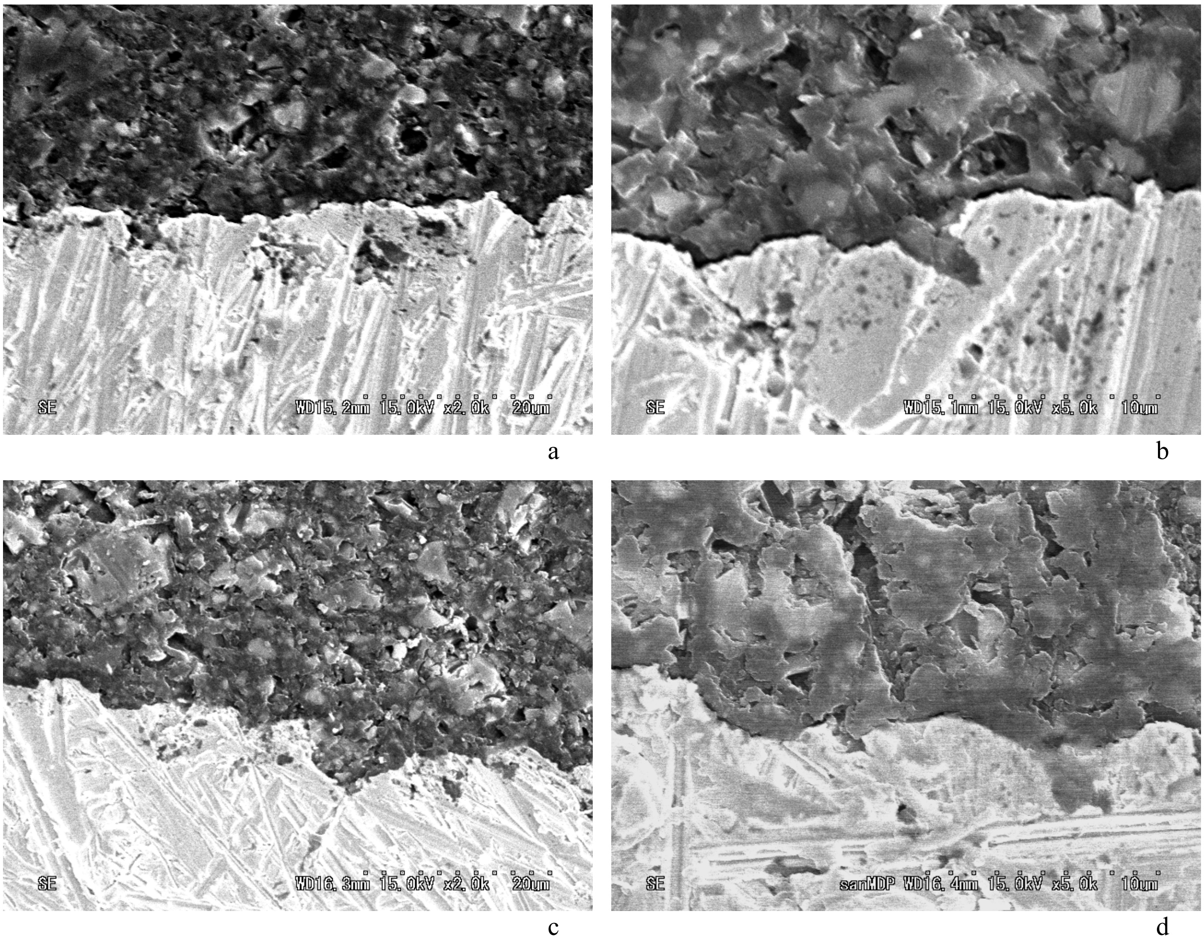

Fig. 1. Scanning electron micrographs of interfaces between Degudent U and Esthetic Cement
a and b, unprimed specimens (pre-thermocycling); c and d, primed with Estenia Opaque Primer (pre-thermocycling)

Figure 1 presents the scanning electron micrographs of the pre-thermocycling adhesive interface between the gold alloy and Esthetic Cement. Interspaces between the alloy and the luting agent were observed in the unprimed specimens (Fig. 1a and 1b), whereas no gaps were seen in the primed specimens (Fig. 1c and 1d).

Discussion

This study aimed to evaluate a total of 15 bonding systems on adhesive bonding to a gold alloy. A gold alloy designed for PFM restorations was selected as the adherend material. This selection was based on the fact that a growing number of collarless PFM restorations are seated with composite luting agents after surface preparations of dentin, porcelain, and metallic substrates.

Among the three luting agents, Esthetic Cement was released as a successional material of the Clapearl DC luting agent. These materials are designed for seating ceramic restorations. Light exposure after seating the restoration is required, even if the composition has a dual-activation mode. However, light exposure activation was not performed for all of the specimens in this study. The authors speculate that the low bond strength for Esthetic Cement was likely due to insufficient polymerization of the monomers.

This hypothesis is supported by the micrographs shown in the Fig. 1. The phosphate monomer MDP in the Estenia Opaque Primer is ineffective for bonding noble metals. Bond strength of Esthetic Cement, however, improved with the application of the Estenia Opaque Primer. The Estenia Opaque Primer contains both MDP and a redox initiator. It is reasonable to consider that the reduction in contraction gap between the gold alloy and Esthetic Cement is due to an improved polymerization reaction at the metal-resin interface with the application of Estenia Opaque Primer.

Panavia EX and other Panavia luting agents were developed for seating metallic restorations. The application of an opacious material is recommended by the manufacturer for seating metallic restorations. This is clinically important for hiding the metallic color in the incisal and proximal areas of anterior dentition. However, opacious material is generally difficult to polymerize with light. The polymerization reaction of Panavia is therefore controlled by a priming agent (ED Primer II) at the interface between the tooth and Panavia. The authors consider that the durable bond strength of the Panavia material in the current study is due to the presence of the redox initiators.

The bonding characteristics of SA Luting appear to be moderate when considering the three luting agents employed. The manufacturer recommends applying Esthetic Cement for seating ceramic restorations, whereas Panavia is recommended for seating metallic restorations. The surfaces to be bonded for collarless PFM restorations consist of the porcelain and the casting alloy. Considering the results of the current study, it may be beneficial to use the Panavia material for seating conventional PFM restorations. However, considering the opacity, bond strength, and other characteristics, the application of SA Luting may be an option for seating collarless PFM restorations.

Acknowledgments

This work is supported in part by Grant-in-Aid for Young Scientists (B 22791906) and Grants-in-Aid for Scientific Research (C 20592283, C 21592474) from the Japan Society for the Promotion of Science (JSPS), Grant from Dental Research Center, and Sato Fund, Nihon University School of Dentistry (2010, 2011).

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Accepted November 28, 2011.

 Online ISSN 2185-3487, Print ISSN 2185-3479