Effect of Er:YAG Laser and Reduced Time of Acid Etching on Bond Strength of Self-adhesive Resin Cement to MTA and Biodentine

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Abstract

Introduction: Considering the recent trend to use mineral trioxide aggregate (MTA) and Biodentine and resin cements, more conservative approaches concurrent with adequate bond strength have always been requested. The present study aimed to evaluate the effect of pretreatment with Er:YAG laser etching versus acid-etching for 5 and 15 seconds on the micro shear bond strength of self-adhesive resin cement (SRC) to MTA and Biodentine.

Materials and Methods: Forty-eight samples of each cement (MTA and Biodentine) were prepared and distributed into four groups based on surface pretreatment: 1) control, no treatment; 2) Er:YAG laser etching with energy of 60 mJ; 3) 5-second acid-etching; 4) 15-second acid-etching. All specimens were cemented using SRC. Microshear bond strengths were tested following 24-hour water storage. Debonded specimens were examined and surface topography was assessed using an atomic force machine (AFM). Data analysis was performed using the two-way ANOVA and Tukey multiple comparisons test.

Results: The three testing groups of laser etch and 5-s and 15-s acid-etch demonstrated a significantly higher SBS than the control group ($P < 0.05$) with negligible differences among them ($P > 0.05$). Furthermore, Biodentine showed better adhesive bonding than MTA in all groups.

Conclusion: Laser etching of 60 mJ and 5-s acid-etching were as beneficial as 15-s acid-etching in terms of bond strength of SRC to MTA and Biodentine.

Keywords: Acid-etching, Bond strength, Biodentine, Lasers, Mineral trioxide aggregate Angelus, Self-adhesive resin cement

Introduction

Mineral trioxide aggregate (MTA) has been approved as a gold standard in vital pulp therapies due to its ideal performance in terms of biocompatibility, bioactivity, adequate seal, setting ability in the presence of moisture and its resistance to dislodgement.¹ On the other hand, Biodentine as another novel calcium silicate-based cement (CSC) was subsequently introduced, presenting several more favorable physical properties such as higher compressive strength, lower porosity and accelerated setting reaction.²

In most coronal pulpotomy-treated teeth, large restorations are needed.³ Indirect approaches are preferred due to improved physical/mechanical properties and simple production of accurate proximal contacts and contours.⁴ CAD-CAM inlay/onlay restorations performed in one session without any need for a temporary phase minimizes leakage and produces an immediate coronal seal.^{5,6}

Besides the importance of appropriate vital pulp treatment materials, the outcome of coronal pulpotomy treatment is correlated with the hermetic seal not only at the dentin-CSC interface but also between the restoration and CSC.⁶ In order to obtain this favorable bond in indirect restorations, using resin cements is highly advantageous. The introduction of self-adhesive resin cements (SRCs) to the dental market was a major development in adhesive dentistry with a combination of ideal mechanical and bonding capability characteristics. They have simplified the process of cementation by providing a less technique-sensitive and time-consuming procedure. The acidic monomer in their composition could partially lead to micromechanical retention, and it lacks the necessity of further etching/priming.⁷ Nevertheless, in spite of available contrary opinions,^{8,9} some researchers believe that pretreatment with acid prior to self-adhesive luting results in a stronger bond to tooth structures.¹⁰⁻¹² It might also be constructive for the bond

of SRCs to CSCs. Bearing in mind the reported adverse effects of acid-etching for 15 seconds on the compressive strength and mechanical properties of CSCs,¹³ reduction in etching time or conservative pretreatment approaches such as lasers could be practical options. In our recent investigations, shortening of acid etching time to 5 seconds has resulted in enhanced resin bond with a universal adhesive to CSCs, preventing the detrimental effect of 15-second phosphoric acid on the compressive strength of MTA and Biodentine.¹³ Also, in the presence of universal adhesive, the Er:YAG laser has appeared to be able to develop satisfactory resin bonding as strong as using 15-s acid etching without compromising the strength of CSCs.¹⁴ The bond strength between SRC and laser-treated dentin in post space has been examined.¹⁵ However, little information is available about the effect of laser pretreatment and acid etching when SRCs are bonded to CSCs. Therefore, an investigation into the effect of Er:YAG laser etching versus reduced acid etching time on microshear bond strength of SRC to MTA and Biodentine was the main goal in the current study.

The supposition that different surface pretreatments would not affect the microshear bond strength of MTA and Biodentine to SRC could be assumed as the null hypothesis of the present examination.

Materials and Methods

Ninety-six cylindrical acrylic specimens with a central hole of 2 mm in diameter and height were prepared and filled with two groups of either MTA (MTA, Angelus, Londrina, Brazil, Lot # 40422) or Biodentine (BD, Septodont, France, lot # B19471) following the manufacturer's recommendations. Wet cotton pellets covered the blocks and subsequently, the samples were stored in an incubator at 37°C under about 100% humidity for 72 hours to reach their final setting. Afterwards, with the purpose of obtaining a uniform surface, the specimens were polished by means of 600-grit silicon carbide paper for 1 min and distributed into four groups (n=12) based on the surface treatment: group 1) No surface conditioning (control); groups 2 and 3) 37% phosphoric acid for 5 and 15 s, respectively; group 4) Er:YAG laser irradiation. In the last group, the Er:YAG laser (LightWalker ATS, Fotona, Slovenia) with 60 mJ energy (11.94 J/cm²) was applied uniformly and perpendicularly to the surfaces in a non-contact mode at a distance of 1-2 mm for 10 seconds. The procedure was performed using a 0.8 mm diameter tip of the 'H14' handpiece with the following parameters: 2940 nm, 10 Hz, MSP mode (100 µs), water 8, and air 4.

Following surface treatment, cylindrical elastic molds (1 × 1 mm) were placed on the specimens and filled with SRC (Kuraray Noritake Dental Inc, Sakazu, Kurashiki, Okayama, Japan, Lot # 033BBA). Using a light-curing unit (VIP Junior, Bisco), they were light-cured at a light intensity of 600 mW/cm² for 20 seconds. The samples were

then stored in distilled water for 24 hours at 37°C. Bond strengths in microshear (µSBS) were tested in MPa, using a universal testing machine (Zwick, Ulm, Germany) at a crosshead speed of 1 mm/min. Subsequently, the debonded surfaces were evaluated under a stereomicroscope at ×20 magnification to categorize the failure modes as follows: (1) adhesive failure between CSC and resin cement; (2) cohesive failure within the resin cement or in the bulk of CSC; and (3) mixed failure, a combination of adhesive and cohesive failure modes.

Two further specimens from each group of both CSCs were prepared with the same method as mentioned previously. In order to observe topographical features, the sample surfaces were scanned with an atomic force machine (AFM) (Naio AFM, NanoSurf, Switzerland) via a sharp silicon cantilever (Tap150Al-G, Budget Sensor, Bulgaria) in a dynamic non-contact mode, with the resonance frequency of 160 kHz and stiffness constant of 0.2 N/m.

Statistical Analysis

The data were submitted to two-way ANOVA analysis and the Tukey test at a significance level of 5%. All statistical analyses were performed by employing SPSS, version 16.0 software (SPSS Inc, Chicago, IL, USA).

Results

The comparison of the mean SBS and contributing standard deviations of the four groups in each CSC are presented in [Table 1](#). According to the two-way ANOVA, the effects of CSC type and treatment were significant ($P = 0.01$, $P < 0.001$); no interaction between CSCs and pretreatments was found. This finding declares that both CSCs demonstrate a similar trend in their reaction to pretreatments. Thus, the data were paraphrased and compared using Tukey post hoc tests. Accordingly, variation in the type of CSC led to a significant effect on the µSBS of SRC to the underneath pulp capping material ($P = 0.01$) in such a way that Biodentine showed a better performance. In addition, although the three testing groups of laser etch and acid etch for 5 and 15 seconds yielded a significantly higher µSBS than the control group ($P < 0.05$), no differences were observed among them ($P > 0.05$).

In the control group of MTA and Biodentine, all failures were adhesive, while in all treated groups of MTA and Biodentine, the adhesive failure was the main failure with a few mixed failures observed in some specimens.

The representative 3D images and micrographs of atomic force microscopy related to the topography of MTA and BD in all groups are illustrated in [Figures 1](#) and [2](#) respectively. The samples were entirely studied at the same magnification (10 × 10 µm). Bright and dark areas indicate height and depth respectively. AFM analysis showed a fairly even and uniform surface in the

Table 1. Means and Standard Deviations of Microshear Bond Strengths (MPa) of Self-adhesive Resin Cement to MTA and Biodentine Affected by Different Etching Modes

CSC	MTA		Biodentine	
	Mean (SD)*	Failure Mode	Mean (SD)	Failure Mode
Self-adhesive	3.28 (1.00) ^{Aa}	12/0/0	4.06 (1.46) ^{Ab}	12/0/0
Acid etch 5s	5.9 (1.50) ^{Ba}	8/2/2	6.58 (1.64) ^{Bb}	10/2/0
Acid etch 15s	5.84 (1.48) ^{Ba}	9/1/2	6.7 (1.30) ^{Bb}	8/3/1
Laser etch 60	5.74 (1.50) ^{Ba}	10/1/1	6.54 (1.52) ^{Bb}	11/1/0
P value	< 0.05		< 0.05	

The numbers in the failure mode column represent adhesive failure, cohesive failure within CSC or resin cement, and mixed failure respectively.

*Different superscript capital letters in each column and lowercase letters in each row indicate a statistically significant difference ($P < 0.05$).

control groups of both CSCs. Almost all etching methods modified the surface topography by raising the roughness; in 15 seconds acid etch and laser etch groups for both MTA and Biodentine, however, this alteration was more obvious, providing a porous pattern through increased height/depth of the peaks/valleys.

Discussion

The present study investigated the impact of laser etching or 5-s acid etching versus 15-s acid etching (as a practical standard etching time) of CSCs on the bonding ability of an SRC. Our previous findings indicated that 5-second etching and 60 mJ laser irradiation had no detrimental effect on the strength of CSCs contrary to 15-second etching time.^{13,14} Therefore, these pretreatments were applied in the current study; consequently, they were as effective as 15-second etching in the promotion of SRC bonding to both CSCs. This result was in agreement with our recent studies with universal adhesive in the case of MTA, but not for Biodentine. The higher viscosity of SRC compared to that of universal adhesive could justify this difference in the obtained results.

The parameters of Er:YAG laser irradiation used in the current study were based on the outcome of a recent study demonstrating 60 mJ laser etching as the optimal energy for the creation of a retentive pattern without a destructive impact on the strength of CSCs.¹⁴ Compared to the control group, both the mentioned laser etching mode and 5-s acid etching were successful in improving the bond strength of SRC to CSCs.

The presence of some acidic monomers in SRCs was demonstrated to interact with the calcium ions.¹⁶ MTA and Biodentine cements produce a high amount of calcium ions from calcium hydroxide, a by-product of hydration, and from the decomposition of calcium silicate hydrate.^{10,17} A chemical bond is established as a result of a chelation process in which functional monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) react with calcium ions, increasing the bond strength.^{2,18} Nevertheless, increased surface energy and wettability of CSCs following acid or laser etching

were more vital. This may provide intimate contact of self-adhesive cement on the surface of CSC, facilitating chemical bonding.¹⁹

AFM observations were in line with obtained results. The porous pattern with multiple heights and depths provided by all treatments could justify the increased bonding to CSCs. It is noteworthy that 15-s acid etch and laser etch groups in both MTA and Biodentine materials altered the surface more considerably. Furthermore, according to our previous SEM observations, these treatments could create micro-retentive patterns on the surface of CSC that might be responsible for higher resin bonding to MTA and Biodentine. The higher bond strength of SRC to MTA and glass ionomer as root perforation materials compared to Portland cement has been attributed to their surface roughness, especially for GI.¹⁸ However, those authors reported that μ SBS values obtained in their study were lower than those of value range required to prevent gap formation at the cement interface. Contrarily, laser and acid etching seem to achieve this level of bond strength.

In order to simulate a clinical environment, MTA and Biodentine should be stored in a humid environment for about 72 hours to complete their ultimate setting.²⁰ While a 3D scan is carried out in the second session as a computerized impression, the pretreatment conditioning could be performed prior to the permanent cementation of final indirect restoration with SRC in the same session; therefore, the bonding of SRC could be carried out after 72 hours without any delay for the fabrication of indirect restoration.

Biodentine demonstrated higher microshear bond strength compared to MTA, regardless of the etching method. This outcome was in line with the available data reported previously^{13,14,21} and this could be as a result of high density and the superficial porosity of Biodentine providing micromechanical bonding. Hypothetically, the 10-MDP monomer may bind chemically to the calcium in Biodentine and promote chemical adhesion and micromechanical attachment.^{21,22}

Present results cannot be directly applied to the clinical situation without taking into account the limitations of

Figure 1. AFM Images of MTA in Two Views of 3D Images (a1-d1) and Topographical Micrographs (a2-d2). Bright and dark areas indicate height and depth respectively. a1 and a2) non-etch surface, relatively flat and uniform surface. b1 and b2) 5s acid etch surface, more irregularities. c1 and c2) 15s acid etch surface, micro retentive appearance as a number of concavities and convexities. d1 and d2) laser etch, valley-like appearance full of peaks and valleys.

Figure 2. AFM Images of Biodentine in Two Views of 3D Images (a1-d1) and topographical Micrographs (a2-d2). Bright and dark areas indicate height and depth respectively. a1 and a2) non-etch surface, relatively flat and uniform surface. b1 and b2) 5s acid etch surface. c1 and c2) 15s acid-etch surface, micro-retentive appearance as a number of concavities and convexities. d1 and d2) laser etch, more irregularities.

this study. In clinical conditions, the thinner thickness of cement layers would be applied in a cavity with higher C-factor compared to the flat surface used for in vitro studies. Moreover, different types of stresses in oral environment like pH changes, thermal-cycling, occlusal loads and challenges due to the presence of enzymes could damage the bonding effectiveness of the luting agents. Future clinical trials are necessary to validate the outcomes of the present study.

Conclusion

Considering the limitations of this study, it could be deduced that the surface pretreatment of MTA and Biodentine with both laser and acid etching is more advantageous than no pretreatment in terms of their bond

strength to SRC.

Since there was no difference between 15s and 5s acid etching and laser etching, treatment with either laser or 5s phosphoric acid could be suggested due to their lower destructive effects.

Ethical Considerations

This article does not contain any studies with human participants or animals performed by any of the authors.

Conflict of Interests

The authors declare that they have no conflict of interest.

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