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CPP–ACP pretreatment effect on microshear bond strength of simplified etch-and-rinse adhesive systems plus a flowable composite to enamel

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This study evaluated the effect of a casein phosphopeptide–amorphous calcium phosphate based paste (MI Paste) on the microshear bond strength (MSBS) of different simplified etch-and-rinse adhesive systems on enamel. Roots were removed from 90 bovine incisors, and the facial enamel was ground flat. Teeth were randomized into nine groups, according to the enamel pretreatment (without or with application of MI Paste for 1 or 2 min) and the adhesive system used (Single Bond, Stae, or Ambar). Composite cylindrical blocks (2 mm height × 1 mm diameter) were built, stored in water for 24 h, and subjected to the microshear test in a universal testing machine with a load of 0.5 mm/min. Failure modes were analyzed using scanning electron microscopy. Data were submitted for statistical analysis by a two-way ANOVA, followed by multiple comparisons with Tukey test ($\alpha = 5\%$). The Ambar group presented the highest MSBS values. Pretreatment with MI Paste for 2 min improved bonding in the Single Bond and Stae groups. In all groups, adhesive failures were most frequent. In conclusion, Ambar presented better performance and did not need any pretreatment. The enamel MSBS of the other simplified adhesive systems was improved by the pretreatment with MI Paste, depending on the time of application.

Keywords: CPP–ACP; enamel; simplified etch-and-rinse adhesive systems; microshear bond strength

Introduction

The long survival of resin composite restorations requires an adequate bonding procedure.[1] Despite the good performance of adhesive systems, enamel is susceptible to fluid infiltration into the dentin in enamel/dentin cavities, which is related to caries progression. Improving enamel adhesion could favor higher retention of composite restorations in deep cavities where dentin residual caries may remain into the cavity. In fact, the presence of residual dentin caries becomes the cavity sealing dependent on a strong enamel adhesion.[2,3] Casein phosphopeptide–amorphous calcium phosphate

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(CPP–ACP) has been used in various strategies for preventing caries. CPP–ACP optimizes enamel remineralization, increasing calcium and phosphate levels in the biofilm.[4,5] The CPP–ACP-based MI Paste/Tooth Mousse (GC Corp.) can be used to promote remineralization and to protect enamel surfaces.[6–8] Application of MI Paste has been associated with improved enamel bond strength [5,6] and may improve the performance of adhesive systems.

To reduce chair time, clinicians prefer to use simplified etch-and-rinse adhesive systems.[9] However, the shortest clinically relevant time for enamel treatment with MI Paste associated with the use of simplified etch-and-rinse adhesive systems has not been well established. Some authors have employed 3 min as the treatment time.[6] With the possibility of lower clinical treatment time, this study aimed to evaluate whether the microshear bond strength (MSBS) values between simplified etch-and-rinse adhesive systems and enamel are influenced by the duration of enamel pretreatment with MI Paste or by the commercial type of adhesive system used. The null hypotheses were that the MSBS would show no differences when the enamel was pretreated with MI Paste for different times or when different commercially available simplified adhesive systems were used.

Material and methods

Experimental design

This *in vitro* study evaluated MSBS to bovine enamel as regards the following two factors: enamel pretreatment (at three levels: none/control; CPP–ACP/1 min; CPP–ACP/2 min) and applied simplified etch-and-rinse adhesive system (at three levels: Single Bond, Stae, Ambar). The chemical components of the materials used in this study are described in Table 1.

Table 1. Details of materials tested in this study.

Material	Type	Manufacturer	Composition by weight (%)
MI Paste	CPP–ACP-based paste	GC Corp., Tokyo, Japan	Glycerol (10–20), CPP–ACP (5–10), D-sorbitol (0–5), propylene glycol (0–2), silicon dioxide (0–2), titanium dioxide (0–2)
Adper Single Bond	Two-step etch-and-rinse adhesive system	3 M ESPE, St Paul, MN, USA	Ethyl alcohol (25–30), silane treated silica (nanofiller) (10–20), Bis-GMA (10–20), HEMA (5–10), glycerol 1,3-dimethacrylate (5–10), copolymer of acrylic and itaconic acids (5–10), water (<5), diurethane dimethacrylate (1–5)
Stae	Two-step etch-and-rinse adhesive system	SDI, Victoria, Australia	Acetone (54), acrylic monomer (44), balance ingredient (nontoxic) (2)
Ambar	Two-step etch-and-rinse adhesive system	FGM, Joinville, SC, Brazil	UDMA (5–40), HEMA (5–40), methacrylate acidic monomers (1–20), methacrylate hydrophilic monomers (5–40), silanized silicon dioxide (<1), camphorquinone (<1), 4-EDAMB (<1), ethanol (<20)

Notes: CPP–ACP, casein phosphopeptide–amorphous calcium phosphate; Bis-GMA, bisphenol-glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; UDMA, diurethane dihydrogen phosphate; EDAMB, ethyl 4-dimethylaminobenzoate.

Preparation of specimens

Ninety similar bovine incisors were selected, cleaned, and stored in a 0.5% chloramine T solution at 4 °C. Roots were removed with a double-faced diamond saw (KG Sorensen, Cotia, SP, Brazil). Each crown was embedded in polystyrene resin to facilitate handling. Enamel was wet-ground with 400- and 600-grit SiC abrasive papers in an APL4 polishing machine (Arotec, Cotia, SP, Brazil).

Specimens were randomized and allocated into nine groups, according to the pretreatment time of the enamel surface (without or with MI Paste pretreatment for 1 or 2 min) and adhesive system (Single Bond, Stae, or Ambar). MI Paste was applied and the enamel was rinsed with water for 10 s and air-dried. Acid etching was performed for 15 s with 37% phosphoric acid (Condac 37, FGM, Joinville, SC, Brazil), followed by washing (30 s) and drying with absorbent paper. Two coats of each adhesive system were applied with a microbrush. Each adhesive layer was gently air-dried, allowing solvent evaporation before photoactivation for 10 s with a light-emitting diode (Coltolux, Coltène, Switzerland; 1264 mW/cm^2).

A cylindrical translucent Tygon mold (Tygon Tubong, TYG-030; Saint-Goubain Performance Plastics, Maime Lakers, FL, USA) with 1-mm internal diameter and 2-mm height was positioned over the specimens. A flowable resin (Opallis Flow A2 shade, FGM) was inserted into the mold. The resin was light cured for 20 s. The mold was removed after 1 h and the specimens were stored in deionized water for 24 h.[10]

Microshear test

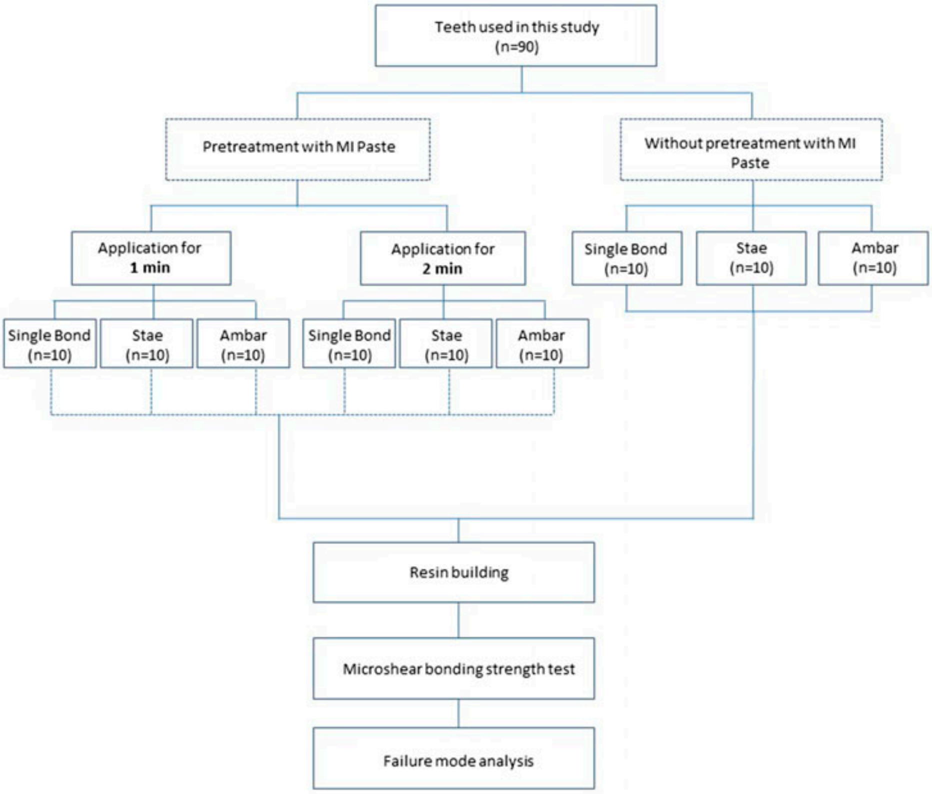
After storage, the specimens were subjected to the microshear test in a universal testing machine (EMIC DL 2000, São José dos Pinhais, PR, Brazil). Specimens were attached to the testing device by looping a thin steel wire around each cylinder. Load was applied at 0.5 mm/min until failure. The MSBS was calculated and recorded in MPa.

The MSBS data were submitted to statistical analysis. The equality of variances and normal distribution of errors was verified using Kolmogorov–Smirnov test. A normal distribution was satisfied, allowing the two-way ANOVA test, followed by multiple comparison with Tukey test with a significance level of $\alpha = 5\%$. Fractured specimens were fixed on aluminum stubs (Procind Ltda., Piracicaba, SP, Brazil) with the fractured interfaces facing upward, sputter-coated with gold (SDC 050 Sputter Coater, Baltec) and evaluated by scanning electron microscopy (SEM; JEOL, Tokyo, Japan) to determine the failure mode (adhesive, mixed, or cohesive in adhesive; Figure 1).

Results

There was a statistically significant interaction between the enamel pretreatment time and the adhesive system. Multiple comparisons are shown in Table 2. In the control group without pretreatment, Ambar showed the highest mean MSBS. When samples were pretreated with MI Paste for 1 min, the mean MSBS values of the Ambar and Stae groups were statistically similar and were higher than the mean MSBS value of the Single Bond group. Application of MI Paste for 2 min provided similar MSBS means for all adhesive systems.

For the Single Bond group, the highest mean MSBS value was found when specimens were pretreated with MI Paste for 2 min. For the Stae group, pretreatment (either 1 or 2 min) increased the MSBS values significantly. For the Ambar group, the application of MI Paste did not increase the MSBS.



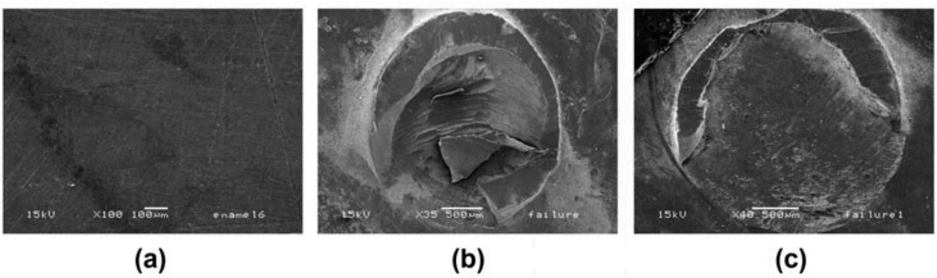


Figure 1. Failure modes after the microshear test. (A) adhesive failure mode, (B) cohesive in adhesive failure mode, (C) mixed failure mode.

Table 2. Means (SDs) of MSBS for each experimental group.

	MI Paste		
Adhesive system	Without	1 min	2 min
Single Bond	51.00 (3.80) Bb	51.74 (7.61) Bb	64.43 (9.6) Aa
Stae	58.22 (10.19) Bb	69.01 (3.63) Aa	70.73 (9.23) Aa
Ambar	72.89 (7.20) Aa	71.32 (7.08) Aa	72.66 (10.78) Aa

Notes: Same lowercase letter indicates no statistically significant difference among columns in each row. Same uppercase letter indicates no statistically significant difference among rows in each column.

Table 3. Distribution of failure modes among adhesive systems in terms of dentin pretreatment.

		Failure modes		
MI Paste		Adhesive	Mixed	Cohesive in adhesive
Without	Single Bond	9	1	0
	Stae	7	2	1
	Ambar	3	3	4
1 min	Single Bond	5	4	1
	Stae	8	2	0
	Ambar	3	6	1
2 min	Single Bond	5	5	0
	Stae	7	3	0
	Ambar	3	5	2

Table 3 shows the different failure modes founded after the MSBS test. The number of adhesive failure was lower with the application of MI Paste for 1 or 2 min. Ambar showed the lowest number of adhesive failures, regardless of the time of MI application.

Discussion

Based on the results of this study, the application of MI Paste before acid etching was effective to improve the enamel MSBS of the adhesive systems, except for the Ambar adhesive. Ambar presented a better performance compared to the other adhesives, even without enamel pretreatment. Therefore, either the duration of pretreatment with MI Paste or commercial type of adhesive influenced the bond strength.

The methodology used here has been described in other studies investigating the influence of MI application.[11,12] Although different methods can be used to measure the bond strength, the microshear test is easily reproduced. Moreover, this test does not create stress related to cutting the specimen before the test, a step that is necessary for the microtensile bond strength test.[13] As in other studies,[11,12] this study used a flowable resin. This material is a resin type that penetrates more easily into the mold space.

The two-step etch-and-rinse adhesive system offers a simplified adhesive technique to bond composite to tooth tissues. However, this type of system presents higher hydrophilicity than the three-step etch-and-rinse adhesive system.[14] As a result, the bonding interface can quickly degrade, leading to marginal infiltration and secondary caries. The performance of two-step adhesive systems can be improved by pretreating the teeth with different substances. Despite the etching step before its application, some content of CPP–ACP might have remained in the enamel and interact with the substrate. In this context, MI Paste may increase the longevity of the bonding interface by delaying the establishment of secondary caries, owing to the caries-preventive effect of CPP–ACP-based pastes.[6,11] A previous study demonstrated an inverse relationship between the bond strength of two-step adhesive systems and nanoleakage,[13] which is related to secondary caries development. Furthermore, the results of this study found an additional effect for the CPP–ACP-based paste, the improvement in the bond strength.

The positive effect of the CPP–ACP-based paste is consistent with findings in previous studies and is probably related to alterations in the etching patterns, so that it had improved interaction between the enamel and the adhesive system due to increased substrate reactivity.[5,6,11] Calcium deposition by MI on enamel may establish strong ionic bonds between the monomer, the acrylic copolymer, and itaconic acids.[6] Acid etching performed after the MI application may have helped to break CPP from the ACP and to release calcium ions, favoring the interaction between calcium and monomer. However, other studies can be performed to confirm the above-mentioned suppositions.

A previous study reported that the bond strength was improved after MI Paste was applied for 60 min for 7 days,[11] which is an unworkable clinical time. Another study [6] managed higher bond strength values after applying MI Paste for 3 min immediately before the bonding procedure. In the present study, a shorter application time (1 or 2 min) provided a positive effect on the bond strength. Only for the Stae group, which uses acetone as a solvent, 1 min was sufficient to promote an improvement in bond strength. The reduction in the application time optimizes the clinical time, resulting in a faster procedure. MI Paste does not alter the morphology of the bonding interface, another advantage for its safe indication.[15]

Regardless of MI Paste use, the Ambar adhesive presented the higher MSBS values among the adhesive systems. Previous studies have demonstrated similar degrees of conversion [16] and bond strength values between Ambar and a three-step etch-and-rinse adhesive. Ambar contains an acid monomer (10-MDP) in its composition, which favors chemical bonding to dental tissues in addition to micromechanical retention.[17,18]

Differences in the failure mode distribution among different pretreatments can provide information on the influence of MI Paste on the bonding interface.[6] In this study, the adhesive failure type was the predominant mode. However, many mixed failures were observed in groups that were pretreated for 1 or 2 min. The Ambar group presented more cohesive failures in the adhesive, which could be related to a stronger bond to enamel. The better MSBS values of this group confirm its greater adhesion to enamel.

The better performance of the Ambar group might be an evidence for its use in clinical practice, although the other adhesive systems presented similar MSBS values when they were pretreated with MI Paste for 2 min. In addition to the previously reported caries-preventive effect of MI Paste,[7,19] the higher MSBS values obtained in this study indicate that MI Paste improved the quality of the bonding interface, which might contribute to increased material retention into the cavity. These data might encourage the use of the CPP–ACP-based paste before the bonding procedure and a reduced application time, but studies with the long-term aging of the specimens are necessary to evaluate the durability of the effect of this treatment. Further *in situ* and *in vivo* studies are also necessary to validate the effectiveness of enamel pretreatment with MI Paste.

Conclusion

The effect of CPP–ACP pretreatment on enamel bond strength was material dependent. For the 10-MDP-based adhesive system, the application of MI Paste on enamel did not favor increased bond strength, regardless of the exposure time. For the adhesive system containing copolymer of acrylic and itaconic acids, the pretreatment with MI Paste for most-reduced exposure time was enough to prove increased bond strength, while for the adhesive system absent of 10-MDP and copolymer of acrylic and itaconic acids only the most-extended exposure time provided increased bond strength.

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