Effect of metal primers on bond strength of resin cements to base metals

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Statement of problem. A strong and durable bond between a metal framework and a resin-based luting agent is desired. Metal primers have been shown to be very effective on noble alloys. However, there is insufficient information about their effect on base metals.

Purpose. The purpose of this study was to evaluate the effect of metal primers on the shear bond strength of resin cements to base metals.

Material and methods. A total of 160 cast commercially pure titanium (CP Ti) and NiCr alloy (VeraBond II) disks were embedded in a polyvinyl chloride ring, and their surfaces were smoothed with silicon carbide papers (320, 400, and 600 grit) and airborne-particle abraded with 50-μm aluminum oxide. Specimens of each metal were divided into 4 groups (n=20), which received one of the following luting techniques: (1) Panavia F, (2) Alloy Primer plus Panavia F, (3) Bistite II DC, or (4) Metaltite plus Bistite II DC. Forty minutes after preparation, all specimens were stored in distilled water at 37°C for 24 hours and then thermal cycled (1000 cycles, 5-55°C). After thermal cycling, the specimens were stored in 37°C distilled water for an additional 24 hours or 6 months before being tested in shear mode. Data (MPa) were analyzed using 3-way ANOVA and the post hoc Tukey test (α=.05). Each specimen was examined under an optical microscope (×30), and the failure mode was classified as adhesive, cohesive, or a combination of these.

Results. The only significant difference between the Panavia F and Alloy Primer plus Panavia F groups occurred in the NiCr alloy at 24 hours, at which point Panavia F demonstrated superior bond strength compared to Alloy Primer plus Panavia F ($P<.001$). The Bistite II DC and Metaltite plus Bistite II DC groups were not significantly different. The Bistite II DC and Metaltite plus Bistite II DC groups demonstrated significantly lower bond strength to CP Ti ($P<.001$) than the Panavia F and Alloy Primer plus Panavia F groups, and significantly lower bond strength to NiCr alloy ($P<.001$) than Panavia F. The Panavia F ($P<.01$) and Alloy Primer plus Panavia F groups' bond strength to titanium presented a significant increase ($P<.001$) in shear bond strength at 6 months. In general, the groups exhibited higher shear bond strength to CP Ti than to NiCr alloy ($P<.01$). The failure mode was 100% adhesive for all groups.

Conclusions. The metal primers did not promote an increase in adhesive bonding of resin cements to NiCr alloy and to CP Ti. Water storage had no adverse effect on the shear bond strength of the groups. The shear bond strengths to titanium were significantly higher than those to the NiCr alloy. (J Prosthet Dent 2009;101:262-268)

> ## Clinical Implications
> The results of this study indicate that the use of Alloy Primer prior to Panavia F is not recommended for cementation of NiCr crowns and is not necessary for the cementation of titanium crowns. For the cementation of NiCr and titanium crowns with Bistite II DC, the prior use of Metaltite is not necessary.

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Strong and durable adhesive bonding between a metal framework and a luting agent is important to withstand the many and varied changes in the oral environment. Adhesion of resin to a substrate depends on both micromechanical interlocking and physicochemical bonding.[1-3] The former can be obtained by airborne-particle abrasion with aluminum oxide, whereas the latter is achieved by functional monomers contained in resin-based materials or metal primers.[4-7]

Metal primers are used to create a strong bond between metal and resin-based materials.[8-11] It is known that metal primers contain active monomers that promote chemical bonding between the cement and the oxides present on the metal surface.[12-15] Metal primers containing MDP (10-methacryloyloxydecryl dihydrogen phosphate),[16-19] MEPS (thiophosphate methacryloyloxyalkyl) derivatives,[11,17,19] or 4-META (4-methacryloyloxyethyl trimellitate anhydride)[20,21] are reported to yield high bond strengths between resin-based materials and base metal alloys.

Although some authors have evaluated the effects of metal primers on resin bonding to base metal alloys,[22] no studies using NiCr were identified, in spite of this alloy being used to fabricate metal ceramic prostheses in many Western countries.[23] Studies evaluating the effect of metal primers on resin bonding to titanium have been conducted, with satisfactory results[7,24,25]; nevertheless, insufficient information remains. These studies[7,24,25] were encouraged by the attractive biologic, physical, chemical, and mechanical properties of titanium,[26,27] as well as the poor bonding between cast titanium and composite resin materials.[11,27-29]

Another important point is that these materials are used in the oral cavity and are subject to the actions of humidity and temperature variations, which may influence the durability of resin bonding to metal surfaces.[21,30,31] The purpose of this study was to evaluate the chemical efficacy of metal primers on the shear bond strength of resin cements to CP Ti and NiCr alloy. The hypotheses were: (1) metal primers would increase the bond strength of their respective resin cements to base metal alloys; and (2) water storage would decrease the bond strength of resin cements used with or without the respective metal primers.

MATERIAL AND METHODS

Cylindrical specimens (9.0 mm in diameter and 3.0 mm thick) were cast from nickel-chromium base metal alloy and CP Ti grade II. Manufacturing information and composition are presented in Table I. The nickel-chromium and CP Ti cylinders were embedded in a polyvinyl chloride ring (2.5 mm in diameter and 27.0 mm high), using polymethyl methacrylate acrylic resin (Clas-Mold; Artigos Odontológicos Clássico Ltd, São Paulo, Brazil). All specimen bonding surfaces were smoothed with silicon carbide paper (320, 400, and 600 grit) (3M Brazil Ltd, Campinas, São Paulo, Brazil) and airborne-particle abraded with 50-μm aluminum oxide for 20 seconds at a pressure of 35 psi and a distance of 10.0 mm from the specimen surface. Eighty specimens of each substrate were divided into 4 groups (n=20). A custom-made metal matrix (5.0-mm internal diameter, 2.0 mm thick) was placed on the surface of the specimen, using a centralizing ring attached to the polyvinyl chloride (PVC) tube. The purpose of this matrix was to allow the addition of the resin cement at a constant diameter and thickness on the metal substrate. The bonding sites received one of the following luting techniques: (1) Panavia F, (2) Alloy Primer plus Panavia F, (3) Bistite II DC, or (4) Metaltite plus Bistite II DC (Table I). Each primer, as a single liquid, was applied to the alloy surface with a brush for 15 seconds and then air dried for 5 seconds. The resin cements were apportioned by weight. To avoid exposure of the resin cements to daylight, they were mixed and inserted into the matrix inside a radiographic developing chamber (Odontologic Indústria e Comércio Ltda, São Paulo, Brazil). A Mylar strip (Probem, Catanduva, Brazil), a glass slab, and a 0.5-kg weight were placed on top of the resin cements to permit overflow of a slight excess of material. Thus, the cements were protected against exposure to oxygen. Forty minutes after preparation, all specimens were stored in distilled water at 37°C for 24 hours before thermal cycling between 5°C and 55°C for 1000 cycles with a 30-second dwell time. After thermal cycling, the specimens were stored in 37°C distilled water for an additional 24 hours or 6 months (n=10) before being subjected to a shear load using a testing machine (MTS 810 Material Testing System; MTS Systems Corp, Eden Prairie, Minn), with a 1-kN load cell and a crosshead speed of 0.5 mm/min. A chisel apparatus was used to direct a parallel shearing force as closely as possible to the luting agent-metal interface (Fig. 1). Shear bond strength values were recorded in MPa.

Each specimen was examined under an optical microscope (Carl Zeiss GmbH, Jena, Germany) at ×30 magnification, and digital images were captured by a computer program (Leica QWin; Leica Microsystems AG, Heerbrugg, Switzerland). Failure mode was recorded by a single calibrated observer as either adhesive (failure at the substrate-resin interface), cohesive (failure within the substrate or within the restorative material), or combination (areas of adhesive and cohesive failure). For this classification, the adhesive area was divided into quadrants, and in each of them, the predominant type of fracture was observed. According to the method of dos Santos et al,[32] the fracture was classified as adhesive or cohesive if either of these types predominated in 3 or more quadrants, and classified as a combination if 2 quadrants presented adhesive failure and the other 2 cohesive failure. Three variables and their interactions were investigated: luting

TABLE I. Materials evaluated

Material	Composition	Trade Name	Manufacturer	Monomers	Lot Number
NiCr	Ni 75%, Cr 11.5%, Mo 3.5%, others 10%	Verabond II	Aalba Dent, Inc, Cordelia, Calif	-	-
CP Ti grade II	Ti 99.56%, others 0.44%	-	RTI Intl Metals, Inc, Niles, Ohio	-	-
Primers		Alloy Primer	Kuraray Co Ltd, Osaka, Japan	MDP, VBATDT	00139A
		Metaltite	Tokuyama Dental Corp, Tokyo, Japan	MTU-6	012M1
Resin cements		Panavia F	Kuraray Co Ltd	MDP	00235B paste A 00022E paste B
		Bistite II DC	Tokuyama Dental Corp	MAC-10	UB 91464

MDP: 10-methacryloyloxydecryl dihydrogen phosphate; VBATDT: 6-(4-vinylbenzyl-η-propyl)amino-1,3,5-triazine-2,4-dithione; MTU-6: 6-methacryloyloxyhexyl-2-thiouracil-5-carboxylate; MAC-10: 11-methacryloyloxundecan 1,1-dicarboxylic acid
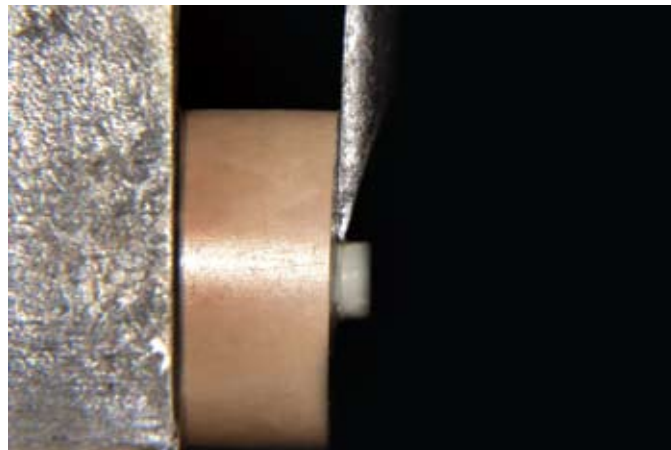
1 Shear bond strength testing apparatus.

technique, storage time, and metal. Data were analyzed by 3-way ANOVA, and the mean values were compared by the Tukey HSD test (α=.05).

RESULTS

Mean shear bond strength values and standard deviations for each group are presented in Table II. The results of the 3-way ANOVA (Table III) showed that luting agent ($P<.001$), storage time ($P<.01$), metal ($P<.001$), and their interactions were significant ($P<.001$). When the luting technique variable was investigated, there was no significant difference between the Panavia F and Alloy Primer plus Panavia F groups, except for the NiCr alloy at 24 hours, at which point Panavia F was superior to Alloy Primer plus Panavia F ($P<.001$). For both metals and storage times, the Bistite II DC and Metaltite plus Bistite II DC groups did not differ statistically. The materials Bistite II DC and Metaltite plus Bistite II DC demonstrated significantly lower bond strength to CP Ti ($P<.001$) than the Panavia F and Alloy Primer plus Panavia F groups ($P<.001$), and significantly lower bond strength to NiCr alloy ($P<.001$) than Panavia F.

With regard to the significant interaction between luting technique and storage time ($P<.001$), only the Panavia F ($P<.01$) and Alloy Primer plus Panavia F ($P<.001$) bond strengths to titanium were affected by water storage, presenting a significant increase in shear bond strength at 6 months. The other groups were not significantly influenced by water storage.

A significant interaction between luting technique and metal was also detected ($P<.001$). Except for the Bistite II DC and Metaltite plus Bistite II DC groups at 24 hours ($P>.05$), all of the other groups exhibited higher shear bond strength to CP Ti than to NiCr alloy ($P<.01$). Finally, there was a significant interaction between

TABLE II. Mean shear bond strength values (standard deviations) (MPa) for all groups

	NiCr		CP Ti	
	24 h	6 mos	24 h	6 mos
Panavia F	8.25 (1.68)^{A,c}	7.21 (1.38)^{A,c}	10.57 (1.77)^{A,b}	13.86 (2.60)^{A,a}
Alloy Primer plus Panavia F	5.39 (1.09)^{B,c}	5.51 (0.61)^{AB,c}	8.74 (0.92)^{A,b}	12.64 (1.26)^{A,a}
Bistite II DC	5.00 (0.98)^{B,ab}	3.74 (0.59)^{BC,b}	5.24 (0.95)^{B,ab}	5.75 (0.81)^{B,a}
Metaltite plus Bistite II DC	4.61 (0.77)^{B,bc}	3.21 (0.34)^{C,c}	6.24 (1.22)^{B,ab}	6.80 (1.30)^{B,a}

Different uppercase letters indicate significant differences in columns ($P<.05$).
Different lowercase letters indicate significant differences in rows ($P<.05$).

TABLE III. Three-way ANOVA

Source of Variation	SS	df	MS	F	P
Luting agent	696.6	3	232.2	146.2	<.001
Storage time	13.7	1	13.7	8.6	<.01
Metal	453.3	1	453.3	285.3	<.001
Luting agent × storage time	42.4	3	14.1	8.9	<.001
Luting agent × metal	103.4	3	34.5	21.7	<.001
Storage time × metal	87.6	1	87.6	55.1	<.001
Luting agent × storage time × metal	12.5	3	4.2	2.6	.053
Error	228.7	144	1.6		
Total	9585.3	160			
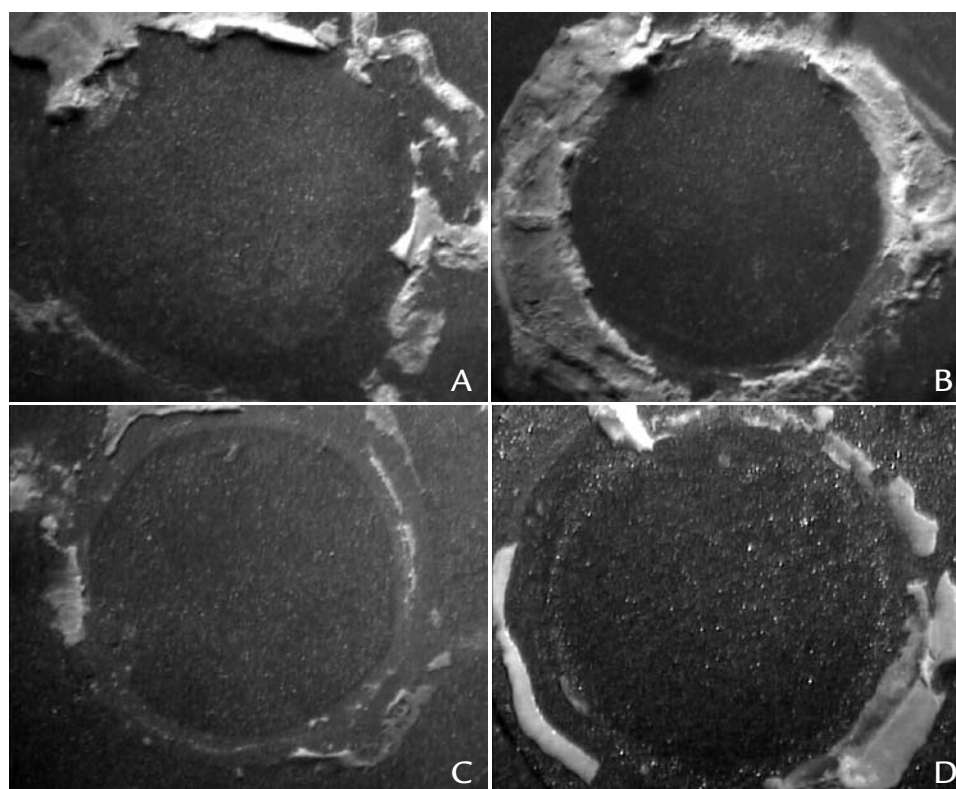
2 NiCr alloy after 6 months of storage time. A, Adhesive failure with Panavia F (original magnification x30). B, Adhesive failure with Alloy Primer plus Panavia F (original magnification x30). C, Adhesive failure with Bistite II DC (original magnification x30). D, Adhesive failure with Metaltite plus Bistite II DC (original magnification x30).
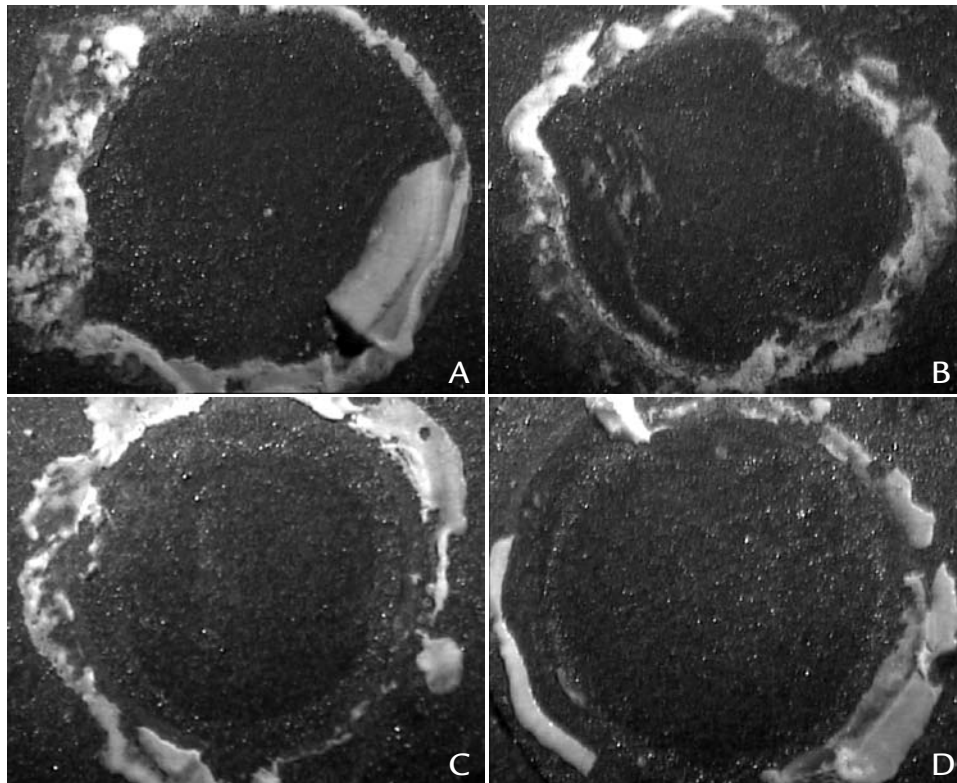
3 CP Ti after 6 months of storage time. A, Adhesive failure with Panavia F (original magnification x30). B, Adhesive failure with Alloy Primer plus Panavia F (original magnification x30). C, Adhesive failure with Bistite II DC (original magnification x30). D, Adhesive failure with Metaltite plus Bistite II DC (original magnification x30).

storage time and metal ($P<.001$). For the NiCr alloy, water storage had no significant influence. For titanium, a significant increase in bond strength occurred at 6 months in the Panavia F ($P<.01$) and Alloy Primer plus Panavia F ($P<.001$) groups.

The failure mode was 100% adhesive for all groups. Predominant types of bond failure at 6 months are reported in Figures 2 and 3 for NiCr alloy and CP Ti, respectively.

DISCUSSION

The results of this study did not support acceptance of the research hypotheses, since metal primers did not increase the bond strength of their respective resin cements to base metal alloys, and water storage did not decrease the bond strength of resin cements used with or without the respective metal primers. In general, the findings of this study demonstrated that there was no significant difference between the Panavia F and Alloy Primer plus Panavia F groups, except for the NiCr alloy at 24 hours, at

which point Panavia F was superior to Alloy Primer plus Panavia F ($P<.001$). Taira et al[24] also observed statistical equality in the shear bond strength of Panavia 21 (Kuraray America, Inc, New York, NY) to CP Ti, with and without the use of Alloy Primer, after 24 hours of water storage and thermal cycling. With regard to the NiCr alloy, no studies evaluating the influence of Alloy Primer on the bond strength of Panavia to this alloy were identified.

Studies have shown that MDP monomer is capable of increasing the bond strength of resin cements to base metals. Tsuchimoto et al[10] showed that the application of 10% in mass of the MDP monomer significantly increased the tensile bond strength of Panavia F to CP Ti after 24 hours of storage in water. Matsumura et al[8] observed that a significant increase in the shear bond strength of Super-Bond C&B (Sun Medical Co, Ltd, Moriyama, Japan) to CP Ni (99.9% Ni) and to CP Ti (99.7%) was promoted by the primer Cesead Opaque Primer (Kuraray Co, Ltd), which contains only the MDP monomer. Taira et

al[9] verified that this primer increased the bond strength of Panavia 21 (Kuraray America, Inc) to CP Ti, and justified this by the increase in the concentration of free phosphate at the resin-metal interface.

However, the constituents of Alloy Primer are MDP and VBATDT (6-(4-vinylbenzyl-η-propyl)amino-1,3,5-triazine-2,4-dithione) monomers. There are reports that the VBATDT monomer harms the polymerization reaction of resin-based materials that contain the benzoyl peroxide-amine initiator system, such as Panavia F.[25,30] Nevertheless, in the present study, the Panavia F groups with or without Alloy Primer presented a predominance of adhesive failure, indicating that the weakest link was not in the cement, but in the layer of oxides. It is speculated that the monomer VBATDT could have interfered in the reaction between the MDP monomer contained in the primer and that in the cement and metal oxides. In spite of this possible influence having been more evident in the NiCr alloy at 24 hours, it is conceivable that if

this monomer did not form part of the composition of the Alloy Primer, this primer would have increased the bond strength in the other groups (CP Ti at 24 hours and 6 months and NiCr alloy at 6 months).

In view of the results presented, and despite the indication of Alloy Primer for noble and base metal alloys, the use of this metal primer on NiCr alloys and CP Ti requires further investigation. There are no studies with evidence of a benefit resulting from the use of this primer on these metals. In the present study, Alloy Primer did not increase the bond strength to CP Ti and significantly decreased the adhesive bonding of Panavia F to the NiCr alloy.

For both metals and storage times, the Bistite II DC and Metaltite plus Bistite II DC groups did not differ statistically. Therefore, this primer neither increased nor reduced the bond strength of its respective resin cement, which is in agreement with the information purported by the manufacturer. It is interesting to note that the MTU-6 (6-methacryloyloxyhexyl-2-thiouracil-5-carboxylate) monomer contained in the primer, which, according to Yoshida et al,[14] has a structure similar to that of VBATDT, did not appear to harm the reaction of the MAC-10 (11-methacryloyloxundecan 1,1-dicarboxylic acid) monomer contained in Bistite II DC (which also has a benzoyl peroxide-amine initiator system) with the oxides present in the metal substrate.

The superiority of the Panavia F and Alloy Primer plus Panavia F groups over the Bistite II DC and Metaltite plus Bistite II DC groups observed in the present study is in agreement with the findings of other studies[7,9,29] that verified that the MAC-10 monomer was shown to be less effective than the MDP monomer, both on cast CP Ti and on Ti6Al7Nb.

With respect to the effect of water storage on the adhesive bonding of resin cements, the statistical equality observed in the majority of the groups could have been the result of: (1) the hydrolysis process occurring slowly, and, therefore, the storage time of 6 months being insufficient for the effects to be detected; or (2) the equivalence of progression of the chemical reaction between the monomers and metal oxides and the effect of hydrolysis at this interface.

In titanium, the significant increase in bond strength at 6 months in the Panavia F and Alloy Primer plus Panavia F groups may have been the result of greater progression of the chemical reaction between the MDP monomer and the metal oxides when compared with that of hydrolysis. According to Yanagida et al,[7,29] the MDP monomer, present in both the Alloy Primer and Panavia F, consists of 3 components that function differently: the methacryloyl, dihydrogen phosphate, and decyl groups. The methacryloyl group is indispensable to copolymerize the MDP monomers in the primer and the matrix monomers in the resin cement. The dihydrogen phosphate group chemically bonds to metal oxides. The decyl group prevents penetration of water into the adhesive interface, either reducing or retarding hydrolysis. Nevertheless, a factor of fundamental importance to consider is the composition of the layer of oxides present on the metal surface, which will establish chemical reactions with the different monomers. In the literature, the only studies[3,7-9,13,17,18,20,21,23,24] identified evaluated the effect of thermal cycling and not of water storage, per se, on the bond strength between the metals used in this study and resinous materials, so there are no parameters for comparison. Except for the Bistite II DC and Metaltite plus Bistite II DC groups at 24 hours, all of the other groups exhibited higher shear bond strength to CP Ti than to NiCr alloy.

Possibly, the chemical bonds between monomers (MDP and MAC-10) and the oxides present at the surface of the CP Ti were stronger than those that occurred between these monomers and the oxides of the NiCr alloy. Kern et al[26] verified that the MDP monomer establishes stable chemical bonds with titanium and that the oxides on the titanium surface are more stable in water than the surface oxides of other metals. Lorey et al[31] also found greater adhesive bonding of Panavia to CP Ti than to NiCr alloy. Nevertheless, there are few studies that compare the adhesive bonding to CP Ti and NiCr alloy.

The success of an adequate bond depends on selecting the best combination of metal/metal primer/resin cement. The monomers of the primer must establish a strong and durable bond with the oxides of the metal surface and with the monomers present in the resinous material.

Furthermore, water ingress is not the only factor that may influence the durability of resin bonds. Other elements, such as pH changes and dynamic fatigue loading, which were not evaluated in the present study, are also capable of influencing this property. Therefore, careful interpretation in the clinical application of the results is suggested. Further in vitro research and long-term clinical studies must be conducted to affirm the efficacy of the tested systems.

CONCLUSIONS

Within the limitations of this in vitro study, the following conclusions were drawn:

1. The Alloy Primer significantly decreased the shear bond strength of Panavia F to NiCr alloy at 24 hours and had no influence on the other groups.

2. For both metals and storage times, Metaltite did not have any effect on the bond strength of Bistite II DC.

3. Water storage affected only the Panavia F and Alloy Primer plus Panavia F groups' bond to titanium, increasing the shear bond strength of these groups at 6 months.

4. The shear bond strengths to titanium were significantly higher than those to the NiCr alloy.

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