A study of self-adhesive resin cements for bonding to silver-palladium-copper-gold alloy — Effect of including primer components in cement base

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This study investigated the efficacies of adhesive resin cements (Clearfil SA Luting, Maxcem, G-CEM, RelyX Unicem Clicker, Vitremer Paste) for bonding to Ag-Pd-Cu-Au alloy not surface-pretreated with metal primer. For control, Panavia F 2.0—developed for use with a proprietary metal primer, Alloy Primer—was tested with and without metal primer application. Pairs of alloy disks (10.0 and 8.0 mm in diameters, 3.0 mm thickness) were air-abraded with alumina and bonded with one of the cements. Shear bond strengths (SBSs) were measured before and after 50,000 times of thermocycling. Among Maxcem, RelyX Unicem Clicker and the control, there were no statistical differences in SBS before and after thermocycling. After thermocycling, Clearfil SA Luting exhibited the highest SBS among all the cements. Results showed that Clearfil SA Luting, Maxcem, and RelyX Unicem Clicker were efficacious for bonding to Ag-Pd-Cu-Au alloy after air abrasion surface treatment for the latter.

Keywords: Self-adhesive cement, Shear bond strength, Silver-palladium-copper-gold alloy

INTRODUCTION

In the cementation of prosthetic devices, the use of resin cements delivers myriad clinical benefits: (1) reduced incidence of secondary caries and subsequent pulp damage due to improved marginal sealing and low cement solubility¹⁻³; (2) effective prevention of root fractures when used for the cementation of cast post-and-core build-ups^{4,5}; (3) improved fracture resistance, and hence extended longevity, for esthetic ceramic and composite resin prostheses when they are luted with adhesive resin cements⁶⁻⁸. In light of these advantages, the clinical use of resin cements has risen steadily⁹.

For bonding with resin cements, proprietary surface treatments must be applied on the adhesive areas to achieve optimum bonding. Therefore, it has become a routine procedure to perform surface treatments on abutment teeth¹⁰⁻¹² and prostheses¹³⁻¹⁸ prior to cementation with resin cements. However, the efficacy of surface treatments is dependent upon the skills and experience of operators in the handling of specific equipment and in the execution of sensitive manipulations^{19,20}. To eliminate such dependency and hence uncertainty in achieving optimal restorative treatment results, there emerged a natural progression toward the development of resin cements which could provide sufficient bond strength but with a simple surface treatment.

Currently, resin cements can be classified into two groups according to their bonding procedures: luting resin cements *versus* adhesive resin cements. Luting resin cements require a priming process or surface pretreatment (such as tin-plating) of the bonding

substrates. Adhesive resin cements, on the other hand, do away with the priming process because they contain adhesive components in the cement base, such that direct bonding with the substrates is provided by these adhesive monomers. Lately, adhesive resin cements that appeared on the market do not specify the use of primers for the cementation of metal restorations in their operation manuals. These adhesive resin cements were developed by different manufacturers and have been assessed in numerous studies.

The purpose of this study was to investigate the potential of five self-adhesive resin cements containing primer components for the cementation of silver-palladium-copper-gold (Ag-Pd-Cu-Au) alloy restorations. The Ag-Pd-Cu-Au alloy was not surface-pretreated with metal primer application, and thermocycling was used to simulate the aging of dental restorations. Before and after thermocycling, the shear bond strengths (SBSs) of these five adhesive resin cements to the alloy were measured and then compared with those obtained from an adhesive resin cement that required metal primer surface pretreatment.

MATERIALS AND METHODS

Materials

Table 1 presents the materials used in this study and their compositions. An Ag-Pd-Cu-Au alloy (Castwell M.C. 12, GC Corp., Tokyo, Japan) was selected for the adherend. Five adhesive resin cements examined in this study were Clearfil SA Luting (SA; Kuraray Medical Inc., Tokyo, Japan), Maxcem (MA; Kerr Corp., Orange CA, USA), G-CEM (GC; GC Corp.), RelyX Unicem Clicker

(UC; 3M ESPE, St. Paul, MN, USA), and Vitremer Paste (VT; 3M ESPE). According to their manufacturers, these adhesive resin cements were not developed for use with any proprietary metal primer.

Panavia F 2.0 (PV; Kuraray Medical), which required

the application of proprietary metal primer Alloy Primer (AP; Kuraray Medical Inc.), was used as a control. Its results with and without metal primer application were then compared with those of the above adhesive resin cements without metal primer surface treatment.

Table 1 Materials used in this study

Material	Brand Name	Content	Manufacturer	Batch number	Code
Adhesive resin Cement					
	Clearfil SA Luting	Paste A: Bis-GMA, TEGDMA, MDP, other methacrylate monomers, silanated barium glass filler, silanated colloidal silica, dl-camphorquinone, benzoyl peroxide, others Paste B: Bis-GMA, other methacrylate monomers, silanated barium glass filler, silanated colloidal silica, surface treated sodium fluoride, accelerators, pigments, others	Kuraray Medical Inc., Tokyo, Japan	0057AA	SA
	Maxcem	Base: UDMA, dl-camphorquinone, fluoroaluminosilicate glass, others Catalyst: Bis-GMA, TEGDMA, GPDM, barium aluminoborosilicate glass, others	Kerr Corp., Orange, CA, USA	2976773	MA
	G-CEM	Powder: fluoroaluminosilicate glass, initiators, pigments, others Liquid: methacrylate ester, 4-MET, phosphate ester monomer, purified water, silica nano filler, initiators, others	GC Corp., Tokyo, Japan	0803261	GC
	RelyX Unicem Clicker	<i>Unknown in detail:</i> glass powder, methacrylated phosphoric acid esters, TEGDMA, silane treated silica, sodium persulfate	3M ESPE, St Paul, MN, USA	274863	UC
	Vitremer Paste	Paste A: silanated glass, HEMA, purified water, others Paste B: polycarboxylic acid, zirconia-silica, HEMA, purified water, Bis-GMA, sulphates, others	3M ESPE, St Paul, MN, USA	NY8MG	VT
	Panavia F 2.0	Paste A: MDP, methacrylate monomer, filler, photo initiator, chemical initiator Paste B: methacrylate monomer, photo initiator, chemical initiator	Kuraray Medical Inc., Tokyo, Japan	A: 00417A B: 00216A	PV
Metal primer					
	Alloy Primer	VBATDT, MDP, acetone	Kuraray Medical Inc., Tokyo, Japan	00350A	AP
Adherend material					
	Castwell M.C.12	silver (46%), palladium (20%), copper (20%), gold (12%)	GC Corp., Tokyo, Japan		

Bis-GMA : Bis-phenol A diglycidylmethacrylate

TEGDMA : Triethyleneglycol dimethacrylate

MDP : 10-Methacryloyloxydecyl dihydrogen phosphate

UDMA : Urethane Dimethacrylate

GPDM : Glycerophosphoric acid dimethacrylate

4-MET : 4-methacryloxyethyl trimellitic acid

HEMA : 2-hydroxyethylmethacrylate

VBATDT : 6-(4-vinylbenzyl-n-propyl) amino-1, 5-triazine-2, 4-dithiol

Bonding procedure

A total of 98 sets of disks (10.0 mm and 8.0 mm in diameters, 3.0 mm thickness) were cast using Ag-Pd-Cu-Au alloy according to the manufacturer's instruction. Bonding surfaces of the disks were polished with #600 silicon carbide papers, followed by airborne-particle abrasion using 50 μ m alumina particles with a 0.5 MPa pressure for 10 seconds. Distance between the nozzle and the alloy surface was set to 5.0 mm.

To demarcate the bonding area, the 10.0 mm diameter disks were covered with a masking tape with a circular hole 5.0 mm in diameter. The 8.0 mm diameter disks were then cemented onto the demarcated bonding area with one of the five adhesive resin cements without metal primer application. All bonding procedures were carried out according to the manufacturers' instructions, and there were 14 specimens in each group.

For the two control groups, Panavia F 2.0 was applied on the airborne-particle abraded alloy surface with (group APV) and without metal primer application (group AP). Similarly, there were 14 specimens in each control group.

Shear bond strength measurement

At 30 minutes after the bonded specimens were prepared, they were immersed in 37°C distilled water and stored for 24 hours. This state was defined as 0 cycles of thermocycling, and shear bond strengths were measured using half of the specimens in each group ($n=7$). The rest of the specimens in each group ($n=7$) were subjected to 50,000 times of thermocycling (one-minute dwell time each in 4°C and 60°C), and then shear bond strengths were measured (Fig. 1).

Shear bond strength testing was performed using a universal testing machine (AGS-5kNG, Shimadzu Corp.,

Kyoto, Japan) at a 1.0 mm/min crosshead speed. The data were statistically analyzed with two-way ANOVA, and multiple comparisons were performed using Tukey's test at 95% of confidence level.

Failure mode analysis

After shear bond strength testing, the fractured surfaces of all specimens with and without thermocycling were analyzed using an optical microscope (SMZ-10, Nikon Corp., Tokyo, Japan) under $\times 20$ magnification. Failure modes were classified as adhesive failure (A), cohesive failure (C), or mixed failure (M).

Failure mode classification was carried out by examining the fracture surfaces of both 10 mm diameter and 8 mm diameter disks. Adhesive failure was thus determined when debonding occurred at the interface between cement and either disk surface. Cohesive failure was when the fracture occurred within the cement layer. Mixed failure was when the specimen exhibited a combination of adhesive and cohesive failures.

RESULTS

Shear bond strength

Figure 2 presents the SBS results of respective adhesive resin cements to the Ag-Pd-Cu-Au alloy and their statistical analyses. Two-way ANOVA showed that there were significant differences in SBS among the cements ($p<0.0001$) as well as between the thermocycled and non-thermocycled groups ($p<0.0001$). In addition, a statistically significant interaction ($p<0.0001$) was found

Fig. 1 Cross-sectional illustration of shear bond strength test.

Fig. 2 Shear bond strengths of adhesive resin cements to Ag-Pd-Cu-Au alloy.

□ Without thermocycling
 ▨ After 50,000 thermocycles

Bars connected with brackets marked with * were not significantly different ($p>0.05$).

Values with the same superscript letters were not significantly different ($p>0.05$) within the same thermocycling state, i.e., before or after 50,000 times of thermocycling.

Table 2 Failure modes of each group

	Before thermocycling			After 50,000 thermocycles		
	Adhesive failure	Cohesive failure	Mixed failure	Adhesive failure	Cohesive failure	Mixed failure
SA	4	0	3	4	0	3
MA	1	0	6	5	0	2
GC	2	0	5	7	0	0
UC	2	0	5	3	0	4
VT	1	0	6	7	0	0
PV	4	0	3	4	0	3
APV	4	0	3	4	0	3

between the factors of cement and the thermocycling regime.

Before thermocycling, the SBSs of SA, MA, and UC were not significantly different from those of control groups PV and APV ($p < 0.0001$). As for the SBSs of GC and VT, they were significantly lower than the control groups PV and APV ($p < 0.0001$).

After thermocycling, all other groups —except SA and GC— exhibited significantly lower SBS values ($p < 0.0001$). Among all the cements, SA exhibited the highest SBS at 50.5 ± 5.8 MPa ($p < 0.0001$). The SBSs of MA and UC were not significantly different from those of control groups PV and APV (MA: $p = 0.9964$, UC: $p > 0.999$), but the SBSs of GC and VT were significantly lower than the control groups ($p < 0.0001$).

Within each adhesive resin cement group, SA and GC showed no significant decrease in SBS after thermocycling (SA: $p = 0.6902$, GC: $p = 0.9716$). Between the control groups PV and APV, there were no significant differences between their SBS values before and after thermocycling.

Failure modes

Table 2 presents the failure mode results of all the specimens in this study. After shear bond strength testing, all the specimens exhibited either adhesive or mixed failure —be it with or without thermocycling. After thermocycling, MA, GC, UC, and VT showed an increase in adhesive failure incidences whereas SA, PV, and APV showed no such increase.

DISCUSSION

Except for PV, the adhesive resin cements evaluated in the current study contained adhesion-promoting monomers for dental metal alloys (Table 1), hence eliminating the need for metal primer application. These adhesive resin cements were introduced to simplify the bonding procedure as well as eliminate unexpected, undesirable clinical incidents. The priming process requires auxiliary materials and additional manipulations, such that patients have heightened risks of accidental ingestion of the primer or aspiration of organic solvents contained in the primer.

Thermocycling was employed in the current study to evaluate bonding durability, chiefly because the mechanical properties and adhesive strengths of resin cements are affected by hydrolysis and thermal expansion/contraction which occurred during thermocycling²¹. SA exhibited superior bonding durability, with no statistically significant difference in SBS before and after thermocycling and yielding the highest SBS among all the cements after thermocycling. Two reasons probably accounted for this result: inclusion of adhesive monomer MDP and an improved polymerization initiation system²².

It has been reported that the MDP monomer demonstrated excellent bonding to non-precious alloys^{23–25}. Ag-Pd-Cu-Au alloy employed in this study contained 46% Ag, 20% Pd, 20% Cu, and 12% Au (Table 1). It was highly probable that the MDP monomer chemically reacted with Cu and hence resulted in the superior bonding durability of SA against thermocycling. Interestingly, PV also contained the MDP monomer but exhibited inferior bonding durability when compared to SA. This disparity might stem from a difference in their polymerization initiation systems. Therefore, in conjunction with the MDP monomer, the improved polymerization system of SA resulted in superior mechanical properties and consequently resulted in improved bonding durability.

MA and UC contained phosphoric functional monomers which render the same effect as MDP^{26–29}. As a result, there were no significant differences in SBS between MA and UC both before and after thermocycling. After thermocycling, the adhesive failure rate of MA increased but not so for UC. However, both MA and UC exhibited significantly lower SBS values after thermocycling. Based on the SBS and failure mode results, the SBS of MA became lower probably because of reduced adhesive strength caused by the thermocycling process; similarly, deterioration in the mechanical properties of UC caused by thermocycling resulted in its significantly lower SBS.

GC contained 4-MET as a functional monomer which ionically interacts with enamel and dentin^{30–32}, whereas HEMA contained in VT chemically interacts with dentin^{33,34}. As HEMA is a hydrophilic monomer, it

might induce hydrolysis. Compared to other resin cements, GC and VT exhibited inferior SBS both before and after thermocycling. After thermocycling, all the specimens of GC and VT exhibited adhesive failure. It is therefore reasonable to conclude that 4-MET and HEMA offered unsatisfactory bonding to Ag-Pd-Cu-Au alloy.

In the present study, the precious metal content of the tested alloy was quite high. Therefore, the availability of sulfur-containing monomers such as VBATDT and MTU-6 (6-methacryloyloxyhexyl-2thiouracil-5-carboxylate) that promote adhesion to precious alloys³⁵⁻³⁸⁾ could have improved the bond strength and bonding durability of cements. However, sulfur-containing monomers are not suitable for resin cements that polymerize by BPO-amine redox system as they disturb the smooth progress of the radical polymerization initiated by BPO-amine reaction, consequently causing the resin cements to have weakened mechanical properties³⁹⁻⁴¹⁾. The Alloy Primer of Panavia F 2.0 contained VBATDT, but there were no statistically significant differences in SBS between APV and PV both before and after thermocycling. It was reasonable to suggest that when the resin cement came into contact with the VBATDT monomer on the primed alloy surface, the sulfur-containing VBATDT monomer inhibited the polymerization reaction, thus causing APV to have weakened mechanical properties and hence its SBS was adversely affected.

For bonding systems that require preliminary application of adhesive primers, it is not necessary to include adhesive monomers in their cement base formulations. From the perspective of long-term stability of cement paste, these systems are advantageous because no adhesive monomers are present in the cement base to interfere with the function of the polymerization initiator. However, herein emerges a new possibility of adhesive monomers in the primer interfering unfavorably with the polymerization initiators in the cement, thereby inhibiting the polymerization reaction. In such cases, it is recommended to add a polymerization accelerator to the primer or apply an intermediate resin layer on the primed surface.

In the present study, none of the specimens exhibited cohesive failure. A few explanations are proposed for this phenomenon. First, mechanical strength at the alloy-cement interface was inferior to the cohesive strength of resin cement itself; alternatively, there were pockets of area within the interface which possessed inferior bonding capacity compared to the cohesive strength of resin cement. For most resin cements in this study—except SA, PV, and APV, their incidences of adhesive failure increased after thermocycling. But for SA, PV, and APV which contained MDP as an adhesive monomer, their failure distribution modes before and after thermocycling remained the same. In the case of SA, this could be attributed to its superior bonding durability which then shielded its SBS from severe deterioration.

It was reported in numerous studies that cohesive failure was the predominant failure mode for adhesive

resin cements bonded to oxidized surfaces of Ag-Pd-Cu-Au alloys before thermocycling⁴²⁾. When Ag-Pd-Cu-Au alloy restorations were cemented with adhesive resin cements, deterioration occurred at the bonding interface rather than within the cement material itself^{43,44)}. Therefore in this study, although SA did not show a significant decrease in SBS after thermocycling, it was probable that the cohesive strength of the cement adjacent to oxidized alloy surface might be greater than the bond strength of SA. This suggestion could find its support and endorsement in clinical studies^{45,46)} which reported that the incidences of adhesive failure at the alloy-cement interface increased with prolonged service period of Ag-Pd-Cu-Au alloy restorations cemented with adhesive resin cements.

To compensate for future decrease in bond strength for Ag-Pd-Cu-Au alloy restorations cemented with adhesive resin cements, the cement adjacent to the alloy-cement interface must have sufficiently high cohesive strength immediately after cementation. This meant that for all the adhesive resin cements tested in this study, their applicability for bonding restorations that require long-term bonding durability (such as fixed partial dentures) would need further investigation, since their cohesive strengths were not investigated in this study. Nonetheless, SA demonstrated durable bonding to Ag-Pd-Cu-Au alloy after 50,000 times of thermocycling. Thus, SA seemed to be applicable for conventional restorations including inlays/onlays or crowns of which their retention is enhanced by mechanical interlocking.

CONCLUSIONS

Within the limitations of the current study, the following conclusions were drawn based on the obtained results:

1. Between the primed and non-primed control groups of Panavia F 2.0, there were no statistically significant differences in SBS before and after thermocycling.
2. Before thermocycling, there were no statistically significant differences in SBS among Clearfil SA Luting, Maxcem, RelyX Unicem Clicker, and the Panavia F 2.0 control groups.
3. After thermocycling, there were no statistically significant differences in SBS among Maxcem, RelyX Unicem Clicker, and the Panavia F 2.0 control groups.
4. Among all the adhesive resin cements, Clearfil SA Luting exhibited the highest SBS (50.5 ± 5.8 MPa) to the airborne particle-abraded alloy after thermocycling.
5. With or without thermocycling, all the cements tested exhibited either adhesive or mixed failure after shear bond strength testing. For Clearfil SA Luting, Panavia F 2.0, and Panavia F 2.0 with Alloy Primer, their failure distribution modes did not change after thermocycling.
6. G-Cem and Vitremer Paste could not achieve sufficient bonding durability with Ag-Pd-Cu-Au alloy.

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