Effects of chemical and physico-chemical surface conditioning methods on the adhesion of resin composite to different mineral trioxide aggregate based cements

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Effects of chemical and physico-chemical surface conditioning methods on the adhesion of resin composite to different mineral trioxide aggregate based cements

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Short title: *Adhesion of resin to mineral aggregate-based cements*

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Abstract: This study investigated the adhesion of resin composite to mineral trioxide aggregate based cements after different chemical and physico-chemical surface conditioning methods. Mineral trioxide aggregate based cements (Biodentine, ProRoot MTA, Imicryl MTA) were embedded in Teflon disks (N=180). After storing at 37°C at 100% humidity for 72 h, substrate surfaces were polished using silicon carbide papers. Specimens were allocated to 3 groups to be conditioned with one of the following (n=15 per group): a) Adhesive resin (Clearfil SE Bond, CSE), b) Adhesive resin (Adper Single Bond 2, SB2), c) air-abrasion with 30 μm alumina coated with silica + silane+adhesive resin (ALB), d) no surface conditioning, control group (CON). Microhybrid resin composite (Filtek Z250) was applied on the conditioned substrate surfaces and photo-polymerized. After storage at 37°C at 100% humidity for 24h, adhesive interfaces were loaded under shear (1 mm/min) in a universal testing machine. After debonding failure types were analyzed. Data were analyzed using 2-way ANOVA and Tukey's test (alpha=0.05). SBS results were significantly affected by surface conditioning ($p<0.05$) and materials ($p<0.05$). Interaction terms were significant ($p<0.05$). Biodentine-ALB resulted in significantly higher SBS values (3.96±1.24) compared to those of other combinations, while ALB and SB2 resulted in no significant difference for ProRoot MTA and Imicryl MTA ($p>0.05$). CSE (1.36±0.5- 1.98±0.76) did not significantly increase SBS for all MTA materials compared to the control group (0.8±0.52 - 2±0.91) ($p>9.05$). While CON groups resulted in exclusively adhesive failures, ALB presented the highest incidence of mixed failures for all materials tested (60-100%).

Keywords: Adhesion; Air-abrasion; Etch and rinse adhesive; Mineral trioxide aggregate; Self-etch adhesive; Shear bond strength

Introduction

Obtaining long-term durable seal and preservation of vitality through restorative procedures in teeth with exposed pulp tissue, is a desirable yet a difficult task to achieve for clinicians 0. Dentin bridge formation is crucial for the preservation of the pulp vitality, which is the main aim of the treatment modalities that can be achieved using pulp capping materials 0. Noxious stimuli, such as thermal, mechanical, and chemical stimuli, may cause damage to the pulp tissue but can be prevented with the use of such materials 0. Although calcium hydroxide has been widely accepted and used as a pulp capping material, poor solubility and adherence and tunnel defect formations throughout the dentin bridges adjacent to the material cause inconsistent performance 0. Therefore, new dental biomaterials have been introduced for such treatments 0.

In 1993, with the introduction of a mixture consisting of tricalcium aluminate, dicalcium silicate, tricalcium silicate, tetracalcium aluminoferrite and bismuth oxide, mineral trioxide aggregate (MTA) became one of the most promising pulp capping materials 0. Unfortunately, long setting time, poor handling characteristics and tooth discoloration are critical clinical disadvantages of this material 0. In addition, its incompatibility with other materials is a disadvantage considering the layered structure of coronal restorations in pulp capping procedures 0. Due to these shortcomings, a variety of different calcium silicate-based materials with improved physical properties have been introduced in order to overcome the disadvantages presented by MTA 0.. With its shorter setting time and high compressive strength, Biodentine (Septodont, Saint-Maur-des-Fosses, France), which contains tricalcium silicate, calcium carbonate, and zirconium oxide, has been advocated for use as a pulp capping material. Biodentine was found to be comparable to MTA in terms of biocompatibility, bioactivity and remineralization properties 0. On the other hand, MTA materials based on tricalcium silicate are considered as bioactive repair cement that can be used universally for vital pulp and other endodontic and pediatric indications in primary and permanent teeth. While MTA materials are a mixture of powder and water-based gel liquid (i.e. Imicryl MTA, Imicryl, Konya, Turkey) or liquid (i.e. ProRoot, Dentsply

Sirona Endodontics, Tulsa, OK, USA) their mixing proportions may vary depending on the product which also could affect their mechanical properties. The manufacturers claim that gel liquid improves the handling properties and results in a stable mixture that resists wash-out.

Adhesion between the mineral aggregate-based cements and restorative resin-based materials is crucial in order to ensure the uniform stress distribution between the capping and restorative material, enabling a prolonged success rate with better sealing abilities 0. Although stronger bonding properties have been achieved with etch and rinse adhesive systems, the deterioration in the microstructure of Biodentine and increased leakage through the bonded interface are inevitable with such adhesive systems 0. In order to improve adhesion of resin-based materials, air-abrasion of the substrate materials used for restorative purposes has been suggested 0. Numerous studies have been published on the adhesion of coronal filling materials to mineral aggregate-based cements 0, to the best of our knowledge, there has been no studies regarding the bond strengths of resin composites to mineral aggregate-based cements after chemical and physico-chemical surface conditioning methods where physical activation is achieved through air-borne particle abrasion and the chemical one through silane coupling agent application.

The objectives of this study therefore were to evaluate the shear bond strength of resin composite to mineral trioxide aggregate based cements used for pulp capping and to classify the failure types after debonding. The null hypothesis tested was that there would be no significant effect of the conditioning methods for all mineral aggregate-based cements to be investigated.

Materials and Methods

Specimen preparation

The brands, manufactures, batch numbers, chemical compositions, and application procedures of the materials used are summarized in Table 1.

Polyethylene (Teflon) moulds (diameter; 3 mm, height: 2 mm) (N=180) were filled with mineral trioxide aggregate based cements (Biodentine, ProRoot MTA, Imicryl MTA) after mixing according to each manufacturer`s instructions.

While ProRoot MTA was prepared by mixing the powder and water at a ratio of 3:1, this ratio for Imicryl MTA was 2:1 where liquid was a water-based gel. Biodentine on the other hand, was mixed in a high-speed amalgamator for 30 s. The mixed materials were introduced into the polyethylene moulds using a spatula. Until the setting process was complete, all the specimens were covered with water dampened cotton pellets. After storing at 37°C at 100% humidity for 72 h, substrate surfaces were polished using silicon carbide papers in sequence of 320, 600, 800, and 1200 grit size (Type S Alox, Presi, Eybens, France) for 15 s per grit size under water cooling.

Surface conditioning methods

Group CSE: In this group, the substrate surfaces were coated with one layer of primer (Clearfil SE Bond, Kuraray Noritake Dental Inc., Kurashiki, Okayama, Japan) for 20 s with a single use applicator. After gently air drying for 5 s, adhesive resin of CSE system was applied one coat and photo-polymerized using a light-emitting diode light-curing unit (LED) (Elipar S10, 3M ESPE, St. Paul, MN, USA) (wavelength: 455 nm; light intensity: 1200 mW/cm_2) for 10 s.

Group SB2: In this group, the substrate surfaces were first etched with 35% H_3PO_4 for 15 s, washed with water for 10 s, and air-dried gently for 2 s. Next, Adper Single Bond 2 (3M ESPE GmbH) (SB2) was applied one coat using a single use applicator, and air-dried gently for 5 s. The adhesive resin was then photo-polymerized for 10 s.

Group ALB: The substrate surfaces were first air-borne particle abraded with 30 μm alumina coated with silica (CoJet Sand, 3M ESPE GmbH) using a chairside air-abrasion device (Dento-Prep, Ronvig, Daugard, Denmark) from a distance of 10 mm distance for 5 s at 2 bar pressure. Excess sand was removed with copious amount of distilled water and gently air- dried. Then, silane coupling agent (Monobond Plus, Ivoclar Vivadent AG, Schaan, Liechtenstein) was applied to the air-abraded surfaces

and waited for its reaction for 60 s using a single use applicator. After air-drying gently for 5 s, adhesive resin (Heliobond, Ivoclar Vivadent AG) was applied to the surface one coat, air-thinned and photo-polymerized for 10 s as described in Group CSE.

Group CON: No surface conditioning was applied prior to the application of resin composite in this group and acted as the control group.

Microhybrid resin composite (Filtek Z250, 3M ESPE) was applied on the conditioned substrate surfaces and photo-polymerized using custom made polyethylene moulds (diameter: 2 mm; height: 3 mm) which was then photo-polymerized for 20 s. The bonded specimens were stored at 37°C with 100% humidity for 24 h prior to testing.

Microshear tests

The specimens were loaded under shear at the substrate-resin interface in a Universal Testing Machine (Shimadzu Autograph AGS-J 5kN, Shimadzu Corporation, Tokyo, Japan) at a cross-head speed of 1 mm/min until failure. The microshear bond strength (SBS) for each specimen was calculated in MPa by dividing the highest load (Newtons) by the adhesive surface area (3.14 mm^2).

Microscopic examination and failure analysis

After adhesion tests, debonded specimen surfaces were examined by two operators (T.E. and K.O.) in order to analyze the failure types using an optical microscope (Axio Zoom.V16, Carl Zeiss, Jena, Germany) at x40 magnification.

Failure types were planned to be classified as follows 0: Score 1: Adhesive failure between the mineral aggregate-based cement and the resin composite with no resin remnants left on the substrate, Score 2: Cohesive failure within the mineral aggregate-based cement, Score 3: Mixed failure with both adhesive and cohesive failure within the substrate.

Statistical analysis

Statistical analysis was performed using SPSS 17.0 software (SPSS Windows, Chicago, IL, USA). Levene test was used to test normal distribution of the data. As the data were not homogeneously

distributed, SBS data were analyzed using 2-way ANOVA and Tukey`s tests where the bond strength was the dependent variable and conditioning methods (4 levels: CSE, SB2, ALB, CON), mineral aggregate based cements (3 levels: Biodentine, ProRoot MTA and Imicryl MTA) as independent variables. P values less than 0.05 were considered to be statistically significant in all tests.

Results

SBS results were significantly affected by surface conditioning ($p<0.05$) and material types ($p<0.05$). Interaction terms were significant ($p<0.05$).

Biodentine-ALB resulted in significantly higher SBS values (3.96±1.24) compared to those of other combinations, while ALB and SB2 resulted in no significant difference for ProRoot MTA and Imicryl MTA ($p>0.05$). CSE (1.36±0.5- 1.98±0.76) did not significantly increase SBS for all MTA materials compared to the control group, CON (0.8±0.52 - 2±0.91) ($p>9.05$) (Table 2).

The multiple comparisons indicated that the difference between the Biodentine and ProRoot MTA was not statistically significant ($p>0.05$), while the differences between Imicryl MTA and Biodentine ($p<0.05$) and Imicryl MTA and ProRoot MTA) were statistically significant ($p<0.05$).

The Biodentine-ALB subgroup had a significantly higher mean SBS value than the Biodentine-CSE ($p<0.05$), Biodentine-SB2 ($p<0.05$), and Biodentine-control ($p<0.05$) subgroups. The ProRoot MTA-control subgroup showed a statistically lower SBS than the ProRoot MTA-ALB ($p<0.05$) and the ProRoot MTA-SB2 subgroups ($p<0.05$). The Imicryl MTA-CON subgroup presented a statistically lower SBS than the Imicryl MTA-ALB ($p<0.05$) and the Imicryl MTA-SB2 subgroups ($p<0.05$). There was no statistically significant difference among the Biodentine-CSE, ProRoot MTA-CSE, and Imicryl MTA-CSE subgroups ($p>0.05$). In addition, among the Biodentine-SB2, ProRoot MTA-SB2, and Imicryl MTA-SB2 subgroups, no statistical significance was detected ($p>0.05$).

The Biodentine-ALB subgroup presented a significantly higher SBS value than the ProRoot MTA-ALB ($p<0.05$) and Imicryl MTA-ALB subgroups ($p<0.05$). However, the differences among the SBS values of the other ALB subgroups were not significant ($p>0.05$). The Biodentine-CON subgroup had a significantly higher SBS value than the Imicryl MTA-CON ($p<0.05$). There was no statistically significant difference among the other CON subgroups ($p>0.05$) (Table 2). Also, there was no significant difference between the CON and CSE subgroups in any of the mineral aggregate-based cements ($p>0.05$) showing infectivity of this self-etch adhesive.

While CON groups resulted in exclusively adhesive failures, ALB presented the highest incidence of mixed failures for all materials tested (60-100%) (Table 3).

Discussion

The present study investigated the effects of different surface conditioning protocols, including an etch and rinse adhesive system, self-etching adhesive system, air-abrasion and silanization where the negative control group did not receive any conditioning, on the adhesion of resin composite to mineral aggregate-based pulp capping material. Since SBS results were significantly affected by surface conditioning and material types, the null hypothesis could be rejected.

The setting reaction of the MTA materials were completed in 15 minutes, according to the manufacturer's instructions while Biodentine requires at least two weeks to set completely. However, after the initial setting reaction of the material which is 12 minutes, coronal restoration is processed 0. In fact, also three days setting time has been suggested for MTA materials for the optimal sealing of the material 0. In this study, based on these reasons, substrate materials were fabricated and kept 72 h in 100% humidity at 37°C to allow complete setting of the materials.

Except for air-abrasion methods, numerous studies have reported on the effect of different surface conditioning methods on the adhesion of restorative materials attached to mineral aggregate-based cements 0. Although it has been previously reported that the self-etching adhesives exhibited better

bond strength values to the mineral aggregate-based cements than the etch and rinse adhesives 0, conflicting results were stated where etch and rinse adhesives exhibited significantly higher bond strength values to White MTA 0. However, the findings of the current study did not support any of the reported studies since. CSE and SB2 surface treatments exhibited no significant effects on the SBS values where the results were comparable to the control group.

The pH values of different adhesive materials may be crucial on bond strengths of the materials to dentin 0. Yet, mineral aggregated-based cements have completely different structure than dentin. In a previous study, no correlation was found between the pH values of adhesive resins and SBS values to white MTA 0. It was however asserted that the solvent type used in the adhesives had a greater influence on bond strength results 0. SB2 included water and ethanol as solvent, whilst CSE had only water which may result in lower bond strength values due to incomplete polymerization in CSE subgroups compared to SB2 treated subgroups. Although the difference in SBS values between SB2 and CSE subgroups were insignificant, the SB2 subgroups had a significantly higher SBS value compared to the control groups. There was no significant difference between the control and CSE subgroups in any of the tested material groups.

In previous studies, the bond strength values between the resin composite and MTA, using different adhesive systems, was reported to range between 5 and 13 MPa 0. In the current study, the mean SBS value in the ALB-Biodentine group was 3.96 MPa, and in the ALB-ProRoot MTA group 2.94 MPa. Overall, the higher mean SBS values in previous studies 0 are not consistent with those of the present study. The reason for this and the non-significant difference between the self-etching adhesive, etch and rinse adhesive, and control groups within each tested material group may be due to the differences in the methods and materials. In the present study, the surfaces of the specimens were polished using 320, 600, 800, and 1200 grit silicon carbide papers for 15 s per paper in order to create very smooth and polished surfaces and standardize the adhesive interfaces for all specimens. However, in previous studies, the surfaces of the specimens were left untouched prior to the adhesive application. The rough

surface might have contributed to the microretentions and eventually increased SBS values 0. Altunsoy et al. reported similar results as in the present study and low SBS values due to surface polishing prior to the application of the adhesive resin 0. Although clinically not possible, the surface polishing of mineral aggregate-based cements was solely made for standardization of the specimens and eliminate any kind of discrepancies.

To the best of our knowledge, no study to date implemented the use of air-abrasion and silanization, the so-called tribochemical silica-coating, for conditioning mineral aggregate-based cements. Air-abrasion is a method used for cleaning and enhancing the surface area for improved surface adhesion 0. In addition, the use of silane coupling agent further acts as chemical conditioning method and improves the adhesion between essentially dissimilar materials, such as organic and inorganic materials. For this reason, the use of silane coupling agents has been promoted prior to the application of an adhesive resin typically based on methacrylate monomersl 0. Higher bond strengths values have been reported previously with use of a coupling agent, such as Monobond Plus followed by Heliobond compared to the non-silanized groups 0. Therefore, a silane coupling agent was used in the current study in the ALB groups. However, due to the physical properties of the MTA 0, the chosen application time was 5 s in order to limit the erosion of the material. In a pilot study, the application time of air-abrasion for 5 and 10 s on the surfaces of mineral aggregate-based cements, resulted in excessive loss from the material surface especially after 10 s. Therefore, a shorter deposition time of 5 s was employed in this study.

The increases in the SBS values in the ALB subgroups, with a significant difference between the ALB and control subgroups within each tested material group, seems to further support the efficiency of physico-chemical conditioning 0 which contributes to surface roughness and improved micromechanical retention along with chemical adhesion with the silane 0. Although in each tested material group, the difference between the ALB and control subgroups was statistically significant, only in the Biodentine group, ALB subgroup exhibited significantly higher SBS values compared to the CSE,

SB2, and control groups. These results could be attributed to the enhanced strength of the material due to the low water/cement ratio used in mixing Biodentine. In each MTA group, the difference in the SBS value between the ALB-CSE subgroups and between the ALB-SB2 subgroups no significant difference was observed. Higher bond strength results could be due to the chemical adhesion of the MDP monomer in the CSE and SB2 composition to the alumina in the MTA. The hydrolytically stable salts (10-MDP-Ca) was reported to bind to the 10-MDP and Vitrebond copolymer to the calcium in the ProRoot MTA and the Biodentine 0. On the other hand, Imicryl MTA, although lower, presented statistically no significant SBS values in all the surface conditioning protocols when compared to the ProRoot MTA. It also showed no significant SBS values for all the surface conditioning protocols, except for the ALB, when compared to Biodentine.

Some of the previous studies reported no adhesive failures 0, while others described adhesive failures in approximately one-half of the specimens 0. The type of failure is not an actual reflection of the true bond strength between the mineral aggregate-based cements and the adhesive resin. However, cohesive failures in the substrate material are more acceptable than adhesive failures for a successful adhesive interface 0. According to the findings of this study, all ALB subgroups showed higher incidence of mixed failures than the other surface conditioning methods. These finding are in accordance with the SBS test results showing high values in the ALB subgroups. Moreover, Imicryl MTA groups showed more cohesive failures than the other mineral aggregate-based cement groups, which could be related to les cohesive strength of the material itself. In the current study, the cohesive failure of the mineral aggregate-based cements indicated poor cohesive strength of the mineral aggregate-based cements and thereby poor adhesion demonstrated by low bond strength obtained in all groups 0. The presence of dentin and enamel surrounding the pulp capping materials may contribute to increased adhesion of the restorative resin material which needs to be further evaluated in future studies.

Conclusions

From this study, the following could be concluded:

1. Air-abrasion followed by silane coupling agent and adhesive resin significantly increased adhesion of resin composite to mineral aggregate-based cements.
2. The application of etch and rinse adhesive and self-etching adhesive tested exhibited results similar to those with no surface treatment indicating no additional benefit to adhesion of resin composite to mineral aggregate-based cements.
3. Adhesion to Biodentine demonstrated higher bond strength results than those of MTA materials regardless of the conditioning method.
4. Failure types were mainly mixed type of failures.

Clinical Relevance

Mineral aggregate-based cement surfaces can best be conditioned physico-chemically using air-borne particle abrasion followed by silane coupling agent and adhesive resin in order to improve adhesion of resin composite.

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Conflict of interest

The authors did not have any commercial interest in any of the materials used in this study.

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Captions to figures and tables:

Tables:

Table 1. Brands, manufacturers, batch numbers, chemical compositions and application procedures of the tested materials.

Table 2. Mean microshear bond strength values (MPa±standard deviations) of resin composite to mineral aggregate-based cements after chemical (CSE: Clearfil SE Bond, SB2: Adper Single Bond 2) and physico-chemical (ALB: air-abrasion with 30 μm Al_2O_3 coated with SiO_2+silane coupling agent+adhesive resin) surface conditioning methods compared to no conditioning, control (CON). *Lower case letters in one column represent statistically significant differences and uppercase letters in one row ($p<0.05$).

Table 3. Distribution of failure types after debonding according to surface conditioning methods. Score 1: Adhesive failure between the mineral aggregate-based cement and the resin composite with no resin remnants left on the substrate, Score 2: Cohesive failure within the mineral aggregate-based cement, Score 3: Mixed failure with both adhesive and cohesive failure within the substrate. See Table 2 for group abbreviations.

Tables:

Brands, manufacturers and batch numbers	Chemical Composition	Application procedure
Biodentine (Septodont, Saint-Maur-Des-Fosses, France) (B19841)	Powder: Tricalcium silicate, dicalcium silicate, calcium carbonate and oxide, iron oxide, and zirconium oxide Liquid: Calcium chloride and hydrosoluble polymer	Five doses of liquid and powder was mixed for 30 s with an amalgamator
ProRoot mineral trioxide aggregate (MTA) (Dentsply Sirona Endodontics, Tulsa, OK, USA) (123922)	Tricalcium silicate, bismuth oxide, dicalcium silicate, tricalcium aluminate, and calcium sulfate dehydrate or gypsum	Mix powder/liquid (ratio: 1/3)
Imicryl MTA (Imicryl, Konya, Turkey) (17350)	Powder: Tricalcium silicate, dicalcium silicate, tricalcium aluminate, ytterbium oxide, and calcium sulfate hemihydrate Liquid: Gelling agent hydrosoluble polymer	Mix powder/liquid (ratio: 1/2)
Adper Single Bond 2 (3M ESPE, St. Paul, USA) (N632127)	10-methacryloyloxydecyl dihydrogen phosphate (MDP) monomer, dimethacrylate, resin, Vitrebond copolymer, filler, ethanol, water and silane	Apply 35% H_3PO_4 for 15 s. Wash with water for 10 s. Air dry for 2 s. Apply adhesive for 20 s with a single use applicator. Gently air dry for 5 s prior to polymerization for 10 s.
Clearfil SE Bond (Kuraray Noritake Dental Inc., Kurashiki, Okayama, Japan) (000224)	Primer: MDP, 2-hydroxyethyl methacrylate (HEMA), hydrophilic dimethacrylate, dl-camphorquinone, N,N-diethanol-p-toluidine, and water Bond: MDP, bisphenol A diglycidylmethacrylate (bis-GMA), HEMA, hydrophobic dimethacrylate, dl-camphorquinone, N,N-diethanol-p-toluidine, and silanated colloidal silica	Apply primer for 20 s with a single use applicator. Gently air dry for 5 s prior to polymerization for 10 s.
Monobond Plus (Ivoclar Vivadent, Schaan, Liechtenstein) (W878421)	Adhesive monomers (silane methacrylate and phosphoric methacrylate sulfide methacrylate) and ethanol	Apply to clean dry surface for 60 s then air dry for 5 s.
Heliobond (Ivoclar Vivadent) (W87841)	bis-GMA, triethylene glycol dimethacrylate (TEGDMA)	Apply 35% H_3PO_4 for 15 s. Wash with water for 10 s. Air dry for 2 s. Apply for 20 s with a single use applicator. Gently air dry for 5 s prior to polymerization for 10 s.

Table 1. Brands, manufacturers, batch numbers, chemical compositions and application procedures of the tested materials.

Conditioning Methods	Biodentine	ProRoot MTA	Imicryl MTA
CSE	1.98 (0.76)$_{a,A}$	1.96 (0.80)$_{acd,A}$	1.36 (0.50)$_{ac,A}$
SB2	2.34 (0.62)$_{a,A}$	2.43 (1.06)$_{bc,A}$	1.82 (0.75)$_{bc,A}$
ALB	3.96 (1.24)$_{b,A}$	2.94 (1.00)$_{bd,B}$	1.99 (0.68)$_{bc,B}$
CON	2.00 (0.91)$_{a,A}$	1.32 (0.87)$_{a,AB}$	0.80 (0.42)$_{a,B}$

Table 2. Mean microshear bond strength values (MPa±standard deviations) of resin composite to mineral aggregate-based cements after chemical (CSE: Clearfil SE Bond, SB2: Adper Single Bond 2) and physico-chemical (ALB: air-abrasion with 30 µm Al_2O_3 coated with SiO_2+silane coupling agent+adhesive resin) surface conditioning methods compared to no conditioning, control (CON). *Lower case letters in one column represent statistically significant differences and uppercase letters in one row ($p<0.05$).

	Imicryl MTA			ProRoot MTA			Biodentine		
	ALB	**SB2**	**CSE**	**ALB**	**SB2**	**CSE**	**ALB**	**SB2**	**CSE**
Score 1	0	0	0	0	0	0	0	0	0
Score 2	4 (26%)	9 (40%)	10 (67%)	0	4 (26%)	6 (40%)	6 (40%)	7 (47%)	7 (47%)
Score 3	11 (74%)	6 (40%)	5 (33%)	15 (100%)	11 (74%)	9 (60%)	9 (60%)	8 (53%)	8 (53%)

Table 3. Distribution of failure types after debonding according to surface conditioning methods. Score 1: Adhesive failure between the mineral aggregate-based cement and the resin composite with no resin remnants left on the substrate, Score 2: Cohesive failure within the mineral aggregate-based cement, Score 3: Mixed failure with both adhesive and cohesive failure within the substrate. See Table 2 for group abbreviations.