

Influence of dry and wet storage conditions on bond strength to gold alloy of four luting agents

Mika Furuchi, DDS, PhD,^{a,b} Akiko Oshima, DDS,^a Yoshifumi Matsumoto, DDS,^c Michiya Ayano, DDS,^a Tsukasa Masuno, DDS,^a and Hideo Matsumura, DDS, PhD,^{a,b}

^aDepartment of Fixed Prosthodontics, Nihon University School of Dentistry, ^bDivision of Advanced Dental Treatment, Dental Research Center, Nihon University School of Dentistry, and

^cNihon University Graduate School of Dentistry, Tokyo, Japan

Purpose: The purpose of the current study was to evaluate the influence of water storage conditions on bond strength of four luting agents joined to gold alloy.

Materials and Methods: Disk specimens (10 and 8 mm in diameter) were cast from a gold alloy (Degudent U), and the surfaces were ground with abrasive paper. Two resin-modified glass ionomers (RMGIs; Fuji Lute and Ionotite), a resin adhesive (Super-Bond C&B), and a conventional glass ionomer (Ketac Cem) were selected for bonding the gold alloy. A metal priming agent (Metalite) was used together with the Fuji Lute and Super-Bond C&B materials. The paired specimens were bonded with five systems and placed in an incubator at 37°C under three different conditions; 1) immersed in water for 24 h, 2) immersed in water for 24 h followed by additional 7-day-immersion in water, and 3) immersed in water for 24 h followed by dry-state storage for 7 days. Shear bond strengths were then determined and statistically analyzed. Weight change of luting agents after storage was determined using disk-shaped specimens (10 mm in diameter by 2 mm in thickness).

Results Shear bond strength varied from a minimum of 0.1 MPa to a maximum of 40.9 MPa. Bond strength of Fuji Lute and Ketac Cem deteriorated after 7 days for both dry and wet storage conditions, whereas bond strength of Super-Bond C&B without priming improved after dry-storage. Bond strength of Ionotite and Super-Bond C&B with priming remain unchanged during the three storage conditions of the current experiment. Weight change of luting agents after storage was +1.6 to +5.5% for condition #1, +2.6 to +6.0% for condition #2, and -4.5 to -0.4% for condition #3.

Conclusion: Within the limitations of this experiment, it can be concluded that the use of either Ionotite alone or Super-Bond C&B combined with Metalite is recommended for bonding gold alloy.

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Key Words: bonding, gold alloy, primer, resin-modified glass ionomer, thione.

Introduction

The use of resin-modified glass ionomers (RMGIs) and resin-based adhesives for cementing ceramic and metallic restorations has increased substantially. This trend is mainly derived from improvement in bonding characteristics and the increasing application of all-ceramic and collarless metal-ceramic restorations. Seating restorations with RMGI or resin adhesive are one of the options due to improved bond strength,¹⁻⁶ adequate sealing ability,⁷ and cement thickness.⁸

According to the literature, RMGI cements provided better bonding property than conventional glass ionomer cements.¹ Microabraded and tin-plated alloy bonded with a RMGI demonstrated strong bond strength.³ However, bond strengths of zinc-phosphate, glass ionomer, and RMGI cements were inferior to those of resin adhesives under the specific conditions.⁹ In addition, the bond strength of glass ionomer joined to gold alloy was inferior to that of composite luting agent.¹⁰

Both prepolymerized alkenoic acids soluble to water and methacrylic monomers are being incorporated into RMGI compositions. Moreover, several RMGI luting agents do not contain water.⁴ This fact encourages application of adhesive functional monomers in combination with RMGIs for bonding both ceramic and cast metal restorations. Although a number of experimental results have been reported on bonding noble metal alloys with priming agents and adhesive resins,^{2,6,11,12} only limited information is available about the effect of priming agents on bond strength of RMGIs to noble alloys,^{4,5} especially as related to storage conditions. This study

evaluated the influence of dry and wet storage conditions on bond strength of four luting agents joined to gold alloy. Short-term weight change of luting agents was also determined.

Materials and Methods

Table 1 summarizes the information on the materials assessed. A gold alloy designed for porcelain-fused-to-metal technique (Degudent U, DeguDent GmbH, Hanau, Germany) was used as the substrate material. A single-liquid metal priming agent (Metaltite, Tokuyama Dental Corp., Tokyo, Japan), with a functional monomer 6-methacryloyloxyhexyl 2-thiouracil-5-carboxylate (MTU-6), was employed. Two RMGIs (Fuji Lute,¹³ FL, GC Corp., Tokyo, Japan; and Ionotite F, IT, Tokuyama Dental Corp.), a resin adhesive (Super-Bond C&B, SB, Sun Medical Co., Ltd., Moriyama, Japan), and a conventional glass ionomer (Ketac Cem μ , KC, 3M-Espe AG, Seefeld, Germany) were selected as the luting agents.

Table 1. Materials assessed.

Material	Abbreviation	Component	Lot number	Composition
Gold alloy				
Degudent U			10011271	Au 77.3, Pt 9.8, Pd 8.9, Others 4.0 mass%
Metal priming agent				
Metaltite			01403	MTU-6, Ethanol
Luting agents				
Resin-modified glass ionomers (RMGIs)				
Fuji Lute	FL	Powder	0711071	Fluoroaluminosilicate glass*
		Liquid	0711051	Polyalkenoic acid, HEMA, Water, UDMA
Ionotite F	IT	Powder	005067	Calcium aluminosilicate glass, Silica**
		Liquid	005067	HEMA, UDMA, Phosphate, MTU-6, BPO, Amine
Resin adhesive				
Super-Bond C&B	SB	Initiator	RL43	Partially oxidized TBB
		Monomer	RK1	4-META, MMA
		Powder	RF1	PMMA, PMMA-coated titanium oxide
Conventional glass ionomer				
Ketac Cem μ	KC	Powder	290553	Glass, Polyethylene polycarboxylic acid
		Liquid	290553	Water, Tartaric acid

MTU-6, 6-methacryloyloxyhexyl 2-thiouracil-5-carboxylate; HEMA, 2-hydroxyethyl methacrylate; UDMA, Dimethacryloxyethyl 2,2,4- (or 2,4,4-) trimethylhexamethylene diurethane; BPO, Benzoyl peroxide; TBB, Tri-*n*-butylborane; 4-META, 4-methacryloyloxyethyl trimellitate anhydride; MMA: Methyl methacrylate; PMMA: Poly(methyl methacrylate).
*Bouillaguest et al.¹³ 2003, **Amamo et al.⁴ 2004.

Bond strength test

A total of 75 pairs of disks (10 mm and 8 mm in diameter by 2.5 mm in thickness) were cast from the gold alloy. All disks were ground flat with #1,500 silicon carbide abrasive paper and cleaned with acetone. A piece of tape 50 μ m in thickness with a circular hole 5 mm in diameter was positioned on the surface of the larger disk to define the bond area. The 75 specimens were divided into five sets of 15 specimens. The first and second sets were unprimed and luted either with IT or with KC, which were spatulated according to the manufacturers' instructions. The third set was primed with Metaltite, and luted with FL. The fourth set was unprimed and bonded with SB using brush-dip technique. The last set was primed with Metaltite and bonded with SB. Immediately after bonding, a constant load of 5.0 N was applied to the bonded specimens for 30 minutes. All specimens were immersed in 37°C distilled water for 24 hours. This state was defined as storage condition #1, and was immediately evaluated by shear bond test. Five sets of five specimens were subsequently immersed in

water for 7 days (condition #2). The remaining five sets of five specimens were subsequently stored in dry-state with 28% humidity for 7 days (condition #3). After storage in each condition, 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. Equality of variance of the bond strength results was primarily analyzed by Levene test and F test. When the Levene test and F test did not show equality of variances, Dunnett's T3 test was performed with the significance level set at 0.05.

Weight change

Four luting agents were spatulated and poured into plastic molds 10 mm in diameter and 2 mm in thickness. For each luting agent, 15 specimens were prepared. Thirty minutes after preparation, the baseline weight of each disk specimen was determined. The 15 specimens were divided into three groups consisting of five specimens. Specimens were stored at the different three conditions, as described in the previous section. The weight of each specimen was determined with an electronic balance (AL204, Mettler Toledo, Greifensee, Switzerland). Average weight change of five specimens for each luting agent was calculated from the baseline and post-storage weights.

Results

Levene test and F test run on the bond strength results revealed that several groups did not show equality of variance. Dunnett's T3 test was applied therefore to evaluate the influence of storage conditions and luting systems on bond strength.

Table 2. Bond strengths (MPa) of five luting systems and statistical categories.

Storage condition		M+FL	IT	M+SB	SB	KC
37°C water for 24 h	Median	31.1	35.8 a	40.3 a	25.5	8.6
	Mean	31.5 b	32.8 b	40.9	23.9	8.5
	SD	1.4	7.0 c	1.0	3.5 c	1.9
37°C water for 24 h + 37°C water for 7 days	Median	20.9 d	23.9 d	38.8	11.0	2.0
	Mean	21.3 e	26.3	39.6	11.2 e	2.5
	SD	5.9	4.2	2.7	1.7	2.4
37°C water for 24 h + 37°C dry state for 7 days	Median	2.8	28.9	39.4 f	35.6 f	0.1
	Mean	3.4 g	29.1	40.3	35.5	0.1 g
	SD	1.9	1.0	4.2	1.4	0.2

Identical letters in the same horizontal lines indicate that the values are not statistically different (Dunnett's T3, $p > 0.05$). N=5. M+FL, Metaltite + Fuji Lute; IT, Ionotite F; M+SB, Metaltite + Super-Bond C&B; SB, Super-Bond C&B; KC, Ketac Cem.

Tables 2 and 3 show the shear bond strength results as well as statistical categories. Bond strength varied from 0.1 MPa to 40.9 MPa. Ketac Cem glass ionomer resulted in the lowest bond strength among the five systems, and reduction after 7 days was significant (category j). The specimens primed with Metaltite and bonded with Fuji Lute also showed considerable reduction in bond strength after 7 days. Deterioration of this group was more remarkable for the dry-stored specimens. The 24-h bond strength of the Super-Bond C&B was 23.9 MPa for the unprimed specimens. Bond strength of this system deteriorated to 11.2 MPa after storage in water for 7 days, whereas it increased to 35.5 MPa after dry-storage for 7 days. Ionotite generated bond strength ranged from 26.3 MPa to 32.8 MPa. The average values of the luting agent, however, were not significantly different from each other (category h). Within the system of the present experiment, Metaltite combined with

Super-Bond C&B exhibited the greatest bond strength (39.6 to 40.9 MPa) regardless of storage conditions. In addition, bond strength of Super-Bond C&B was not reduced after 7 days and was unaffected by the dry and wet storage conditions (category i).

Table 3. Statistical categories of the storage conditions.

Storage condition		M+FL	IT	M+SB	SB	KC
37°C water for 24 h	Median	31.1	35.8 h	40.3 i	25.5	8.6
37°C water for 24 h + 7 days	Median	20.9	23.9 h	38.8 i	11.0	2.0 j
37°C water for 24 h + 37°C dry state for 7 days	Median	2.8	28.9 h	39.4 i	35.6	0.1 j

Identical letters in the same vertical columns indicate that the values are not statistically different (Dunnett's T3, $p > 0.05$). N=5.

Table 4 summarizes the change of weight of the four luting agents as compared with the baseline set at 30 minutes of specimen preparation. Weight change of luting agents after storage was +1.6 to +5.5% for condition #1 (immersion in water for 24 h), +2.6 to +6.0% for condition #2 (immersion in water for 24 h followed by additional 7-day-immersion in water), and -4.5 to -0.4% for condition #3 (immersion in water for 24 h followed by dry-state storage for 7 days).

Table 4. Weight change (%) of four luting agents after three storage conditions.

		FL	IT	SB	KC
37°C water for 24 h	Median	104.4	103.5	101.6	105.7
	Mean	104.4	103.5	101.6	105.5
	SD	0.4	0.3	0.4	0.3
37°C water for 24 h + 37°C water for 7 days	Median	105.7	105.8	102.5	105.8
	Mean	106.0	105.6	102.6	105.8
	SD	1.2	0.6	0.2	0.3
37°C water for 24 h + Dry state for 7 days	Median	97.4	99.9	99.6	95.4
	Mean	97.4	99.6	99.5	95.5
	SD	0.4	0.7	0.3	0.8

Baseline determination, After 30 minutes of preparation.

Discussion

This study evaluated the effect of specimen storage conditions on bond strength to gold alloy of four luting agents. The effect of MTU-6 functional monomer on bonding to gold alloy was also assessed. The 24-h bond strength results showed that bonding to gold alloy of Super-Bond C&B without bonding agent and Ketac Cem provided no significant advantages. The original Super-Bond C&B material contains a carboxylic monomer for bonding enamel and dentin, and does not contain any functional monomer capable of bonding noble alloys. The Ketac Cem glass ionomer does not contain any polymerizable monomer, hence its adhesive performance is not satisfactory, except for interaction between polyalkenoic acid and tooth structure. The other three systems (Metalite and Fuji Lute, Iontite, and Metalite and Super-Bond C&B) employed MTU-6 as a bonding promoter for the gold alloy.^{2,12} In addition, these three systems contain a considerable amount of co-polymerizable monomers in the liquid component. The authors speculate that the bond strength of three groups (categories a and b) in Table 2 is derived from the interaction between MTU-6 and gold alloy, and from subsequent copolymerization between MTU-6 and monomer component of the luting agents.

The difference in bond strength between Fuji Lute and Iontite was apparent after dry-storage of the

specimens. The former contains water in the liquid component, whereas the latter does not contain water. It is not surprising, therefore if bond strength of the water-based material is affected negatively after dry-storage. However, this result does not discourage the use of water-based luting agents, since luting agents are applied generally to oral environments in the presence of saliva.

Weight change of luting agents clearly demonstrates that water-based materials are susceptible to changes in wet and dry environments. However, this phenomenon is not particularly useful for metal priming systems. These results may promote the combined application of hydrophilic monomer such as 2-hydroxyethyl methacrylate (HEMA) and adhesive functional monomer such as 4-methacryloyloxyethyl trimellitate anhydride (4-META) for bonding dentin. This is due to the fact that dentin is composed of a complex of hydrophilic collagen fibers and inorganic hydroxyapatite.

Within the limitations of the current experiment, it can be concluded that the bond strength to gold alloy of two water-based luting agents (Fuji Lute and Ketac Cem μ) and a resin adhesive without priming (Super-Bond C&B) was negatively affected by dry or wet storage conditions. In particular, the weight loss of two water-based luting agents was remarkable after dry-storage. Consistent bonding to gold alloy was generated with the combined application of MTU-6 thione monomer and two polymerizable luting agents (Ionotite and Super-Bond C&B).

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Correspondence to:

Dr. Mika Furuchi
Department of Fixed Prosthodontics, Nihon University School of Dentistry
1-8-13, Kanda-Surugadai, Chiyoda-ku, Tokyo, 101-8310, Japan
Fax: +81-3-3219-8351 E-mail: nemoto@dent.nihon-u.ac.jp

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