BJOS
Brazilian Journal of Oral Sciences

Volume 18
2019
e191406

Do metal alloy primers increase the bond strength of orthodontic tubes?

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Received: September 13, 2018
Accepted: September 11, 2019

Aim: To evaluate the bond strength (BS) and failure mode of orthodontic tubes treated with different alloy primers at the interface among enamel, resin and orthodontic tubes. **Methods:** Orthodontic tubes were bonded to the enamel of 80 bovine incisors with the orthodontic resin (Transbond XT, 3M Unitek). Prior to bonding, the tubes were chemically treated with (n=20) Metal/Zirconia Primer (MZ, Ivoclar), Scothbond Universal (SB, 3M Espe); Orthoprimer (OP, Morelli) or left untreated (Control - C). Specimens were submitted to 5,000 thermal cycles (5 and 55° C) to age the bonded interface. A shear BS test and failure modes were conducted, and the results were analyzed using one-way analysis of variance and Fisher's exact test, respectively. **Results:** No differences were observed among groups regardless of the type of alloy primer used ($p = 0.254$). However, no differences were observed among the failure modes of the groups tested ($p=0.694$). The adhesive failure mode between the resin and enamel was the most prevalent failure (45%) for groups OP and C, whereas cohesive failure in the orthodontic resin was the most prevalent failure (40%) for groups SB and MZ. **Conclusion:** Alloy primers were unable to increase the BS of the orthodontic tubes to enamel.

Keywords: Shear strength. Dental bonding. Materials testing. Alloys. Orthodontics.

http://dx.doi.org/10.20396/bjos.v18i0.8657247

Introduction

The use of tubes as an option for orthodontic treatment was responsible for a major advancement in orthodontics, and it was possible due to improvements in adhesive systems and orthodontic composite resins, which provide reliable and long-term enamel adhesion[1]. The orthodontic tubes offer advantages over traditional systems, such as orthodontic bands, as they facilitate hygiene, eliminate the need for spacers, and decrease the possibility of periodontal disease and secondary carious lesions[2].

To be effective, tubes must be able to withstand the forces that orthodontic mechanics generate, as well as masticatory forces. In addition, they should present a shear bond strength (BS) greater than 8 MPa, which is the minimum value necessary to maintain enamel adhesion under such circumstances[3]. However, under clinical conditions, enamel adhesive failures leading to the debonding of orthodontic tubes can occur[4-6]. In fact, debonding is the reason for many failures, causing 66% of total failures in molars[7]. Adhesive failures are frequently associated with complications with the bonding technique, moisture contamination, occlusal contacts, the presence of aprismatic enamel, and changes in the enamel-etching pattern[8]. Although the tubes can be replaced or submitted to a new bonding procedure, these procedures involve additional chairside appointments, which compromises the treatment progress.

The interface between the orthodontic tube and enamel is not the only site susceptible to failure. Debonding can also occur between the tube and the orthodontic resin, and although a number of treatments are described to improve adhesion to enamel[4-6], only a few are proposed to increase bonding on the metal surface. Mechanical treatments designed to increase roughness, such as aluminum oxide blasting, the use of a diamond drill[9], and chemical treatment with the use of silane[10], are amid the reported alternative treatments for the metal surface.

Base metal alloys containing chrome in their composition (Ni-Cr, Ni-Cr-Be, Co-Cr) can oxidize when in contact with atmospheric air, forming a passivation layer[11]. Some adhesive monomers that can chemically bond to the oxide film on the metal surface have been incorporated into resin cements with the goal of improving bonding across the interface. Therefore, bonding between adhesive monomers and alloys is the result of a micromechanical interocking promoted by surface roughness, and a chemical interaction between the oxide film of the metal surface and the carboxylic of phosphate acidic monomer of the resin cements[12]. With the aim of overcoming the absence of adhesive monomers in the composition of resin cements, metal alloy primers were developed to be applied on the surfaces of alloys prior to cementation[13]. Previous, studies have demonstrated alloy primers' ability to improve bonding between polymer-based materials and metallic surfaces[14,15].

The first monomer to be used for adhesion to metal was 4-META (methacryloyloxyethy trimellitate anhydride), which was designed to eliminate the need for mechanical retention and increase adhesion[16]. Later, monomers containing either phosphoric or carboxylic acid groups, such as 11-methacryloxyunden, decarboxylic acid (MAC10), 10 methacryloxydecyl dihydrogen phosphate (10 MDP), vinylbenzyl-n-propylamino-triazine-dithiol (VBATDT), and 6-methacryloxyhexyl-2-thiouracil-5-caboxylate (MTU-6), were synthesized[14-16]. The primers applied on the surface of the metal may contain MDP, VBTDT, MTU-6, MAC-10, or the combination of these monomers. The functional monomer of 10-MDP is able to react chemically with the chromium oxide of the casting surface to promote adhesion[14,16]. MDP has two functional groups: One is a divalent phosphoryl group that chemically bonds to the metal atoms of the metal surface, and the other is a methacryloyl group that copolymerizes with resin monomers either in the adhesive or in the resin cement composition[14,16]. Recently, "universal "or "multi-mold" adhesives containing both silane and a functional monomer have been developed and are indicated to bond to metal surfaces. However, these universal adhesives' ability to bond to metal alloys is still being debated[16].

Since alloy primers are indicated for metal surfaces, the potential of increasing the BS of orthodontic tubes seems to be promising[17]. However, the literature is scarce in studies evaluating the use of primers in metal orthodontic devices. Therefore, the aim of this study was to evaluate the BS and failure mode at the interface among enamel, resin, and orthodontic tubes treated with various metal primers. The null hypothesis was that no differences would be found in the BS and failure mode among groups.

MATERIALS AND METHODS

Experimental design

The experimental units consisted of 80 bovine incisors, and orthodontic tubes were bonded to the enamel buccal surfaces. Before bonding, the orthodontic tubes received (n=20) two alloy primers (Metal/Zirconia Primer – *MZ* and Orthoprimer - *OP*) and one multi-mode adhesive (Scothbond Universal – *SB*). The control group was left untreated (n = 20). The response variables were shear BS (in MPa) and the failure mode of the deboned area. The commercial name, manufacturer, and composition of the alloy primers are shown in Table 1.

Table 1. Composition of the metal primers and orthodontic composite.

Commercial name and manufacutrer	Composition
Metal/Zirconia primer (MZ, Ivoclar Vivadent, Schaan, Liechtenstein)	Methacrylate of phosphonic acid and methacrylate cross-linked in organic solvent.
Scotchbond Universal (SB, 3M/ESPE, St. Paul, MN, USA)	BIS-GMA, HEMA, silane treated silica, water, ethanol, decamethylenedimethacrylate,10-decanediol phosphate methacrylate, acrylic copolymer and itaconic acid, camphorquinone, N, N-dimethylbenzocaine, 2-dimethylamonoethyl methacrylate, methyl ethyl ketone.
Orthoprimer (OP, Morelli, Sorocaba, Brazil)	Bis-GMA, TEG-DMA, HEMA, DMPT, camphorquinone, hydroxytoluenebutylated, dimethyl aminoethyl methacrylate.
Transbond XT (3M-Unitek, Monrovia, USA)	Primer: Bisphenol A diglycidyl ether dimethacrylate, TEG-DMA, triethylene glycol dimethacrylate, triphenylantimonium, 4- (dimethalamino) -benzethanol, d-1-camphorquinone, hydroquinone. Resin: Bis-GMA, bisphenol A bis (2-hydroxyethyl ether) dimethacrylate, quartz treated silane, silane treated silane, silanodimethacrylate, Diphenyliodonium hexafluorophosphate

BIS-GMA- bisphenolglicedyl methacrylate; TEG-DMA-triethylene glycol dimethacrylate; HEMA-hydroxyethyl methacrylate; DMPT-dimethyl-p-toluidine.

Specimen preparation

Eighty bovine incisors crowns with enamel free of defects or cracks were selected and were stored in a 0.1% thymol solution for 24 hours. After debridement and pumicing, the teeth were embedded in polystyrene resin with the buccal surfaces facing up and sonicated.

Enamel bonding was performed according to the manufacturer's instructions for the orthodontic composite (Transbond XT). Teeth were cleaned with water spray (15 s), air dried (15 s), and acid etched with 37% phosphoric acid gel for 30 s. The surface was rinsed with air-water spray for 20 s and air dried for 10 s. The primer of the Transbond XT system was applied on the enamel surface, then sprayed with a mild air spray for 5 s and light cured for 20 s. The orthodontic composite (Transbond XT) was applied on the inner surface of the tube and fixed on the enamel. Light curing was performed for 40 s (20 s in the mesial and 20 s in the distal sites) using a LED light-curing unit (Bluephase, Ivoclar, Liechtenstein, with irradiance of 1200 mW/cm^2).

Before the tube was bonded to the enamel surface, the base of the orthodontic tube was treated according to each experimental group:

- *MZ*: The primer was applied on the base of the tube (180 s) and air dried (5 s).
- OP: The primer was applied on the base of the tube (180 s) and air dried (5 s).
- *SB*: The universal adhesive was applied on the base of the tube (20 s) and air-dried (5 s).
- *Control:* No treatment was performed on the tube.

Thermal cycling

The samples were stored for 24 h in distilled water at 37 ºC, and for the purpose of aging the bonding interface, 5,000 thermal cycles were performed (MSCT-3, Marcelo Nucci ME, São Carlos, Brazil) at 5 and 55 °C (± 1°C) with a dwell time of one minute each.

Shear bond strength test

Forty-eight hours later, specimens were submitted to a shear test in the occlusal-cervical direction, with the blade placed at the enamel and resin/tube interface. The tests were performed in a universal testing machine (EMIC- DL 2000, Instron Brasil Scientific Equipment LTDA, São José dos Pinhais, Brazil) with a load cell of 1kN at a crosshead speed of 0.5 mm/min. The maximum force (N) up to failure was recorded. The shear BS (in MPa) was calculated from the force and the bonded area of the tube to the enamel surface.

Failure mode

The failure mode of the debonded interface was observed under a stereomicroscope at 40x magnification. Debonding was classified (Table 2) based on a previous report[18].

Table 2. Failure mode classification

Standard	Type of Failure
1	Adhesive failure between orthodontic composite and the base of the orthodontic tube (100% of the composite remains on enamel surface)
2	Cohesive failure in the orthodontic composite (50% of the composite remains at the base of the orthodontic tube and 50% bonded on enamel)
3	Adhesive failure between orthodontic composite and enamel (100% of the composite remains on the tube)
4	Cohesive enamel fracture

Statistical analysis

Shear BS data were submitted to exploratory analysis to verify normality and homoscedasticity, and they were also submitted to parametric one-way analysis of variance (ANOVA). The fracture mode was analyzed using Fisher's exact test. In all analyses, SAS software (SAS Institute Inc., Cary, NC, USA, Release 9.2, 2010) was used considering the level of significance of 5%.

RESULTS

No significant difference was found among groups in terms of shear BS values (p = 0.254) as observed in Table 3. Additionally, no significant difference (p = 0.694) was observed in the failure mode distribution of the adhesive interface as a function of the treatments. The most common failure mode was type 3 (adhesive failure between the orthodontic composite and enamel), in which no orthodontic composite remnant was found in the enamel (45% of the experimental units). The least prevalent failure was type 1 (only in the SB group), in which the base of the orthodontic tube did not exhibit a resin remnant and all of the resin remained on the enamel (1.2% of the experimental units) (Table 3).

Table 3. Mean and standard deviation of shear bond strength and distribution of the failure mode according to treatments.

Group	Shear BS	Failure mode			
		Type 1 (Adhesive orthodontic composite/tube)	Type 2 (Cohesive in orthodontic composite)	Type 3 (Adhesive orthodontic composite/enamel)	Type 4 (Cohesive in enamel)
MZ	13.64 (6.24) a	0 (0%)	11 (55%)	5 (25%)	4 (20%)
SB	12.26 (5.38) a	1 (1.25%)	11 (55%)	6 (30%)	2 (10%)
OP	13.15 (4.18) a	0 (0%)	4 (20%)	13 (65%)	3 (15%)
Control	10.40 (5.70) a	0 (0%)	6 (30%)	12 (60%)	2 (10%)
Total		1 (1.25%)	32 (40%)	36 (45%)	11 (13.75%)

MZ - Metal/Zirconia Primer; SB - Scotchbond Universal; OP – Orthoprimer; N- number of samples per group. Means followed by the same letter indicate no statistical differences. No differences were observed in failure modes among experimental groups, according to Fisher's test (p = 0.1119).

DISCUSSION

The BS results showed no difference among the orthodontic tubes that were treated with various primers, even those containing acidic phosphate monomers (SB and MZ). Dias et al.[19] observed that the application of an alloy primer (Kuraray) did not increase the BS of the resin cement to zirconia, and the primer did not prevent BS decrease after six months of water storage. The alloy primer contains two functional acidic monomers VBATDT (vinylbenzyl-n-propylamino-triazine-dithiol) and 10-MDP in an acetone-based solution, and it is indicated to cement metal fixed prosthodontics structures.

Similarly, it was previously reported that an Orthoprimer application to polycarbonate-based brackets did not influence the BS results[15], which is comparable to the findings of our study. The Orthoprimer agent does not contain acidic phosphate monomers except for methacrylate monomers and non-phosphoric hydrophilic monomers (TEGDMA e HEMA) (Table 1). Therefore, it is speculated that this primer is responsible for increasing wettability and improving the resin permeation of the composite in the irregularities of the base of the orthodontic tube. However, it does not chemically bond to the alloy surface as the function monomer does.

Contrary to the present results, Cal Neto et al.[20] observed that the application of an alloy primer with the acidic monomer 4-META (4-methacryloxyethyl trimellitic anhydride) increased the BS between the composite and the metallic brackets 48% compared with the control group, whereas in our research, alloy primers were able to increase the BS 17.9 to 31.2%. It should be noticed that these authors used a different primer (4-META) and that this agent could have been more effective in increasing BS compared with the primers selected in this research. The absence of thermal cycles to age the bonded interface, and the fact that the authors used human pre-molars instead of bovine incisors cannot be ruled out.

In a previous study[20], it was observed that treating the surface of zirconia with alloy primers increased the BS of the zirconia to the metallic bracket. That study used Z-Prime Plus (10-MDP and carboxylic acid) and the Zirconia Liner premium (silane with MDP)[20]. The authors credited the good performance of Z-Prime Plus to the presence of MDP and the ability of the primer to co-polymerize with the resin monomers, as the functional group binds to the metallic oxide of the substrate via the phosphoric group[21]. It is important to notice that MZ, SB, and Z-Prime Plus exhibit different acidic phosphate monomers: the phosphonic acid methacrylate, 1,10-decanediol phosphate methacrylate, and 10-methacryloyloxydecyl dihydrogen phosphate, respectively. Thus, different monomer compositions and application modes may influence BS results[22]. Furthermore, the presence of organophosphate monomers in the universal adhesive SB could promote the instability of the silane component[22]. Therefore, it is possible that the presence of silane hampered the monomer performance and that the adhesive presented similar BS results compared with the other primers.

Thermal cycling (5,000 cycles) was performed for all groups after bonding to age the interface. Previous studies observed that MDP-based primers were effective in preserving the bonding stability to zirconia even after thermal cycling[22-24]. On the other hand, Imai et al.[25] observed that the BS between composite and alloy surfaces decreased after 20,000 thermal cycles. The authors suggested that the lower BS could have been the result of water infiltration in microgaps between the composite and the alloy[25]. As no differences were observed in our study, it is possible that the number of cycles performed could have been insufficient for influencing the BS.

No significant differences were found among the failure modes. In the current study, the shear BS test evaluated multiple interfaces—enamel, orthodontic resin, and the base of the orthodontic tube. Numerically, the most predominant failure was the adhesive type, between the enamel and orthodontic resin (45%). This may be an indication that the BS at the base of the orthodontic tube where the primer was applied was acceptable. In addition, it should be kept in mind that in shear bond tests, the knife of the apparatus slides down the bordering enamel, stressing the orthodontic resin more than the orthodontic tube. Therefore, as expected, the second more prevalent failure was cohesive failure in the orthodontic composite (40%). The least prevalent failure was the adhesive type between the orthodontic composite and the base of the orthodontic tube (1.25%), which might indicate that although the primers did not improve the shear BS, they did not compromise it. In addition, the fact that no differences were found among the BS and failure modes implies that the irregularities at the base of the orthodontic tubes were sufficient for promoting a reasonable BS regardless of the presence of primers.

MZ, OP, and SB exhibited 15%, 20% and 10% respectively, of enamel cohesive defects. Although this type of failure indicates an acceptable performance of the primers applied to the metal, enamel fracture is undesirable when the tubes are removed at the end of orthodontic treatment. Therefore, because no differences were observed among groups, we believe that the decision to apply primers to metallic surfaces should be reexamined, as it could cause enamel fracture during debonding.

Based on the above, the null hypothesis could be accepted, as the application of alloy primers to the base of the orthodontic tubes did not increase the BS to the enamel surface and did not influence the failure modes of the tubes bonded to the enamel surface. Moreover, based on the failure modes and the existence of enamel cohesive failures when primers were applied, we endorse that no need exists to apply alloy primers to orthodontic tubes.

Considering that the application of alloy primers to orthodontic tubes can be dismissed and that debonding occurs due to adhesives (enamel/orthodontic resin) at the orthodontic resin, future research should focus on developing an orthodontic resin that will not fail cohesively and that possesses optimal enamel adhesion that will resist shear forces without compromising enamel integrity when the tube is removed at the end of the orthodontic treatment.

In conclusion, the application of alloy primers did not increase the BS of the orthodontic tube to the enamel. In addition, its application should be better evaluated, as enamel fracture could occur during debonding.

CONFLICT OF INTEREST STATEMENT

The authors do not have any financial interest in the companies whose materials are included in this article.

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