



ORIGINAL ARTICLE

Influence of different surface treatments of nickel chrome metal alloy and types of metal primer monomers on the tensile bond strength of a resin cement



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KEYWORDS

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Abstract Objective: To evaluate the effect of different surface treatments of nickel chrome (NiCr) with the type of metal primer monomers on the conventional tensile bond strength (CTBS) of resin cement.

Methods: Forty disks of NiCr alloy were prepared for CTBS test and grouped as follows: group (1) no surface treatment (control group), group (2) oxide layer only, group (3) air abrasion, and group (4) air abrasion with an oxide layer. Each main group was subdivided into two subgroups ($n = 5$) depending upon the type of metal primer used for metal treatment. All specimens were bonded with resin cements. The CTBS was tested using a tensile testing machine. The data were statistically analyzed with One-way ANOVA, Two-way ANOVA, and T-test at 0.05 level of significance.

Results: Significant differences in the mean value of the CTBS between different surface treatments ($P \leq 0.05$) were observed. Tukey's test showed that air abrasion surface treatment had the highest mean value followed by the air abrasion with an oxide layer and oxide layer only. The control group showed the lowest value of significant difference compared to all treated groups ($P \leq 0.05$).

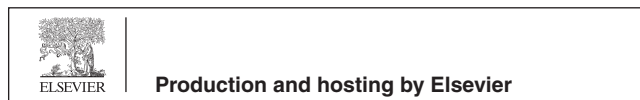
Conclusions: CTBS of self-adhesive resin cement to NiCr is dependent on surface treatment. Two types of the metal primer of different monomer contents 10-methacryloyloxydecyl dihydrogen phosphate (MDP) or Thiophosphoric methacrylate (MEPS) show similar behavior on the tensile bond strength.

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1. Introduction

Metal alloys have been used for the fabrication of fixed prosthesis, such as crowns, ceramometal restorations, and cast dowel with cores (Bottino et al., 2007; Eliasson et al., 2007). The clinical success of fixed restorations is strongly influenced by the success of the bond between the prosthesis and the tooth. Since high retention is obtained from the proper bond, it enhances marginal adaptation, reduces microleakage, and improves fracture resistance (Janda et al., 2007). Advancement of dental technology is associated with different efforts to initiate adhesion of the luting cement to the inner side of metal restorations (Tsujimoto et al., 2018). This would be useful to achieve durable retention of the restoration, particularly, when there is a shortness of the crown and/or tapering caused by preparation (Abreu et al., 2009). Up to a short time, the interaction with metal has not been obtained. This is because the luting agents have a minimum chemical affinity with metal (Dundar et al., 2007; Tanaka et al., 2007). In an attempt to increase the retention of luting resin cement to the metal alloy, different surface treatments have been proposed prior to the cementation procedure to create micromechanical and chemical interlocking. Clinically simple treatments that revealed an acceptable result are acid etching, air abrasion with aluminum oxide (Al_2O_3), and more recently, the treatment of the inner surface with different chemical components included the metal primer and combination between them (Freitas and Francisconi, 2004; Gurbuz et al., 2008; Ishii et al., 2008; Fonseca et al., 2012).

Metal primers contain active monomers incorporated within their composition which are either MDP (10-methacryloyloxydecyl dihydrogen phosphate) or MEPS (thio-phosphate methacrylate) derivatives. Monomers of the different chemical structure may perform a different action on the metal surface and subsequently, different prosthesis retention. Metals treated with primers are thought to be effective in improving the bond strength of resin cement because phosphate monomer contains a hydrophilic phosphate terminal end capable to form a chemical bond with an oxide layer created on the metal surface. On the other side, hydrophobic methacrylate terminal ends are bonded to the resin. Such an intimate bond will prevent penetration of water into adhesive interface which may lead to decrease hydrolysis (Abreu et al., 2009; Francescantonio et al., 2010; Griffin et al., 2010; Lisboa et al., 2006; Matsumura et al., 2011; Siqueira et al., 2016; Tsuchimoto et al., 2006).

Recently, dual cure self-etch self-adhesive resin cement was introduced as a new cement of newly developed multifunctional phosphoric-acid methacrylates. This type of cement does not have some of the drawbacks of traditional type of cement, such as zinc phosphate cement (Znp), glass ionomer cement (GIC), and resin-modified glass ionomer cement (RMGIC), and does not require any conditioning of the tooth surface. Studies explained that a newer version of phosphate resins could interact chemically with the treated surfaces of metals and improve the retention of restorations (Ferracane et al., 2011). Besides that, such cement could offer flowing advantages, better esthetic, high mechanical properties, more dimensionally stable and wear resistance. The micromechanical adhesion will improve the bond between the tooth and

the restoration (Braga et al., 2002; Radovica et al., 2008; Tsujimoto et al., 2017; Tsujimoto et al., 2018).

Alteration of the metal surface is an accepted method to get a higher bond. In addition, the chemical adhesion using different metal primers prior to their application may lead to further improvement of the bond strength and a combination between them. Due to the limited information on the relation between the inner surface of the metal prosthesis and resin cement luting agent to the bond strength, this research was designed.

The hypothesis to be tested in this research was that neither surface treatments of the metal nor type of metal primers would affect the tensile bond between the resin cement and metal alloy. The objective of this research was to evaluate the conventional tensile bond strength (CTBS) of the self-adhesive resin cement to the metal after different surface treatments and the application of two types of metal primers of different monomers.

2. Materials and methods

2.1. Specimen preparation

Forty disks of 9 mm diameter and 3 mm thickness were fabricated from nickel chrome (NiCr) alloy (Kera NH/Nickel Chromium alloy, Eisen bacher Dentalwaren ED GmbH, Germany; Ni 60%, Cr 25.80%, Mo 12.25%, Si 1.80%, Mn 0.03% C 0.01%) using conventional lost wax technique. The wax patterns were obtained by pouring modeling wax (T.P. Regular, Italy) into the copper band. The wax pattern was removed from the copper band and attached to the sprue. Casting ring was placed over the sprued wax pattern and filled with the investment material (Biosint-Supra, Degussa, Germany). The casting ring was inserted in a metal furnace (Manfredi 7C, BEGO, Italy) to burn out the wax pattern at 950 °C. NiCr alloy was melted at 1200 °C and forced into the mold space using centrifugal casting machine. Disks were cleaned and sandblasted using the sandblasting machine (perlsrahl 2, Degussa, Germany). Specimens were fixed on a glass slide and surrounded with a plastic tube of (15 * 15 mm), and then filled with self-cure resin (Self-curing, Vertex dental, Netherlands). The exposed metal surface was wet-polished with a 400, 600 grit silicon carbide abrasive disc using the (1000 MAX Milling machine/Bio-art, Brazil) to standardize the surface characteristic for all the samples.

2.2. Specimens treatments

Forty specimens were divided randomly to equal groups as follow:

Group 1: No surface treatment (Control group).

Group 2: Oxide layer only: in which the metal disks were subjected to oxide layer formation. Metal specimens were placed in a digitally calibrated furnace at 980 °C without a vacuum for 5 min to promote the formation of an oxide layer on the metal surface.

Group 3: Air abrasion: In this group, specimens were air abraded with 125 μ m aluminum oxide (Al_2O_3) for ten seconds under pressure of 80 Psi at 10 mm distance using a sandblasting unite (Microjato Removedor/Brazil).

Group 4: Air abrasion with oxide layer: specimens were treated as group 3 and then as group 2 to create oxide layer.

2.3. Bonding procedure

Each main group was subdivided into two subgroups ($n = 5$) depending on the type of metal primer used before the bonding procedure of resin cement. The used primer in this study was either Metal Primer II (GC Corp., Tokyo, Japan), composed of Thiophosphoric methacrylate (MEPS) and methylmethacrylate (MMA), or WP Metal prime (Willmann & Pein GmbH, Germany) containing 10-methacryloyl oxydecyl dihydrogen phosphate, Ethanol 96%, and water. Bonding area on the specimen was demarcated at the center of the metal disk surface using an adhesive tape with 4 mm circular hole. The metal primer was brushed on the demarcated area of the metal surface and allowed to be chemically reacted for 15 s. A translucent standardized plastic tube (4 * 4 mm) was fixed onto the bonding region of metal surface using sticky wax. The dual cure self-adhesive resin cement [TOTAL-CEM, Itena clinical, France, Matrix: UDMA (Urethane dimethacrylate), Bis-GMA (bisphenol Aglycidyl methacrylate), TEGDMA (Triethylene glycol dimethacrylate), Filler: Barium glass, Fumed silica] packaged in an auto dispensing device was mixed and incrementally filled inside a plastic tube in two layers of 2 mm thickness for each layer. The first layer was cured by a light cure device (LED.F, WOOD PECKER, China) with a light output of 1000 mw/cm² for 20 s from each side. To accommodate the CTBS, the second layer of the cement was applied, and a small metal screw with a ring head was embedded inside in the perpendicular position, and then cured as the first layer. The tested specimens were stored in distilled water at 37 °C for twenty-four hours and thermocycled for 300 cycles with a 30 s dwell time at a temperature ranging from 5 °C ± 2 °C to 55 °C ± 2 °C (Haselton et al., 2001).

2.4. Conventional Tensile Bond Strength Test (CTBS)

Each sample was gripped by a clamp of the tensile testing machine, and the ring was connected to the upper hook of the device (Tensile testing machine, SJX-500N-200 mm electric push pull test station 500N, Model; AEL.1000-400, China) as shown in Fig. 1. A tensile force was applied at a cross head speed of 0.5 mm/min. The CTBS was calculated by dividing the load of failure over the surface area of the bonded surface. The data were recorded in Mega Pascal (MPa).

2.5. Statistical analysis

The collected data were analyzed statistically using the SPSS software (version 19.0; SPSS Inc., Chicago, IL, USA). One-way ANOVA was applied to determine the significant difference between all different surface treatments. Two-way analysis of variance (ANOVA) was applied to determine the significant differences between tested groups (surface treatment and type of primers), then followed by Tukey's Post hoc test to compare the significant groups. Student T-test was applied to evaluate the differences between the two tested

groups of metal primers. P -values of ≤ 0.05 were considered statistically significant.

2.6. Failure mode evaluation

The cement-metal interface of each fractured sample was examined at 20X magnification under a stereomicroscope (Hamilton Altay, Italy). Images were captured by a computer program, then examined visually by two observers to detect the failure modes. The mode of failure was recorded as either: (1) adhesive failure when cement dislocate from the metal (Fig. 2), (2) cohesive failure when fracture occurs within the resin cement material and a thin layer of cement adhered to the metal (Fig. 3) or (3) mixed failure areas of cohesive and adhesive failure, part of the cement adhered to the metal (Fig. 4).

3. Results

The mean and standard deviation of the resin cement's bond strength to NiCr metal following different surface treatments and primer types of application are listed in Table 1.

For both primers, One-way ANOVA showed that there was a significant difference in the mean values of CTBS between all different surface treatments ($P \leq 0.05$) (Tables 2 and 3).

Tukey's Post hoc test showed that air abrasion surface treatment had the highest mean value among the different surface treatments followed by air abrasion with an oxide layer and the oxide layer only, while no surface treatment showed the lowest mean value (Table 1).

Two-way ANOVA showed that there was a significant difference in the mean value of CTBS for both parameters primer types and surface treatments ($P \leq 0.05$). However, there was no significant interaction between them ($P > 0.05$) (Table 4).

Independent T-test showed that there was no significant difference in the value of CTBS between two primers for all groups ($P > 0.05$) except in air abrasion with an oxide layer group, where metal primer II (12.62 MPa) had a significantly higher mean value than WP metal prime (11.32 MPa) in which the difference between these mean values was 1.3 MPa (Table 5).

Different modes of failure recorded and their percentages for all groups are shown in the Table 6.

4. Discussion

The success of the bond between the metal prosthesis and the resin cement depends upon the combination of metal treatment, metal primer, and resin cement (Hill and Lott, 2011; Raeisosadat et al., 2014). In this research two parameters that affect metal/resin bonding were evaluated.

The commonly applied surface treatments including air abrasion, oxide layer formation or combination of them and different types of active monomers included in metal primers were examined. For WP Metal prime, the monomer was 10-methacryloyloxydecyl dihydrogen phosphate (MDP), and Thiophosphoric methacrylate (MEPS) for Metal Primer II.

CTBS test is one of the methods used to determine the adhesive strength of bonding resin cement to the metal used in the fabrication of the prosthesis. The results did not support the hypothesis of our research because the different metal sur-

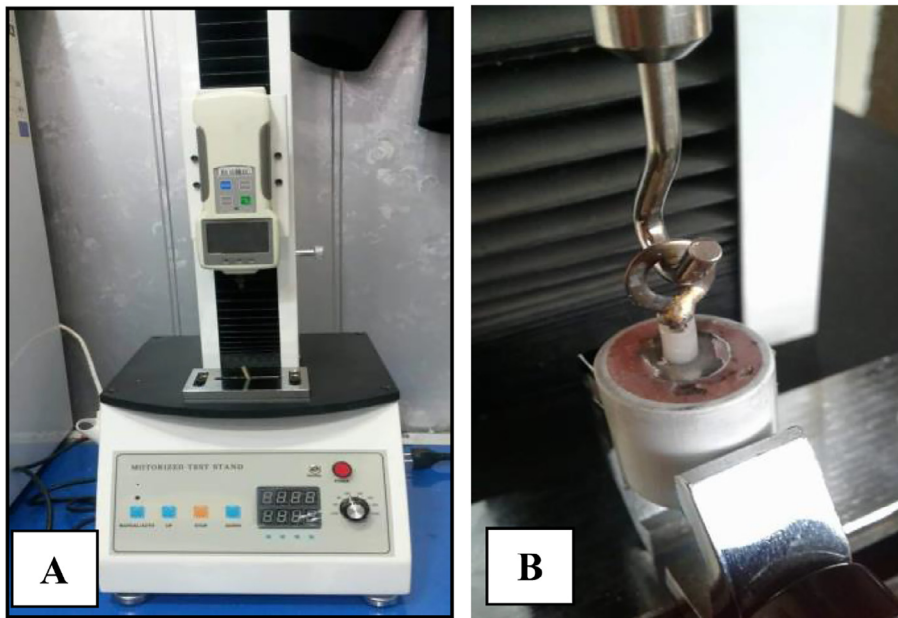


Fig. 1 Tensile bond strength. (A) Tensile testing machine. (B) Tested specimen.



Fig. 2 (Original magnification $\times 20$) Resin cement shows adhesive failure.

face treatments influenced the tensile bond of resin cement to the metal alloy.

The highest mean value of CTBS was recorded in the air abrasion group followed by air abrasion with an oxide layer group in both primers. Several studies tested the influence of air abrasion with aluminum oxide particle with a similar result. This may be related to the fact that such treatments will transform the surface of metal from smooth to rough micro-retentive texture. However, increasing the metal contact surface area permits the incorporation of the resin into the micro porosities of surface metal. Following resin polymerization

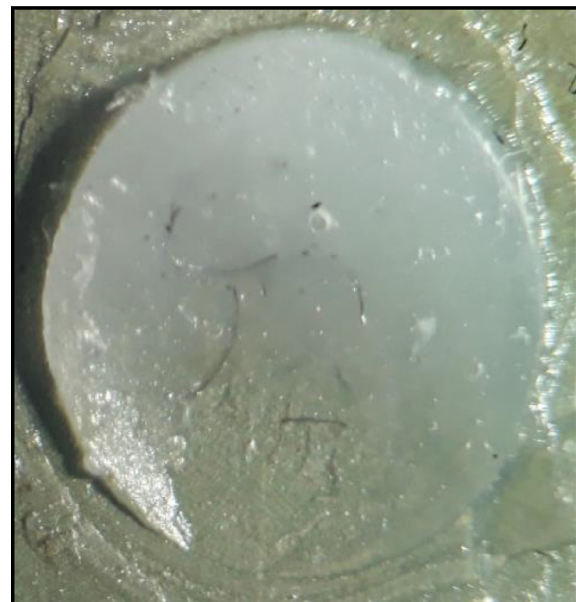


Fig. 3 (Original magnification $\times 20$) Resin cement shows Cohesive failure.

within undercuts, a micromechanical bond will be accomplished and subsequently, will make the dislodgment between cement and metal more difficult (Petridis et al., 2004).

Additionally, air abrasion removes the possible impurities, such as: oil, unfavorable oxides, smear product, and other contaminants, from the surface to improve cement wettability specially when proceeded with priming procedure (Gargari et al., 2010; Haneda et al., 2009; Pazinato et al., 2006; Rodríguez et al., 2010; Yucel et al., 2018). Freitas and Francisconi (2004) showed that air abrasion group with $100\ \mu\text{m}$ aluminum oxide presented the highest values of bond strength, while the group that had no surface treatment presented low values.

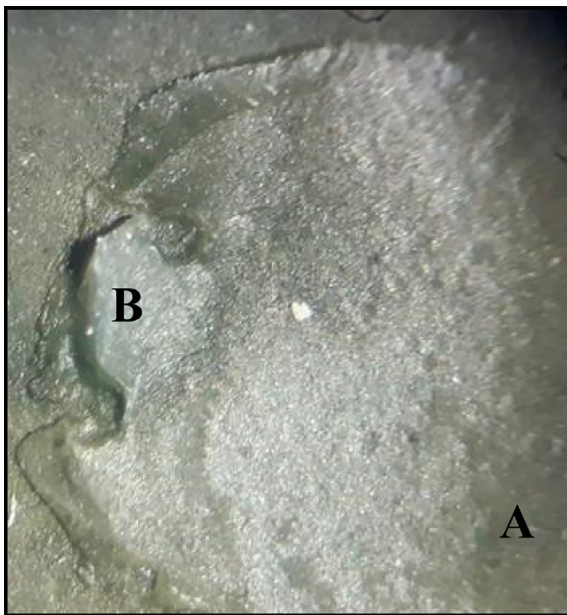


Fig. 4 (Original magnification $\times 20$). Resin cement shows mixed failure (A) Metal surface. (B) Resin cement.

Table 1 Mean, standard deviation of the *conventional* tensile bond strength for the different surface treatments for both Metal primers.

	Group	Mean (Mpa)	SD
Metal primer II	Group1 D	3.67	0.33
	Group2 C	4.82	0.51
	Group3 A	15.44	0.39
	Group4 B	12.62	0.40
W&P metal prime	Group1 D	3.15	0.49
	Group2 C	4.39	0.34
	Group3 A	14.92	0.53
	Group4 B	11.32	0.67

SD = Standard deviation, Number of samples = 5.
Different letters are statistically significantly different according to Tukey's test.

Although our results showed a significantly low bond in the oxide layer group compared to the abrasion one, the result of the oxide layer group still has an advantage over no surface treatment group (control). This may be related to the nature of an oxide layer which provides a reactive surface for enhancing chemical interaction of the primer monomer and resin cement monomer as well. The oxides formed on the metal surface have an important role in wettability, resulting in the formation of chemical bonds with resin cements (El-Guindy et al., 2010). In addition, the presence of the oxide layer will serve to rough the surface of the metal and give some micromechanical retention (Abreu et al., 2009).

A significant decrease in the value of CTBS of an abraded group when exposed to oxide layer formation for both primer types could be explained that with the creation of oxide layer over the abraded metal surface, a thin layer of impurity will be precipitated over the metal surface. Such surface changes will have a detrimental effect on the possibly formed micro undercuts within the metal, leading to the decrease in the surface area available to the resin materials and consequently, less interlocking between resin cement and the metal surface.

Generally, no significant differences were recorded in CTBS between two types of primers in non-surface treated and treated groups (oxide layer, air abrasion). Except in air abrasion with an oxide layer group, the Metal Primer II showed a significantly higher mean value than WP Metal prime. These results might be related to the monomer reaction portions of both primers. Both primers have active monomers containing phosphoric acid derivatives. It has been reported that this type of monomers chemically reacts with the metal surface, even with the existence of the oxide layer, leading to improving the bonding of resin cement to the metal surface (Ferracane et al., 2011; Gargari et al., 2010; Rodríguez et al., 2010).

The results agree with many researchers who concluded that the use of air abrasion with phosphate based primers together achieve long-term durable bond to the metal because metal primers improve the wettability of resin cement to air-abraded substrate surface (Abi-Rached et al., 2012; Al-Heou and Swed, 2016; Fonseca et al., 2012; Griffin et al., 2010; Özcan et al., 2008; Rodríguez et al., 2010; Yeon Yun et al., 2010; Yucel et al., 2018).

Table 2 One-way ANOVA of the conventional tensile bond strength for all different surface treatments with Metal primer II.

	Sum of squares	df	Mean square	F-value	P-value
Between the four groups	501.988	3	167.329	966.370	0.000*

* Significant differences, df = degree of freedom.

Table 3 One-way ANOVA of the conventional tensile bond strength for all different surface treatments with WP Metal prime.

	Sum of squares	df	Mean square	F-value	P-value
Between the four groups	473.137	3	157.712	567.402	0.000*

* Significant differences, df = degree of freedom.

Table 4 Two-way ANOVA of the conventional tensile bond strength between primers and surface treatments.

Variable	Sum of squares	df	Mean square	F	Sig.
Primers	4.851	1	4.851	21.508	0.000*
Treatments	973.864	3	324.621	1439.219	0.000*
Primers × Treatments	1.261	3	0.420	1.864	0.156

* Significant differences, df = degree of freedom.

Table 5 Independent samples *t*-test of conventional tensile bond strength for all groups between two primers.

	Surface treatment	t	df	SE	P-value
Tensile Bond Strength	Group 1	1.96	8	0.26	0.084
	Group 2	1.54	8	0.27	0.161
	Group 3	1.76	8	0.29	0.116
	Group 4	3.71	8	0.35	0.006*

* Significant difference, SE = Standard Error, df = degree of freedom.

Table 6 The modes of failure percentages for all groups.

	Mode of failure groups	Cohesive	Adhesive	Mixed
Metal primer II	Group 1	5%	80%	15%
	Group 2	15%	70%	15%
	Group 3	83%	7%	10%
	Group 4	68%	20%	12%
	Group 1	0%	75%	25%
WP Metal prime	Group 2	10%	65%	25%
	Group 3	75%	10%	15%
	Group 4	68%	15%	17%

The mode of failure (Table 6) showed that the predominant failure mode was an adhesive failure at the cement-metal interface for control and oxide layer groups for both primers which corresponded the weakest bond strength. This result agrees with Abreu et al., 2009 who reported the samples with a higher incidence of adhesive failure than cohesive in the oxide layer only group.

The specimens treated with air abrasion and air abrasion with oxide layer for both types of primers resulted in cohesive and mixed failure. These results explained that the bond at the cement-metal interface was sufficiently strong enough to prevent the failure at this level. These results agree with (Antoniadou et al., 2000; Abreu et al., 2009) who concluded that cohesive and mixed failure was the prevalence mode of failure in the specimens treated with air abrasion and a metal primer containing phosphate groups. However, the microscopic mode of failure analysis was in agreement with the CTBS values.

5. Conclusions

The conventional tensile bond strength (CTBS) of self-adhesive resin cement to NiCr metal alloy is surface treatment dependent. Two types of the metal primer of different monomer contents (MDP or MEPS) show similar behavior on the tensile bond strength.

Conflict of interest

The authors declare that there are no conflicts of interest.

Acknowledgments

None.

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