Original Article

Bond strength of self-adhesive resin cement to base metal alloys having different surface treatments

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ABSTRACT

Background: This study aimed to assess and compare the shear bond strength of self-etch and self-adhesive resin cement to nickel-chromium-cobalt alloy with different surface treatments.

Materials and Methods: In this *in vitro* study, a total of 120 disks were fabricated of VeraBond II base metal alloy. Specimens were divided into 15 groups of 8 based on the type of cement and surface treatment. The five surface treatments studied included sandblasting alone, application of Alloy Primer with and without sandblasting, and application of Metal Primer II with and without sandblasting. The three cement tested included Panavia F2.0, RelyX Unicem (RU), and G-Cem (GC). After receiving the respective surface treatments, the specimens were thermocycled for 1500 cycles and underwent shear bond strength testing. Data were analyzed using SPSS 20.0 and three-way analysis of variance. *P* values of the significant level of 0.05 were reported.

Results: The results exhibited that the mean bond strengths in sandblasted groups were higher than nonsandblasted one. These differences were significantly higher in the sandblasted groups of Panavia F2.0 and RU cement ($P < 0.05$). The mean bond strength values between GC and Panavia F2.0 were not statistically significant ($P > 0.05$). The highest bond strength was recorded for Panavia F2.0 with the surface treatment of both sandblasting and Metal Primer II.

Conclusion: Based on the results, sandblasting improves the shear bond strength of self-etch and self-adhesive resin cement to base metal alloys. The best results can be achieved with a combination of sandblasting and metal primers. The performance of resin cement depends on to their chemical composition, not to the type of system.

Key Words: Dental alloys, self-adhesive, resin cement, bond strength

INTRODUCTION

The longevity of an indirect restoration is dependent on different factors including those related to the patient, dentist, kind of material, type of luting cement, and the technique of luting procedures.[1] Although zinc phosphate cement are used more than 100 years as a luting material in dentistry, they have some drawbacks such as the low strength, poor esthetics, inadequate adhesion, and high solubility. These disadvantages make them an improper material in many clinical situations.[2] Resin cement are

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achieving favor as they have several characteristics that make them as superior luting agents in dental clinics. Resin cement have high bond strengths to tooth, high-tensile, and compressive strengths, and also they have lower solubility compared to conventional cement; furthermore, flexural properties which are important for prevention of debonding are also the highest in resin cement.[3]

The shortcoming of resin cement is considered with their technique sensitivity and difficult bonding procedures. Hence, the operator must be careful to do all steps in proper order as recommended by manufacturer.[3,4] Self-adhesive resin cement were introduced as a new division of resin cement in 2002. These new materials were designed with the purpose to overcome some limitations of traditional and resin-based cement. These cement simplify bonding process and no pretreatment is requiring on the tooth surface. Since their application is in a single clinical step, and so compared to total-etch resin cement, the risk of contamination is less, and hence, better adhesion to tooth surfaces can be achieved.[5,6]

Several tests have been used to evaluate the bond strength of adhesive resins.[7] Although the shear bond strength has been questioned by some researchers,[7,8] it is the most common method which is used for evaluation of bond strength. Some reasons can explain the preference of shear bond strength test to other methods, one of these reasons is that the values of shear bond strength test are higher than tensile strength test, also this test is closer to clinical situation and so can be a better simulation for oral conditions,[9] and on the other hand, this method is reproducible and almost easy and not a complicated procedure.[10,11]

The bond between a resin cement to a metal framework dependent on to various factors including the type of alloy, the kind of resin cement, and the surface treatment on the metal framework which the latter is the first important step. The surface treatment of metal causes changes in chemistry or the surface texture of the metal and therefore enhancing the chemicomechanical bond between the metal and the adhesive cement.[12]

Airborne-particle abrasion with Al_2O_3 particles is the most common method used for promoting micromechanical retention.[13] This method is inexpensive and may improve the mechanical bond between cement and the substrate by removal of debris from the surface.[14] On the other hand, chemical bonding between resin cement and metals can be achieved with various adhesive monomers, metal primers, and silane.[15-17]

Metal primers include active monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP), methacryloyloxyalkyl thiophosphate (MEPS), 4-methacryloxyethyl trimellitate anhydride (4-META), and others which bond to metals and alloys due to their affinity to metal oxides that present on metal surfaces.[18,19]

The 6-(4-vinylbenzyl-n-propyl) amino-1, 3, 5-triazine-2, 4-dithione (VBATDT), specifically enhances bonding to metals particularly to noble metals.[18] On the other hand, studies showed MDP that is a phosphoric acid monomer, and MEPS improves retention of resins to a base metal alloy.[18,20,21]

This study aimed to assess the shear bond strength of a self-etch and two self-adhesive resin cement to VeraBond II base metal alloy receiving different surface treatments. The null hypothesis tested was that Alloy Primer (AP) application, type of cement, and sandblasting would not affect increasing the shear bond strength of the base metal alloy.

MATERIALS AND METHODS

Preparation of specimens

This study was supported by the research Grant No. 11737 from Tehran University of Medical Sciences. In this *in vitro* study, 120 disk-shaped specimens (1 cm in diameter, 2 mm in height) were cast from nickel-chromium (VeraBond II) base metal alloy. These specimens were divided into 15 groups according to the surface treatments and the kind of cement. A dual-cured resin cement Panavia F 2.0(P) (Kuraray Medical Co. Ltd., Osaka, Japan) and two self-adhesive resin cement, RelyX Unicem (RU) (3M ESPE; St Paul, MN, USA) and G-Cem (GC) (GC, Tokyo, Japan), were used. Two different metal primers including Metal Primer II (MP) (GC Corp., Tokyo, Japan) and AP (Kuraray Co., Ltd., Osaka, Japan) also were chosen. The compositions of base metal alloy, adhesive resins, and metal primers used in this study are listed in Table 1.

All specimens were mounted in a self-polymerizing acrylic resin block (DeguDent, Dentsply, UK) for the purpose to hold the base metal alloy specimens to the

Table 1: Materials used in this study

Materials	Composition	Manufacturer	Lot number
Alloy Primer	Acetone MDP VBATDT	Kuraray Medical Co., Osaka, Japan	-
Metal Primer II	MEPS thiophosphoric methacrylate	CG International, Tokyo, Japan	-

Panavia F2.0	Paste A
	  MDP, dimethacrylates, silanated silica filler, silanated
	  colloidal silica, dl-camphorquinone, catalysts, initiators
	Paste B
	  Dimethacrylates, silanated barium glass filler,
	  surface-treated sodium fluoride, catalysts, accelerators,
	  pigments
		Kuraray Medical Co., Osaka, Japan	51217

RelyX Unicem	Powders: fluoroaluminosilicate glass, initiator, pigments, light-curing initiator (filler load 72% wt, filler size <9.5 µm), liquid: Methacrylated phosphoric esters, dimethacrylates, acetate, stabilizers, self-curing initiator, pigments, light-curing initiator	3M ESPE	347167
G-Cem	Powders: Glass filers, silica, calcium hydroxide, self-curing initiator, pigments Liquid: 4-MET, phosphoric acid ester monomer, water, UDMA, dimethacrylate, silica powder, stabilizers, initiator	GC, Tokyo, Japan	0901291
VeraBond II alloy	Ni 77.95%, Cr 12.60%, Mo 5.00%, Al 2.90%, Co 0.45%, Be 1.95%	Alba Dent (USA)	-

MDP: 10-methacryloyloxydecyl dihydrogen phosphate; VBATDT: 6-(4-vinylbenzyl-N-propyl) amino-1,3,5-triazine-2,4-dithione; 4-MET: 4-methacryloxyethyl trimellitic acid; UDMA: Urethane dimethacrylate; MEPS: Methacryloyloxyalkyl thiophosphate

test machine. The bonded surfaces of all specimens were smoothed with 600, 800, and 1000 grit silicon carbide (Pirasanat, Iran) abrasive papers under water cooling and then ultrasonicated in distilled water for 5 min. According the surface treatment procedure, the specimens were divided into five groups as follows: (1) Sandblast: In the sandblasting groups, the specimens ($n = 72$) were abraded using 50-µm aluminum oxide airborne particles under 3 bar pressure for 10 s at a distance of 10 mm; (2) Alloy Primer: One coat of AP was applied on the metal surface and then left for a few seconds according the manufacturer instruction; (3) Metal Primer II: A thin layer of Metal Primer II was applied using a brush to the bonding surface of metal and left for a few seconds to dry; (4) Sandblast + AP: A thin coat of AP was applied to the sandblasted metal surface; and (5) Sandblast + Metal Primer II: A thin layer of Metal Primer II was applied to the sandblasted metal surfaces with alumina.

The cement (4 mm in diameter and 3 mm high) then were used on the treated surfaces according to the manufacturer's recommendations as follows: Panavia F2.0: The mixed ED Primer II first was applied and gently air dried, the equal amounts of each paste of Panavia F2.0 were mixed with each other, and the mixture was used; GC: The activated capsule was placed into an amalgamator and mixed

for 10 s. Then, the mixed capsule was load into the capsule applier and placed on the metal surface; and RU: The activated capsule was placed into a mixing device (amalgamator) and mixed for 10 s; subsequently, the mixed capsule was inserted into the applier to place on the surface. The abbreviations of the groups are listed in Table 2.

The cement were prepared as described above, bonded to treated surfaces, and cured by QTH curing unit (Coltolux® 75, Germany) for 1 min. The light output was tested by radiometer (Optilux, Model 100, 10503, Kerr, USA), which registered over 600 mW/cm².

All specimens were stored in distilled water for 24 h at 37°C. Following storage, they subjected to thermal cycling between 5°C and 55°C for 1500 cycles with a dwell time of 20 s.

Shear bond strength test
The shear bond strength was measured using a universal testing machine with a 50.0-kg load cell (Bongshin®, Bongshin Loadcell Co., LTD, Seoul, Korea) at a crosshead speed of 0.5 mm/min. The specimens were mounted in the jig of UTM, and the load was applied using a chisel apparatus to the interface of adhesive resin and base metal alloy until failure occurred.

Shear bond strengths were calculated by dividing the maximum load (N) to the bonding area (mm²) and recorded in MPa.

Table 2: Abbreviations of testing groups according to the types of resin cement, kind of Alloy Primer, and surface treatments

Group	Resin cement	Sandblasting	Metal primer
P (S)	Panavia F2.0	Yes	No
RU (S)	RelyX Unicem	Yes	No
GC (S)	G-Cem	Yes	No
P (AP)	Panavia F2.0	No	Alloy Primer
RU (AP)	RelyX Unicem	No	Alloy Primer
GC (AP)	G-Cem	No	Alloy Primer
P (MP)	Panavia F2.0	No	Metal Primer II
RU (MP)	RelyX Unicem	No	Metal Primer II
GC (MP)	G-Cem	No	Metal Primer II
P (S + AP)	Panavia F2.0	Yes	Alloy Primer
RU (S + AP)	RelyX Unicem	Yes	Alloy Primer
GC (S + AP)	G-Cem	Yes	Alloy Primer
P (S + MP)	Panavia F2.0	Yes	Metal Primer II
RU (S + MP)	RelyX Unicem	Yes	Metal Primer II
GC (S + MP)	G-Cem	Yes	Metal Primer II

Failure mode observation

After shear bond strength testing, the fractured surfaces of each specimen were examined using a stereomicroscope (EZ4D; Leica Microsystems Ltd., Singapore) at ×20 magnification. Failure modes were classified as adhesive failure, cohesive failure, or mixed failure. Adhesive failure was determined when debonding occurred on the interface of nickel-chromium to adhesive cement whereas 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.

Statistical analysis

The effect of surface treatment, type of adhesive cement, and kind of AP were evaluated statistically using a 3-way analysis of variance (ANOVA). Since the interaction effect of the sandblasting and cement and the interaction effect of AP and cement were significant, a separate analysis was performed in each group.

To compare the shear bond strength between three different resin cement and three different surface treatments, Kruskal–Wallis test and a subsequent pairwise comparison were applied and adjusted *P* values of the significant level of 0.05 were reported. All statistical analysis was carried out using SPSS version 20.0 (IBM, Armonk, NY, USA). The significance level was set at *P* = 0.05.

RESULTS

The mean shear bond strength and standard deviations of all groups are presented in Table 3. A bar chart

is also plotted for better comparison of the effect of the type of cement and surface treatment on the shear bond strength of resin cement to base metal alloy [Figure 1]. Kruskal–Wallis ANOVA exhibited that the mean shear bond strengths in sandblasted groups were higher than nonsandblasted one. These differences were significantly higher in the sandblasted groups of P and RU cement ($P < 0.05$).The mean bond strength values between GC and *P* were not statistically significant ($P > 0.05$).The highest bond strength was recorded for P (S + MP) and the lowest one was obtained for RU (MP).

According to the type of cement, the following results were obtained:

Panavia F2.0 (P)

For this dual-cured resin cement, these statistical differences were observed. (1) In sandblasted groups, the shear bond strength of specimens pretreated with both metal primers, P (S + AP) and P (S + MP), was significantly higher than the specimens which nontreated with these metal primers ($P < 0.001$). (2) In the specimens pretreated with both metal primers, those specimens were sandblasted before priming had the shear bond strength significantly higher than nonsandblasted one ($P < 0.001$). (3) The highest shear bond strength was obtained for specimens pretreated with sandblasting followed by priming with MP, and (4) the lowest shear bond strength was related to the group which only pretreated with AP.

G-Cem

Kruskal–Wallis test showed no statistically significant difference in the specimens, those cemented with GC associated with the surface treatment. Comparison of the surface treatments showed that GC luting cement exhibited similar behavior for all the surface treatments [Figure 2].

RelyX Unicem

Pairwise comparisons of different surface treatments on shear bond strength subsequent to Kruskal–Wallis analysis of variance exhibited statistically significant difference only between the groups of RU (MP) and RU (S + MP). In other groups, according to surface treatment, no significant difference was found between groups ($P > 0.05$).

In nonsandblasted specimens, pairwise comparisons of cement indicated that the shear bond strengths between groups of RU (AP) and GC (AP), RU (MP), and P (MP) and also between groups RU (MP) and GC (MP) were significantly different ($P < 0.05$).

Table 3: Mean shear bond strength and standard deviation of experimental groups

Resin cement (*n*=8)	Surface treatment (mean±SD)				
	Sandblasting	**Metal Primer II**	**Alloy Primer**	**Sandblasting + Metal Primer II**	**Sandblasting + Alloy Primer**
Panavia F2	2.79±1.11[a,b]	3.39±0.75[c,e]	2.73±6.45[d]	5.31±1.21[a,c]	4.89±1.28[b,d]
RelyX Unicem	3.12±1.07	1.92±0.8[e,f]	2.57±0.6[g]	4.62±1.09	3.28±1.48
G-Cem	4.25±1.51	3.72±0.51[f]	3.6±1.59[g]	4.27±0.79	4.36±1.25

The same letters denote the groups which have statistically significant differences. [a,b]The difference between the shear bond strenghth(MPa) of sandblasted specimens and the specimens those treated with both metal primers before sandblasting is *P*<0.001; [c,d]The difference between the shear bond strenghth (MPa) of nonsandblasted specimens and the specimens those sandblasted before treating with both metal primers is *P*<0.001; [e]In the specimens those treated with Metal Primer II, the difference between the shear bond strength of RelyX Unicem and Panavia F2 groups is *P*=0.027; [f]In the specimens those treated with Metal Primer II, the difference between the shear bond strength of RelyX Unicem and G-Cem groups is *P*=0.030; [g]In the specimens those treated with Alloy Primer, the difference between the shear bond strength of the RelyX Unicem and G-Cem groups is *P*=0.044. SD: Standard deviation
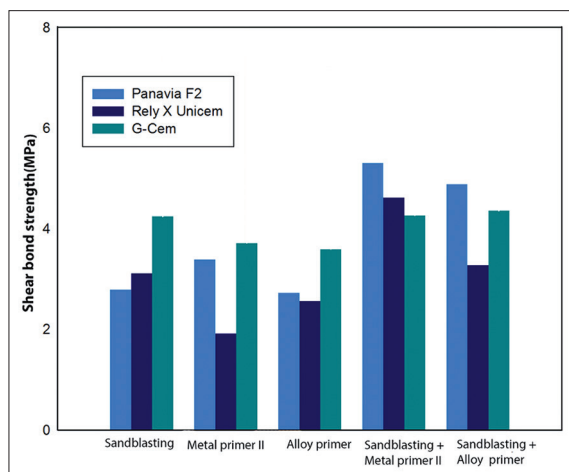

Figure 1: Bar chart of the effect of cement type and surface treatment on the shear bond strength of resin adhesive cement to base metal alloy.
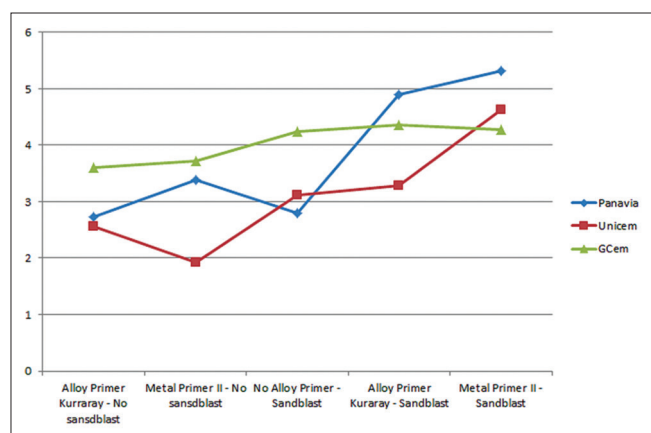

Figure 2: Effect of surface treatment procedures on the shear bond strength of base metal alloy to three different resin adhesive cement.

According to that the shear bond strength of groups, GC (AP), P (MP), and GC (MP) were higher than groups RU (AP) and RU (MP).

Among all surface treatments, the one associated with the MP without sandblasting showed worse shear bond strength results, whereas MP + sandblast

exhibited the highest shear bond strength values for RU cement. These results were as similar as P.

Mode of failure

The results of mode of failure observed by optical microscopy revealed that except for P (S + MP) group, which its mode of failure was 100% mix, all specimens showed a mixture of adhesive and mix failures on the bond surface regardless of the luting material or the surface treatment [Table 4 and Figure 3].

DISCUSSION

Metal-ceramic fixed dental restorations are widely used due to their few technical and biological complications and also good patient satisfaction.[22]

The long-term survival of metal-ceramic restorations depends on the accuracy of all steps of treatment including the kind of luting cement and the cementation procedure. Various types of cement are used for luting of fixed partial dentures (FPDs) to tooth structure. The use of self-adhesive cement for this purpose is increasing due to their easy handling, biocompatibility, adequate working time and setting time, adequate primary strength against functional loads, insolubility, sealability, radiopacity, high esthetics, and affordability. For bonding with resin cement, a property surface treatment must be applied before cementation to achieve a good bonding between resin cement and substrates.[23]

This study aimed to compare the shear bond strength of two self-adhesive cement (RU and GC) and Panavia F2 conventional resin cement to base metal alloy treated with sandblasting and metal primers alone or in conjunction with each other.

The results of this study exhibited that shear bond strength was improved in the sandblasted group in both self-etch and self-adhesive resin cement,
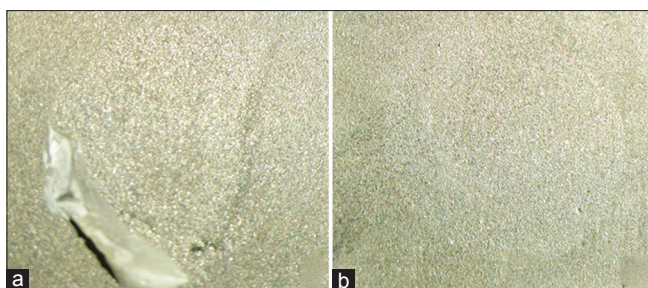
Figure 3: Representative examples of mode of failure observed using stereomicroscope. (a) Mixed failure, (b) adhesive failure.

Table 4: Frequency of modes of failure observed under a stereomicroscope

Group	Mode of failure		
	Adhesive (%)	Mixed (%)	Cohesive (%)
YAP	3 (37.5)	5 (62.5)	0
YAR	4 (50)	4 (50)	0
YAG	3 (37.5)	5 (62.5)	0
YMP	0	8 (100)	0
YMR	4 (50)	4 (50)	0
YMG	3 (37.5)	5 (62.5)	0
NAP	6 (75)	2 (25)	0
NAR	7 (87.5)	1 (12.5)	0
NAG	6 (75)	2 (25)	0
NMP	4 (50)	4 (50)	0
NMR	2 (25)	6 (75)	0
NMG	4 (50)	4 (50)	0
YNP	4 (50)	4 (50)	0
YNR	4 (50)	4 (50)	0
YNG	7 (87.5)	1 (12.5)	0

which these changes were statistically significant in groups of P and RU [Table 3]. This finding is consistent with the results of Sarafianou *et al.*[24] Several reasons can be proposed for this finding. The sandblasting procedure roughens the surface of the metals and so increases the surface area and wettability and creates a mechanical interlocking and greater affinity between adhesive resins and base metal alloys.[17,25]

Different studies determined the effectiveness of metal priming on the shear bond strength of dental resins to metal substrate.[26,27] Chemical formulation of resin adhesives and primers determines their clinical application. Monomers are important constituents of adhesives and primers and are available in two forms of cross-linkers and functional monomers. Functional monomers mainly undergo linear polymerization increasing the mechanical strength. Whereas, cross-linker monomers undergo cross-linking polymerization and are important for reinforcing the adhesive resins.

Panavia F2.0, dual-cured adhesive resin cement, and AP consist of 10-MDP monomer which can bind chemically to metal oxides of nickel, chromium, and cobalt.[28] 10-MDP is a functional monomer originally manufactured by Kuraray, Japan. It is mainly an etchant agent due to the dihydrogen phosphate group in its composition that the presence of water can disintegrate into two H + ions.[29] MDP is an organic ester which can chemically bind to the oxide layer created on the metal surface through covalent bonds and also mechanical retention to the sandblasted surface. Among phosphate monomers, MDP seems to be more suitable for bonding to base metal alloys and provides greater bond strength. In the current study, the highest shear bond strength was observed in the specimens that P cemented to Ni-Cr base metal alloy treated with sandblasting + MP. It seems that the MDP present in Panavia F2.0 is responsible for high bond strength to base metal alloys.[28,30,31] Unexpectedly, in the case of P, the shear bond values recorded for the specimens treated with AP either with sandblasting or nonsandblasting were lower than the specimens those treated with MP. These differences were not significant though.

The constituents of AP are VBATDT and MDP in acetone.

VBATDT monomer seems to interfere with the polymerization reaction of resin-based materials containing the benzoyl peroxide-amine as their initiator such as RU and P.[32,33] VBATDT contains sulfur group to promote adhesion to noble metal alloys but is not always effective for bonding to base metal alloys. The functional monomer of MP and MEPS which consists of similar functional groups (hydrogen phosphate) to MDP has a high affinity to the oxide layer produced on the surfaces of base metal alloys, contributing to a high and durable bond strength.[30,31] According to the results of the present study, for all resin cement, the shear bond strength was improved with usage of metal primers in conjunction with sandblasting which these changes were significant for P and RU. This finding is not in consistent with the results of Di Francescantonio *et al.* that they concluded the P can be used directly without a metal primer on the titanium surface.[34] They explained their results by chemical reaction of MDP with the metal oxides that we previously discussed. In the case of RU, combining of sandblasting and MP increased the shear bond strength significantly. In other cases, the shear bond strength improved by sandblasting before usage of metal primers, and these changes were not statistically significant though. Moreover, our

study exhibited the lowest results for RU self-adhesive resin cement. This result was in agreement with the result of Hitz *et al.*,[35] Hattar *et al.*,[36] and Xinyu and Xiangfeng.[37] RU is a Bis-GMA-based resin cement which may have low affinity to base metal alloys; in addition, the high filler content of RU causes a high viscosity and consequently low wetting ability of metal surfaces.[38] Obviously, the wettability of resin cement is a crucial factor for the improvement of bond strength.

GC is self-adhesive resin cement contained 4-MET as its functional monomer. 4-META monomer which has an aromatic acid anhydride functional group is effective in adhesion of cobalt-chromium or nickel-chromium alloys to resins.[28,39] In our study, GC exhibited an almost similar bond strength for all surface treatments. The low filler content and presence of 4-MET in GC seems are sufficient factors for promotion of bonding regardless the kind of surface treatment.

Regarding the failure mode, since in the current study, no cohesive failure was found, the obtained shear bond strength values can be considered as the representative of the cement/metal interfacial bond strength. Investigation of the mode of failure exhibited the highest mixed failure (100%) in the P (S + MP) group which showed the highest shear bond strength values. Studies described that the high fracture in the substrate as cohesive or mixed failure may be an indicative of better bond strength.[40-42] In our study, the lowest bond strength group with exception of RU (MP) tended to display adhesive failure. Similar results described by Al-Hana *et al.*[42] and Chung *et al.*[43] who reported the high adhesive failure mode in the lowest bond strength groups. On the other hand, Armstrong *et al.* explained that the cohesive fracture of substrate in either the tensile or shear bond strength tests cannot be attributed to the greater bond strength at the interface than of the strength of the adherend.[44]

Since our study was an *in vitro* study and did not attend clinical complications and oral environment such as pH changes and long-term water aging, careful interpretation have to be considered for clinical application. Further *in vitro* investigations and long-term clinical studies are required to confirm the result of this *in vitro* study.

CONCLUSION

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

1. Sandblasting is an important factor for improving the bond strength of resin cement to base metal alloys
2. Comparison of self-adhesive and self-etch resin cement revealed that the chemical composition has a crucial role in the performance of adhesive systems
3. The kind of metal primer does not have a significant effect on the bond strength, neither of the conventional nor for the new generation resin cement
4. The best bond strength of resin cement to base metal alloys can be achieved by sandblasting in conjunction with metal primers.

Conflicts of interest
The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or nonfinancial in this article.

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