Effect of adhesion promoting monomer addition to MMA-TBBO resin on bonding to pure palladium

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This study evaluated the effects of combined use of metal primers and modified monomers on the bonding of MMA-TBBO resins to pure palladium (Pd). Bonding surface was polished with 600-grit silicon carbide paper and primed with one of these four metal primers: V-Primer, M.L. Primer, Metaltite, or Alloy Primer. Four monomers, including three modified ones, were added to MMA-TBBO resin. One was a methyl methacrylate monomer containing no adhesion promoting monomers, while the other two modified monomers contained the functional monomer of either V-Primer or Alloy Primer. Bonded specimens were prepared by incremental build-up of MMA-TBBO resin on primed Pd surfaces. Shear bond strengths were measured after thermal cycling. Bonding to Pd was significantly improved when modified monomer containing the functional monomer of Alloy Primer was used in combination with M.L. Primer or Metaltite applied on the bonding surface.

Keywords: MMA-TBBO resin, Adhesion promoting monomer, Pure palladium, Modified monomer, Shear bond strength

INTRODUCTION

Resin-bonded fixed partial dentures with cast metal frameworks have emerged as a minimally invasive, aesthetically pleasing, and predictable treatment option for abutment teeth¹⁻³. Two key factors contribute to this emerging trend: improvement in retainer design⁴⁻⁶ and availability of metal primers which react directly and chemically with the bonding surfaces of noble metal alloys, such as silver-containing gold-palladium alloys^{7,8} and type IV gold alloy⁸⁻¹⁰.

Noble metal alloys are not suitable for cases that require high-quality aesthetic appearance. To satisfy both aesthetic and functional demands, the optimal alternative is resin-bonded fixed partial dentures fabricated with metal alloy frameworks and porcelain-faced pontics. Traditionally, nickel-chromium¹¹⁻¹³ and cobalt-chromium^{12,14} alloys are used for the retainers. This is largely because of well-established, durable bonding of adhesive resin cements to nickel-chromium^{15,16} and cobalt-chromium^{17,18} alloys. However, noble metal ceramic alloys are preferable and superior in terms of castability, workability, corrosion resistance, and biocompatibility^{19,20}.

Macro-mechanical retention and micro-mechanical retention are two critical factors that influence the clinical success and longevity of fixed partial dentures. Macro-mechanical retention depends on abutment preparation and precision of fit of retainers. Micro-mechanical retention is achieved by chemical bonding, which in turn can be achieved with surface modification of metal substrates or application of metal primers⁷⁻¹⁰.

On the former, currently available surface treatment methods range from tin (Sn) plating^{13,21,22} to thermal silica coating (Silicoater MD)²² and tribochemical silica coating (Rocatec)²³. On the latter, adhesion promoting monomers play an important role in improving bonding, which is especially vital for restorations with compromised retention form.

For base metals, it was shown that the presence of both TBBO initiator and 4-META functional monomer at the resin-metal interface during the setting reaction improved the bonding to cobalt-chromium alloy¹⁸. For noble metals, inclusion of thiirane monomers as an adhesive monomer component of MMA-TBBO resin improved the bonding to dental precious metal alloys²⁴. Studies^{25,26} have also shown the positive effect of metal primers on the bonding of 4-META/MMA-TBBO resin to high-gold-content metal ceramic alloys. At the same time, these studies^{25,26} revealed the inadequacy of these primers on bonding to pure palladium (Pd) and high-palladium-content metal ceramic alloys. Although some studies⁷⁻⁹ have reported on the efficacy of metal primers in improving the bond strength to Pd-containing alloys, it must be highlighted that these alloys contained other metals such as Ag and Cu, which are compatible with numerous primers²⁵.

The purpose of this study was to evaluate the effect of adhesion promoting monomers on bonding of MMA-TBBO resin to Pd. Although pure Pd is not used clinically, it was employed in this study to investigate how to improve the bonding of adhesive resins to high-palladium-content alloys. Shear bond strengths of MMA-TBBO resins containing different adhesion promoting

monomers to Pd were evaluated after thermal cycling. The null hypothesis was that the presence of adhesion promoting monomer derived from commercial metal primers would have no effect on the bonding of MMA-TBBO resin to Pd.

MATERIALS AND METHODS

Metal primers and Pd adherends

Table 1 lists the materials used in this study. Figure 1, on the other hand, is a flow diagram which illustrates the sequence of steps entailed in preparing the bonded specimens for shear bond strength test, using the materials listed in Table 1.

Pd disks (10×2.5 mm) of >99.9% purity were provided by a manufacturer (Kojundo Chemical Laboratory Co., Ltd., Saitama, Japan) and used as the adherends. Bonding surface of each Pd adherend was polished using 600-grit silicon carbide paper (CarbiMet Paper Discs, Buehler, Lake Bluff, IL, USA) with ample water coolant. After ultrasonic cleaning in distilled water for 10 min, Pd adherends were dried with filtered air.

Modified monomers

Table 2 lists the four kinds of monomers added to MMA-TBBO resin: proprietary Super-Bond monomer (Sun Medical Co., Ltd., Shiga, Japan), a methyl methacrylate monomer (MMA; Wako Pure Chemical Industries, Ltd.,

Table 1 Materials used in this study

Material	Brand name	Composition	Manufacturer	Batch number
<i>Adherend material</i>				
Pure palladium	Pure Pd	Pd (>99.9% purity)	Kojundo Chemical Laboratory Co., Ltd., Saitama, Japan	111245
<i>Metal Primers</i>				
Single-liquid primer	Alloy Primer	VBATDT, MDP, Acetone	Kuraray Noritake Dental Inc., Tokyo, Japan	0371AA
Single-liquid primer	Metaltite	MTU-6, Ethanol	Tokuyama Dental Corp., Tokyo, Japan	052080
Single-liquid primer	M.L. Primer	MDDT, 6-MHPA, Acetone	Shofu Inc., Kyoto, Japan	091048
Single-liquid primer	V-Primer	VBATDT, Acetone	Sun Medical Co., Ltd., Shiga, Japan	VM1

VBATDT: 6-(4-vinylbenzyl-n-propyl)amino-1,3,5-triazine-2,4-dithiol

MDP: 10-methacryloyloxydecyl dihydrogen phosphate

MTU-6: 6-methacryloyloxyhexyl 2-thiouracil-5-carboxylate

MDDT: 10-methacryloyloxydecyl-6,8-dithiooctanoate

6-MHPA: 6-methacryloyloxyhexyl phosphonoacetate

Priming for bonding surface

Polishing of Pd adherend with #600 silicon carbide paper

Application of 1 out of 5 metal primers onto polished Pd surface

1. Non-primed 2. V-Primer 3. M.L. Primer 4. Metaltite 5. Alloy Primer

Mixing of MMA-TBBO resin

Building-up MMA-TBBO resins onto primed Pd surface

Fig. 1 Flow diagram of preparing bonded specimens for shear bond strength test.

Osaka, Japan) which did not contain any adhesion promoting monomers, and two kinds of modified monomers derived from commercial metal primers.

To prepare the two modified monomers, a single drop of V-Primer (Sun Medical Co., Ltd., Shiga, Japan) or Alloy Primer (Kuraray Noritake Dental Inc., Tokyo, Japan) was placed in a ceramic mixing dish. The solvent (acetone) was completely evaporated by mild blowing of filtered air. Finally, four drops of MMA monomer were added to complete the preparation process of these two modified monomers (Fig. 1). Figure 2 shows the chemical structures of the adhesive monomers employed in the present study.

Priming and bonding procedures

Each Pd bonding surface was primed using one of the four commercial metal primers (Table 1). After a single drop of primer was applied on the Pd surface, it was dried with filtered air for 5 s. Non-primed Pd adherends were used as controls (Fig. 1).

A 30- μ m-thick tape (Mending Tape, Kokuyo Co., Ltd., Osaka, Japan) with a 5-mm-diameter opening was placed on the primed/non-primed bonding surface to define the bonding area. Bonding area was covered with a brass ring of 6 mm inner diameter and 2.5 mm height.

A single drop of TBBO polymerization catalyst was added to each monomer (Fig. 1). Each MMA-TBBO resin

Table 2 MMA-TBBO resins used in this study

Material	Brand name	Composition	Manufacturer	Batch number
Polymer	Super-Bond polymer	PMMA	Sun Medical Co., Ltd., Shiga, Japan	EF22
	Super-bond monomer	MMA, 4-META	Sun Medical Co., Ltd., Shiga, Japan	EF3
	Methyl Methacrylate Monomer	MMA	Wako Pure Chemical Industries, Ltd., Osaka, Japan	EPK3235
Monomer	Alloy Primer+MMA monomer	VBATDT, MDP, MMA	—	—
	V-Primer+MMA monomer	VBATDT, MMA	—	—
Catalyst	Super-Bond catalyst	TBBO, Acetone	Sun Medical Co., Ltd., Shiga, Japan	EE1F

PMMA: polymethyl methacrylate

MMA: methyl methacrylate

4-META: 4-methacryloyloxyethyl trimellitate anhydride

TBBO: tri-*n*-butylborane oxide

Fig. 2 Chemical structures of adhesive monomers employed in this study.

was built up in small increments in the brass ring using a brush-dip technique (Fig. 3). A total of 100 bonded specimens were prepared for 20 groups, stemming from five types of primed/non-primed Pd adherends and four kinds of monomers added to MMA-TBBO resin. All specimens were kept at room temperature ($22\pm 2^\circ\text{C}$) for 60 min, followed by storage in 37°C distilled water for 24 h.

Shear bond strength test

After 24-h water storage, specimens were subjected to 2,000 times of thermal cycling (Rika-Kogyo, Hachioji, Japan) by immersing in $5\pm 1^\circ\text{C}$ and $55\pm 1^\circ\text{C}$ water baths with 1-min dwell time at each temperature.

Shear bond strength (SBS) test was performed using a universal testing machine (AGS-5kNG, Shimadzu Corp., Kyoto, Japan) at 1.0 mm/min crosshead speed until failure occurred. Figure 4 shows the cross-sectional view of the SBS test setup. A chisel-like, stainless steel

apparatus with a 0.5-mm-wide blunt edge was used to apply the shear force flush against the masking tape and parallel to the bonding interface between the Pd adherend and MMA-TBBO resin. SBS (MPa) was obtained by dividing the maximum load (N) by the bonding area (mm^2).

Statistical analysis

Data were statistically analyzed using two-way analysis of variance (ANOVA) with the primer type and the kind of monomer added to MMA-TBBO resin as independent factors ($\alpha=0.05$). Multiple comparisons were carried out using the Bonferroni-Dunn test to identify the significant differences among the groups ($\alpha=0.05$).

Failure analysis

Debonded surface of each specimen was analyzed using an optical microscope (SMZ-10, Nikon Corp., Tokyo, Japan) at $\times 10$ magnification. Failure modes were classified as cohesive failure of MMA-TBBO resin, adhesive failure at adherend-resin interface, or mixed failure consisting of both cohesive and adhesive failures.

RESULTS

Figure 5 shows the mean SBSs of 20 groups evaluated in this study. Tables 3 and 4 show the statistical analysis results for SBSs among monomers and among metal primers respectively. Two-way ANOVA showed that there were significant differences among monomers ($p<0.0001$) and among primers ($p<0.0001$). There was also a significant interaction between primers and monomers ($p<0.0001$) for SBS. One-way ANOVA results for monomers were $df=3$, $F=17.0$, $p<0.0001$, Power=1.0;

Fig. 3 Bonded specimen for shear bond strength test, where a: pure Pd adherend; b: masking tape; c: brass ring; and d: MMA-TBBO resin.

Fig. 4 Cross-sectional view of shear bond strength test setup.

Fig. 5 Shear bond strengths to pure Pd adherends after 2,000 thermal cycles as function of monomer and metal primer types. Groups with asterisk (*) debonded during thermal cycling.

- Super-Bond monomer
- ▨ MMA monomer
- ▩ Alloy Primer+MMA monomer
- ▤ V-Primer+MMA monomer

Table 3 Statistical analysis results for shear bond strengths (MPa) among monomers within each metal primer group

Priming for Pd surface	Monomers for MMA-TBBO resin	Mean (Standard Deviation)	Difference within the same primer groups
Non-primed	Super-Bond monomer	0.0 (0.0)	A
	MMA	0.0 (0.0)	A
	Alloy Primer+MMA	11.8 (2.0)	B
	V-Primer+MMA	5.0 (1.8)	C
V-Primer	Super-Bond monomer	3.3 (0.6)	D
	MMA	3.8 (0.9)	D
	Alloy Primer+MMA	3.9 (0.8)	D
	V-Primer+MMA	1.9 (0.9)	D
M.L. Primer	Super-Bond monomer	7.2 (1.0)	E
	MMA	7.7 (1.1)	E
	Alloy Primer+MMA	16.3 (2.0)	F
	V-Primer+MMA	6.9 (2.0)	E
Metaltite	Super-Bond monomer	8.1 (0.9)	G
	MMA	8.6 (0.9)	G
	Alloy Primer+MMA	15.1 (2.3)	H
	V-Primer+MMA	7.3 (2.2)	G
Alloy Primer	Super-Bond monomer	3.9 (0.8)	I, J
	MMA	5.3 (1.0)	I, J
	Alloy Primer+MMA	6.8 (1.4)	J
	V-Primer+MMA	3.1 (1.2)	I

The same uppercase letter denotes no significant difference among monomers within the same primer group.

Table 4 Statistical analysis results for shear bond strengths (MPa) among metal primers within each monomer group

Priming for Pd surface	Monomer for MMA-TBBO resin	Mean (Standard Deviation)	Difference within the same monomer groups
Non-primed	Super-Bond monomer	0.0 (0.0)	a
V-Primer		3.3 (0.6)	a, b
M.L. Primer		7.2 (1.0)	c, d
Metaltite		8.1 (0.9)	d
Alloy Primer		3.9 (0.8)	b, c
Non-primed	MMA	0.0 (0.0)	e
V-Primer		3.8 (0.9)	f
M.L. Primer		7.7 (1.1)	g
Metaltite		8.6 (0.9)	g
Alloy Primer		5.3 (1.0)	f, g
Non-primed	Alloy Primer+MMA	11.8 (2.0)	h
V-Primer		3.9 (0.8)	i
M.L. Primer		16.3 (2.0)	j
Metaltite		15.1 (2.3)	j
Alloy Primer		6.8 (1.4)	i
Non-primed	V-Primer+MMA	5.0 (1.8)	k, l
V-Primer		1.9 (0.9)	k
M.L. Primer		6.9 (2.0)	l
Metaltite		7.3 (2.2)	l
Alloy Primer		3.1 (1.2)	k

The same lowercase letter denotes no significant difference among primers within the same monomer group.

for primers, the results were $df=4$, $F=15.7$, $p<0.0001$, Power=1.0.

Statistical analysis for SBSs among monomers within each primer group

In the non-primed group, the specimens debonded during thermal cycling when Super-Bond monomer and MMA monomer were employed. In comparison, Alloy Primer+MMA monomer and V-Primer+MMA monomer yielded significantly higher ($p<0.0001$) SBSs at 11.8 and 5 MPa respectively (Fig. 5 and Table 3).

In V-Primer group, SBS ranged between 2 and 4 MPa. There were no significant differences among the four monomers ($p>0.05$) (Fig. 5 and Table 3).

In Alloy Primer group, there were no significant differences between Super-Bond and MMA monomers ($p>0.05$). However, difference between Alloy Primer+MMA and V-Primer+MMA monomers was statistically significant ($p=0.0001$) (Fig. 5 and Table 3).

M.L. Primer and Metaltite groups showed similar results (Fig. 5 and Table 3). Highest SBS values (M.L. Primer: 16.3 MPa; Metaltite: 15.1 MPa) were obtained by using Alloy Primer+MMA monomer, which were significantly greater than the other three monomers ($p<0.0001$). There were no significant differences among the remaining three monomers for both primers ($p>0.05$).

Statistical analysis for SBSs among primers within each monomer group

In Super-Bond monomer group, application of M.L. Primer, Metaltite, and Alloy Primer yielded significantly higher SBSs than the non-primed specimens ($p<0.0001$) (Fig. 5 and Table 4).

In MMA monomer group, all the primed specimens showed significantly higher SBSs than the non-primed specimens ($p<0.0001$) (Fig. 5 and Table 4).

In V-Primer+MMA monomer group, there were no statistically significant differences between primed and non-primed specimens ($p>0.05$). Application of M.L. Primer and Metaltite yielded significantly higher SBSs than V-Primer- and Alloy Primer-treated specimens ($p<0.0001$) (Fig. 5 and Table 4).

In Alloy Primer+MMA monomer group, SBSs of M.L. Primer-treated specimens (16.3 MPa) and Metaltite-treated specimens (15.1 MPa) were significantly greater compared to the non-primed specimens at 11.8 MPa ($p<0.0001$). In contrast, SBSs of V-Primer- and Alloy Primer-treated specimens were significantly lower than the non-primed specimens ($p<0.0001$). There were no significant differences between M.L. Primer- and Metaltite-treated specimens and between V-Primer- and Alloy Primer-treated specimens ($p>0.05$) (Fig. 5 and Table 4).

Debonding failure

Both Non-primed/Super-Bond monomer and Non-primed/MMA monomer specimens which debonded during thermal cycling showed adhesive failure. All other specimens exhibited mixed failure consisting

Fig. 6 Typical view of specimen showing mixed failure, where a: Pure Pd bonding surface showing adhesive failure; b: MMA-TBBO resin remaining on Pd adherend; and c: Masking tape.

of both adhesive failure and cohesive failure of MMA-TBBO resin, whereby a thin layer of MMA-TBBO resin partially remained on the pure Pd adherend surface (Fig. 6).

DISCUSSION

The present study evaluated the effect of combined use of metal primers and modified monomers on the bonding of MMA-TBBO resins to pure Pd. This study served as a preliminary investigative study on improving the bonding of MMA-TBBO resins to high-palladium-content alloys. Therefore, the surfaces of pure Pd adherends were finished into regular flat surfaces with 600-grit silicon carbide papers to exclude the influence of mechanical retention, focusing only on the efficacy of each adhesion promoting monomer. In addition, a relatively small number of thermal cycles was employed so that results could be obtained in a short time period.

Results of the present study partially supported the null hypothesis. On the one hand, addition of 4-META to MMA monomer seemed to have no significant effect on bonding to Pd since both Super-Bond monomer and MMA monomer yielded almost similar SBS results within each metal primer group (Table 3). On the other hand, contradictions were observed in the SBS values of MMA-TBBO resins added with the adhesion promoting monomers of Alloy Primer. These SBS values ranked the highest among the other monomers when the bonding surface was primed with M.L. Primer or Metaltite (Fig. 5).

Adhesion promoting monomers are typically the primary component of surface treatment agents used to improve interfacial bonding to different kinds of metals. In adhesive resin formulations, they are also key constituents that play a dominant role. For example,

the monomer liquid of Super-Bond C&B adhesive resin cement contained 4-META, which was used as a control in the current study. Self-adhesive resins are increasingly being used in clinical practice, and they also contain adhesive monomers in their formulations. When an adhesion promoting monomer is included as an active ingredient in an adhesive resin formulation, a part of it is adsorbed on the adherend surface as well as the applied metal primer, and the remnant is copolymerized with the MMA monomer²⁷⁾.

Efficacy of V-Primer+MMA monomer

For the non-primed Pd surfaces, the use of V-Primer+MMA monomer significantly increased the SBS value compared to Super-Bond monomer and MMA monomer (Table 3). However, this value attained (5.0 MPa) was comparable to the performance of V-Primer+MMA monomer when used in combination with other metal primers (Table 4). Within V-Primer+MMA monomer group, SBSs attained with M.L. Primer- and Metaltite-treated surfaces were significantly higher than those treated with V-Primer and Alloy Primer. Both V-Primer and Alloy Primer contained VBATDT. These results implied that an excess amount of VBATDT might have an adverse effect on bonding to Pd.

An adhesion promoting monomer bears two different types of adhesion promoting groups in its molecular design. The tautomer type utilizes tautomerism—where a stable structure with the thione type (-NH-CS-) is tautomerized to the reactive tautomer with a thiol type (-N=C(SH)-) on a precious metal surface²⁸⁾. Sulfur-containing monomers, such as triazinedithione (VBATDT, Fig. 2), belong to the tautomer type²⁸⁾. Within the V-Primer+MMA monomer group, the decreased SBSs of V-Primer- and Alloy Primer-treated specimens was probably due to the thiol compound inhibiting the polymerization of MMA²⁴⁾.

Efficacy of Alloy Primer+MMA monomer

When Alloy Primer+MMA monomer was added to MMA-TBBO resin, the SBSs attained with both primed and non-primed Pd surfaces were significantly higher than those attained with V-Primer+MMA monomer, except for specimens applied with V-Primer (Table 4).

Alloy Primer contained both VBATDT and MDP. This seemed to indicate that MDP played a major role in bonding to Pd. In a previous study²⁵⁾, it was shown that diverse metal primers containing different kinds of adhesion promoting monomers revealed affinity to different types of pure metals. Nonetheless, none of the primers, including M.L. Primer and Metaltite, was effective in bonding to pure Pd²⁵⁾. Interestingly in this study, when Alloy Primer+MMA monomer was used in combination with M.L. Primer or Metaltite, the highest SBS values were obtained (Table 4), suggesting a significant bonding effectiveness to Pd. Therefore, the efficacy of adhesion promoting monomers contained in metal primers could be improved by being compatible with the adhesion promoting monomer(s) added to MMA-TBBO resins.

Within the Alloy Primer+MMA monomer group, comparison among the different types of primed surfaces revealed that the SBS of Alloy Primer-treated group was significantly lower than the non-primed group. This result could be caused by a twofold reason: the inhibitory effect of excessive amount of VBATDT on polymerization²⁴⁾ and the negative effect of MDP²⁹⁾ when both functional monomers coexisted.

Limitations of present study

It must be put into perspective that the limitations of the present study's experimental design could have affected the efficacy of adhesion promoting monomers in promoting the bonding of MMA-TBBO resin to pure Pd.

First, a thin layer of adhesive resin cement was not achieved in this study as MMA-TBBO resin was filled into the brass ring placed on the Pd adherend. Therefore, further fatigue testing investigations based on reduced cement thicknesses are needed.

MDP monomer has a high affinity for ceramic materials³⁰⁻³²⁾. This meant that a certain amount of MDP might have reacted with the ceramic mixing dish. Besides, MDP monomer could have vaporized alongside the evaporation of organic solvent. Together, these phenomena most probably reduced the actual amount of MDP in the modified monomer since the preparation process entailed the evaporation of acetone. Therefore, to evaluate the efficacy of MDP monomer in bonding to pure Pd, it might be expedient to use pure MDP solution in MMA monomer to prepare the modified monomer. This approach would also help to determine the optimal concentration of MDP to be used in clinically applicable bonding procedure.

CONCLUSIONS

This *in vitro* study evaluated the efficacy of adding modified monomers to MMA-TBBO resin on bonding to pure Pd. Within the limitations of this study, it was concluded that bonding to pure Pd was significantly improved when modified monomer containing the functional monomers of Alloy Primer was used in combination with the application of M.L. Primer or Metaltite on pure Pd surface.

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