

Influence of Hemostatic Solution on Bond Strength and Physicochemical Properties of Resin Cement

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The aim of this study was to evaluate the degree of conversion, color stability, chemical composition, and bond strength of a light-cured resin cement contaminated with three different hemostatic solutions. Specimens were prepared for the control (uncontaminated resin cement) and experimental groups (resin cement contaminated with one of the hemostatic solutions) according to the tests. For degree of conversion, DC (n = 5) and color analyses (n = 10), specimens (3 mm in diameter and 2 mm thick) were evaluated by Fourier transform infrared spectroscopy (FTIR) and CIELAB spectrophotometry (L*, a*, b*), respectively. For elemental chemical analysis (n = 1), specimens (2 mm thick and 6 mm in diameter) were evaluated by x-ray energy-dispersive spectroscopy (EDS). The bond strengths of the groups were assessed by the microshear test (n = 20) in a leucite-reinforced glass ceramic substrate, followed by failure mode analysis by scanning electron microscopy (SEM). The mean values, except for the elemental chemical evaluation and failure mode, were evaluated by ANOVA and Tukey's HSD test. The color stability was influenced by storage time (p<0.001) and interaction between contamination and storage time (p<0.001). Hemostop and Viscostat Clear contamination did not affect the DC, however Viscostat increased the DC. Bond strength of the resin cement to ceramic was negatively affected by the contaminants (p<0.001). Contamination by hemostatic agents affected the bond strength, degree of conversion, and color stability of the light-cured resin cement tested.

Introduction

The increasing demand for porcelain laminate veneers has led to the continuous development of dental ceramics with improved optical properties, translucency, and esthetics (1). It is well known that restorative ceramic materials require a clinical stage of bonding to tooth structures with adhesive resin cements (2). Thus, the optical behavior of a ceramic restoration is determined by the combination of the color of the underlying tooth structure, the thickness/composition of the ceramic veneer, and the color of the luting cement (3).

The use of light-cured cements is based on the concept that the light emitted by curing units could pass through some indirect restorations, due to their translucency and reduced thickness (1). When bonding is effective, adhesive luting promotes increased fracture resistance of ceramic materials by cement penetration into the irregularities present at the internal surface of the restoration, leading to higher bond strength and lower crack propagation (4). Moreover, photoactivated resin cements (light-cured) have better color stability compared with that of chemically or dual-cured resin cements (5). This fact is probably related to the absence of chemical activator binders (tertiary amines) present in the catalyst, which can cause color

changes over time (5).

The degree of conversion (DC), which is the proportion of single carbon-carbon bonds in a polymer matrix to double carbon bonds between monomers (6), implies the conversion of monomers to polymers and is an index for the extent of polymerization. It has been shown that DC can affect the clinical performance of dental composites (6). An inadequate degree of conversion (e.g. low DC) might weaken bonding strengths, lower physicochemical properties, release toxic materials such as monomers and initiators (7), and cause permeability of bonded interfaces for resin-based materials. Suboptimal polymerization might lead to unfavorable changes, causing restoration detachment, caries formation, or discoloration around the adhesive, all of which are of clinical concern (8).

Usually, indirect restorations, including inlays, onlays, veneers, and crowns, have cervical margins intentionally placed in the gingival sulcus to meet esthetic and functional requirements (9). This leads to subgingival restorations that are often bonded without rubber dam isolation. However, it is well known that contamination by saliva, gingival crevicular fluid, or blood during luting procedures can negatively affect the adhesive properties of resin cements

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(10). To overcome this situation, gingival retraction cords immersed in different hemostatic solutions can be inserted into the gingival sulcus to promote chemomechanical clearance (11).

Chemical solutions used as hemostatic agents, and usually containing aluminum chloride, ferric sulfate, or epinephrine, have the power to control bleeding and gingival flow, allowing practitioners to work in a relatively dry operative field. Aluminum chloride acts on the contraction of blood vessels, inducing the precipitation of mucosal proteins, therefore decreasing capillary fragility. In turn, iron sulfate is a concentrated form of astringent, causing superficial and local coagulation (12). The use of hemostatic agents can be important during luting procedures to avoid blood contamination along the prepared tooth interface and into the luting agent. However, some studies have indicated that hemostatic agents can remain on the tooth surface, causing a significant decrease in bond strength to dentin (13). In addition, previous studies have demonstrated that hemostatic agents are highly acidic, with pH varying from 0.7 to 3.0. The acidity of these solutions may also contribute to changes in the dentin surface (14).

Thus, the aim of this study was to evaluate the degree of conversion, color stability, chemical composition, and bond strength of a light-cured resin cement contaminated with different hemostatic solutions containing aluminum chloride or ferric sulfate. The null hypothesis tested was that the hemostatic solutions would not alter the chemomechanical, the bonding interaction with ceramic or optical properties of the light-cured resin cement.

Material and Methods

The materials used in this study are shown in Table 1. The control group (CT) consisted of uncontaminated specimens of the light-cured resin cement (Variolink Veneer,

Shade +1, Ivoclar Vivadent, Schaan, Liechtenstein). For the experimental groups, the resin cement specimens were contaminated with three different hemostatic solutions, two of them containing aluminum chloride, Viscostat Clear (VC - Ultradent Products Inc., South Jordan, UT, USA) and Hemostop (HS - Dentsply Herpo, Petrópolis, RJ, Brazil), and one containing ferric sulfate, Viscostat (VS - Ultradent Products Inc.).

Specimen Preparation (Degree of Conversion, Color Stability and EDS Analysis)

The control group specimens were made by insertion of the light-cured resin cement into silicon molds. The material was covered with Mylar strips, followed by photoactivation with a LED curing unit (1000 mW/cm², Radium Plus, SDI, Victoria, Australia) for 40 s. For the experimental groups, specimens were made by the mixing of 10wt% of one of the hemostatic solutions with 100% of the resin cement (nearly 5 µL of hemostatic solution to 0.0775 g of cement) for 5 s, according to each experimental group. The contaminated resin cement was then placed into the silicon molds and photoactivated by the same protocol described above.

Cylindrical resin cement specimens (2 mm in height and 3 mm in diameter) were made in silicon molds for evaluation of the degree of conversion (n = 5) and color stability (n = 10). For x-ray energy-dispersive spectroscopy (EDS) analysis, specimens 6 mm in diameter and 3 mm in height were produced for each group (n = 1).

Degree of Conversion (DC)

First, a non-photoactivated portion of the resin cement from each group was subjected to initial readings. The cement was inserted into the crystal of a Fourier transform infrared spectrometer unit (Vertex 70, Bruker Co., Ettlingen, Germany). This first reading was considered the control, since it provided the values of the areas corresponding

Table 1. Materials used in the study

Material	Name	Composition	Manufacturer
Light-cured resin cement	Variolink Veneer, Shade +1	Dimethacrylates, silicon dioxide and ytterbium trifluoride. Additional contents: catalysts, stabilizers and pigments	Ivoclar Vivadent
Leucite-reinforced glass ceramic	IPS Empress CAD, Shade LTA1	SiO ₂ Additional contents: Al ₂ O ₃ , K ₂ O, Na ₂ O, CaO, and other oxides, pigments	Ivoclar Vivadent
Bonding agent	Monobond Plus	Ethanol, 3-trimethoxysilylpropyl methacrylate, Methacrylated phosphoric acid ester	Ivoclar Vivadent
Hydrofluoric acid	Condac Porcelana	Hydrofluoric acid 10% Water, thickener, surfactant and dye.	FGM
Hemostatic solutions	Hemostop	Aluminum chloride, propylene glycol, hydroxyquinoline, ethyl alcohol, water	Dentsply Herpo
	Viscostat	20% anion ferric sulphate	Ultradent Products
	Viscostat Clear	25% aluminum chloride	Ultradent Products

to the bands of the aromatic rings and aliphatic double bonds prior to polymerization of the material under study (reading of the monomer). The aliphatic carbon chains (C=C) corresponded to the intensity of the absorbance peak located at the 1638 cm⁻¹ band of each spectrum, while the 1608 cm⁻¹ band corresponded to the aromatic carbon chain (15). The analysis of the latter band, considered inherent to the material composition, was used as an internal control for calculation of the percentage of carbon double bonds remaining after polymerization (16). In this study, for the calculation of the DC, the absorption peak of the C=O ester groups (1716 cm⁻¹ band) was used as reference, because no aromatic peaks were identified for the resin cement tested. According to Gajewski VE et al., 2012 (17), composites containing urethane should be analyzed with the C=O bond (1716 cm⁻¹).

The measurement of the DC was then determined following 24 h of initial curing (photoactivation), performed for all groups. The infrared spectra were obtained in the crystal with the spectrum software (OPUS, version 6.5, Bruker) used in the microcomputer controlling the spectrometer. This software was used with a resolution of 4 cm⁻¹, and the spectra were collected in the range 1665–1580 cm⁻¹.

Color Stability

For the analysis of color stability, the specimens were ground flat with 320- to 600-grit silicon carbide papers for 30 s, immersed in an ultrasonic bath with deionized water for 10 min (Ultrasonic Cleaner, Odontobras, Ribeirão Preto, SP, Brazil), and then stored in artificial saliva. The immersion in artificial saliva aimed to simulate the oral environment and is frequently used in studies of color evaluation as the media for samples hydration and storage (18). The artificial saliva composition was chloramine 0.5%; potassium chloride 20.9 g; sodium chloride 28.8 g; magnesium chloride 1.86 g; calcium chloride 5.54 g; potassium phosphate 10.86 g; monobasic potassium phosphate 26.78 g; sorbitol 1.42 g; sodium benzoate 0.034 g and distilled water 1000 ml.

The color analysis was first carried out 24 h after specimen preparation, after which the specimens were stored in artificial saliva and analyzed again after 30 days and 6 months of storage. The readings assessed at 24 h were used as baseline for the analysis of color variation.

The baseline color coordinates were assessed in standard conditions by means of a reflectance spectrophotometer (CM-2600d, Konica Minolta, Osaka, Japan). The device was adjusted for the D65 light source, with 100% ultraviolet and specular reflection included. The observer angle was set at 2 degrees, and the device was adjusted to a small reading area (SAV), with a total area of 3 mm². The spectrophotometer was adjusted for three consecutive

readings, which were later averaged. The results of the color readings were quantified in terms of the L*, a*, b* coordinate values established by the *Commission Internationale de l'Eclairage* (CIELAB system), in which the L* axis represents the degree of lightness within a sample and ranges from 0 (black) to 100 (white). The a* plane represents the degree of green/red, and the b* plane represents the degree of blue/yellow (18). The measurement of color change after aging process was made calculating the variation (delta - Δ) of L* (ΔL), a* (Δa), and b* (Δb). The total color change (ΔE) was calculated according to the following formula (18).

$$\Delta E = (\Delta L^2 + \Delta a^2 + \Delta b^2)^{1/2}$$

Elemental Chemical Analysis

A specimen from each group was gold-sputtered (IN SCD050, Leica, Wetzlar, Germany) and subjected to elemental chemical evaluation performed by x-ray energy-dispersive spectroscopy (EDS/EDX, Oxford Instruments, Abingdon, Oxfordshire, UK) coupled to a scanning electron microscope (EVO MA 10, Carl Zeiss, Jena, Oberkochen, Germany). The equipment performs the analysis based on backscattered electrons, detecting qualitatively and semi-quantitatively the constituent chemical elements of the resin cement specimens and the chemical changes that occurred due to contamination with hemostatic agents. All readings were performed 24 h after specimen preparation.

Microshear Bond Strength Testing (μSBS)

Forty leucite-reinforced glass ceramic blocks (IPS Empress CAD LT A1, Ivoclar Vivadent) were used as the substrate for μSBS testing, with ten blocks per group. The blocks were embedded with polystyrene resin (Aerojet, Santo Amaro, SP, Brazil) in PVC cylinders, and the ceramic surfaces were ground flat with silicon carbide papers (#600-, 800-, 1200-grit, Norton, Campinas, SP, Brazil) for 20 s for surface standardization. The surfaces of the ceramic blocks were then etched with 10% hydrofluoric acid (Condac Porcelana, FGM, Joinville, PR, Brazil) for 60 s, cleaned with water spray for 1 min, and air-dried with an oil-free jet stream. A silane coupling agent (Monobond S, Ivoclar Vivadent) was then actively applied for 20 s and left to react for an additional 60 s. The light-cured resin cement was then used to fill small molds cut from micro-bore tygon tubing (TYG-030, Small Parts Inc., Miami Lakes, FL, USA), with internal diameter and height of approximately 0.75 and 1 mm, respectively. Six cylinders were placed in the glass-ceramic surface, and a separating space of 3.0 mm between the cylinders was established, since a safe distance was necessary to prevent unwanted stress from reaching the adhesive interface of the non-loaded cylinders during the test. The cylinders were photoactivated for 40 s each

according to the same protocol described previously. The tygon tubing was carefully removed with a scalpel blade, and the specimens were stored in artificial saliva. From the six cylinders placed, two were randomly selected for immediate testing after 24 h. The remaining specimens were stored in artificial saliva and tested following 1 month and 6 months of storage.

For the μ SBS test, a metallic jig was coupled to a mechanical testing machine (OM100, Odeme Dental Research, Luzerna, SC, Brazil), and the polystyrene cylinder containing the ceramic block was placed on the device so that the resin cylinder specimens were positioned perpendicular to the looped-orthodontic wire with a diameter of 0.2 mm (NiCr, Morelli, Sorocaba, SP, Brazil). This wire was placed around one of the resin cylinders, and the μ SBS test was performed by stressing specimens to failure under tension at 0.7 mm/min. The bond strength of each specimen (MPa) was calculated according to the following formula:

$$S=F/A$$

where F is the force required to failure (N), and A is the bonded area of the specimens (mm^2).

Failure Mode

The fractured surfaces resulting from the μ SBS test were cleaned in an ultrasonic bath and gold-sputtered for examination by scanning electron microscopy (EVO MA 10, Carl Zeiss). Failure modes were divided into five types: 1) adhesive failure; 2) mixed failure with prevalence in ceramic; 3) mixed failure with prevalence in resin cement; 4) cohesive failure in ceramic; and 5) cohesive failure in resin cement.

Statistical Analysis

After being analyzed for normal distribution, data from the color was analyzed by Repeated Measures ANOVA. μ SBS data were subjected to analysis of 2-Way ANOVA. Regarding degree of conversion, data was submitted to One-way ANOVA. The post hoc Tukey's test was performed

Table 2. Mean degree of conversion (%) and standard deviation (\pm) for the experimental and control groups

Groups	Means \pm SD	p value - Dunnnett's test
Control group	59.5 \pm 3.4	–
Hemostop	56.7 \pm 4.9 ^B	0.382
Viscostat Clear	57.4 \pm 1.9 ^B	0.583
Viscostat	65.5 \pm 1.5 ^A	0.026 [*]

Different letters indicate statistically significant difference among the experimental groups, ($p < 0.05$, Tukey's HSD test). * Indicate statistically significant difference between each experimental group compared with control group ($p < 0.05$, Dunnnett's test).

after all variance tests ($p < 0.05$). Also, One-way ANOVA and Dunnnett's test were used on μ SBS and DC to compare the control group (positive) with experimental groups. The failure modes were evaluated qualitatively followed by the chi-square test, and elemental chemical analysis (EDS) of the fracture surfaces was performed.

Results

Degree of Conversion (DC)

The results for the DC are shown in Table 2. Significant differences were verified among the groups ($p = 0.002$), with Viscostat (VS) presenting higher DC values than Hemostop (HS) and Viscostat Clear (VC). No significant differences were observed between HS and VC with the control group. However VS had significantly higher DC than control group (Table 2).

Color Stability

Figure 1 show the color readings quantified in terms of the L^* , a^* , b^* coordinate values established by the *Commission Internationale de l'Eclairage* (CIELAB system). The data for ΔE are presented in Table 3. The aging ($P < 0.001$) and interaction between contamination and aging ($P < 0.001$) influenced color stability. Tukey test demonstrated that HS and VC had significant lower ΔE than VS and CT, and no difference was found between ΔE values for CT and VS at 1 month. On the other hand, at 6 months the VS had significant lower ΔE values than HS, and VC had significant higher ΔE values than VS (Table 3). Color stability was found for the CT and VS groups, however, ΔE increased with time for the HS and VC groups.

Elemental Chemical Analysis (EDS)

Table 4 shows the overall results for the EDS performed on the specimens from each group. The elements carbon (C), nitrogen (N), oxygen (O), and silicon (Si) were detected in all groups. For the resin cement specimens contaminated with HS and VC, the presence of other elements, such as chlorine

Table 3. Mean DE and standard deviation (SD) for the experimental and control groups

Groups	Storage periods	
	1 month	6 months
Control group	2.94 \pm 0.97 ^{Aa}	3.34 \pm 1.17 ^{Ba}
Hemostop	1.90 \pm 0.68 ^{Bb}	5.40 \pm 2.35 ^{Aa}
Viscostat Clear	2.00 \pm 0.55 ^{Bb}	10.03 \pm 1.18 ^{Aa}
Viscostat	3.55 \pm 1.82 ^{Aa}	3.51 \pm 2.13 ^{Ba}

Means followed by the same letter (uppercase in columns and lowercase in rows) did not differ by Tukey's multiple-comparison test with 5% level of significance.

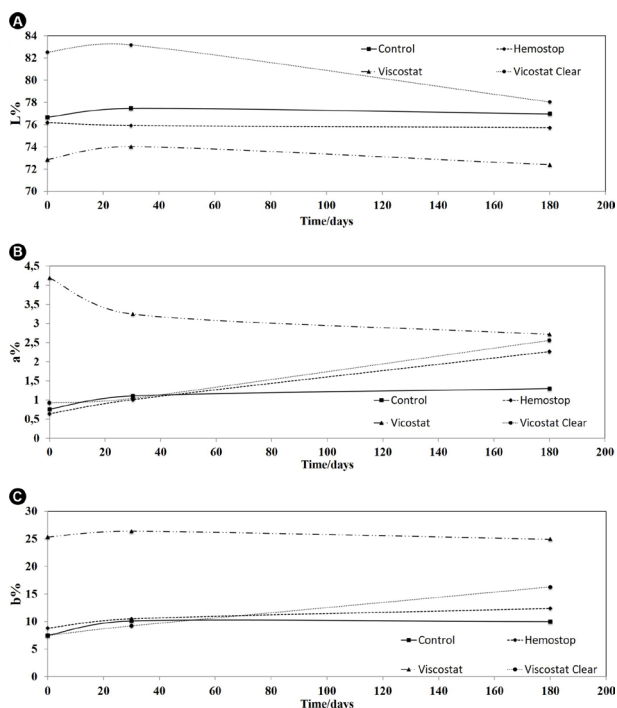


Figure 1. L* ΔL color change of light-cured resin cement contaminated with hemostatic solutions. The L* axis represents the degree of lightness within a specimen and ranges from 0 (black) to 100 (white). ΔL = measurement of color change after the aging a* and Δa = color change of light-cured resin cement contaminated with hemostatic solutions. a* represents the degree of green/red. Δa = measurement of color change after aging, and B* and Δb represent color change of light-cured resin cement contaminated with hemostatic solutions. b* represents the degree of blue/yellow. Δb = measurement of color change after aging.

Table 4. Elemental chemical constitution (%) of the groups

Groups	Chemical elements (%)									
	C	N	O	Si	Al	Cl	Ca	Fe	S	Total
Control	34.9	25.9	12.9	26.1	-	-	-	-	-	100.0
Hemostop	33.6	2.2	21.2	38.4	1.1	2.2	1.1	-	-	100.0
Viscostat Clear	32.5	11.9	11.1	40.5	1.9	1.2	0.4	-	-	100.0
Viscostat	47.4	20.8	3.1	26.1	-	-	-	0.2	2.07	100.0

C: carbon; N: nitrogen; O: oxygen; Si: silicon; Al: aluminum; Cl: chlorine; Ca: calcium; Fe: iron; S: sulfur.

Table 5. Mean and standard deviations of bond strength (MPa) in the study groups

Groups	Storage periods		
	24 h	1 month	6 months
Control Group	24.0 ± 3.1 ^{Aa}	26.1 ± 3.6 ^{Aa}	24.2 ± 3.6 ^{Aa}
Hemostop	14.2 ± 4.1 ^{Ab}	13.7 ± 4.1 ^{Ab}	11.1 ± 3.9 ^{Ab}
Viscostat Clear	11.2 ± 2.7 ^{Ac}	8.9 ± 4.6 ^{Ac}	9.3 ± 3.4 ^{Ac}
Viscostat	13.0 ± 2.9 ^{Ab}	11.2 ± 3.5 ^{Ab}	10.8 ± 3.1 ^{Ab}

Means followed by the same letter (uppercase in columns and lowercase in rows) did not differ by Tukey's HSD test (p<0.05).

(Cl), aluminum (Al), and calcium (Ca), was also verified. In the specimens contaminated by VS, the presence of iron (Fe) and sulfur (S) was also detected.

Bond Strength (μSBS)

The mean and standard deviation values of bond strength for the groups according to storage time are shown in Table 5. Significant effect was verified for the hemostatic agent factor (p<0.001), but no significant effects were observed for the time factor (p= 0.054) or for the interaction between hemostatic agent and time factors (p = 0.316). Dunnett's test showed that the control group had significantly higher bond strength than HS, VS and VC irrespective of time evaluated, 24h, 1 and 6 months. Comparing the experimental groups, Tukey test showed HS and VS had significant higher bond strength than VC, irrespective of the time evaluated, 24h, 1 and 6 months.

Failure Mode

The different types of failures observed in the specimens after the μSBS test are illustrated in Figure 2, and the failure mode distribution (%) for each group is shown in Figure 3. The failure mode was influenced by time (p = 0.021) and the type of hemostatic agent (p<0.001). A prevalence of mixed failures in ceramic was observed for the CT group. The HS group exhibited a prevalence of adhesive failures, while the VS and VC groups showed both adhesive and mixed failures in cement.

Discussion

The null hypothesis of this study was rejected, since the degree of conversion, color stability, and bond strength of the light-cured resin cement tested were influenced by the contamination with hemostatic solutions containing aluminum chloride or ferric sulfate.

In the present study, the DC was measured following 24h of specimen preparation, since the polymerization of resin-based materials can increase over time, (19) with higher values being reached after 24h (18). The results showed higher DC values for the VS group as compared with the other groups, which can probably be explained by the lower amount of oxygen observed for this group in the EDS chemical evaluation (Table 4). During polymerization, oxygen reacts rapidly with free radicals, and its presence slows the reaction. This probably did not occur in this group, allowing the polymerization mechanism to be more effective. Conversely, analysis of the EDS data showed a significant increase in the oxygen percentage for the HS group, which presented

lower DC. Again, this can probably be explained by the same mechanism, in which free radicals can react with oxygen, affecting the polymerization process (20).

Some studies that evaluated the effects of hemostatic agents on dentin bonding strength used direct contamination of the dentin (13,21). In our research, the hemostatic agent was mixed with the resin cement because, in clinical situations, the direct contact of the hemostatic solution can contaminate the resin cement first, rather than the ceramic substrate. This simulates what might happen, especially in the cervical region, where the possibility of contamination of the resin cement by hemostatic agents is real when these products are associated with retraction cords during luting procedures.

Regarding bond strength, it is known that hemostatic solutions can interfere with the bond strength to tooth structures (13). Therefore, the evaluation of the effects of such substances on the bond strength of resin cements to ceramic substrates also becomes important. When ceramic restorations are bonded to dental substrates, it is crucial that optimal adhesion of the resin cement to both the dentin and ceramic surfaces be ensured (22). Our findings showed that

the contamination of a resin cement by hemostatic agents reduced bond strength to the ceramic substrate, since the CT group showed better bond strength results (Table 5). These results indicate that the cement contamination with hemostatic solutions affected its chemical performance and decreases its bonding ability.

Additionally, it was suggested that a slight increase in the viscosity of resin-based materials may cause a decrease in their bond strength. In this study, despite the fact that the VS group presented a higher degree of conversion, the VS and VC groups had lower bond strength values, possible due their viscosity, which was not tested in this study, but should be included in future researches. Lower bond strengths may lead to detachment of the restoration on some level, leading to early failures, and consequently reducing the longevity of the restorative procedure. Thus, the integrity of dentin–cement and cement–ceramic interfaces is essential for the survival of adhesive restorations (22).

The distribution of failure modes showed the prevalence of adhesive and mixed failures, with a prevalence of mixed failures in the ceramic for the CT group, probably due to strong bonding at the adhesive interface (23). A prevalence

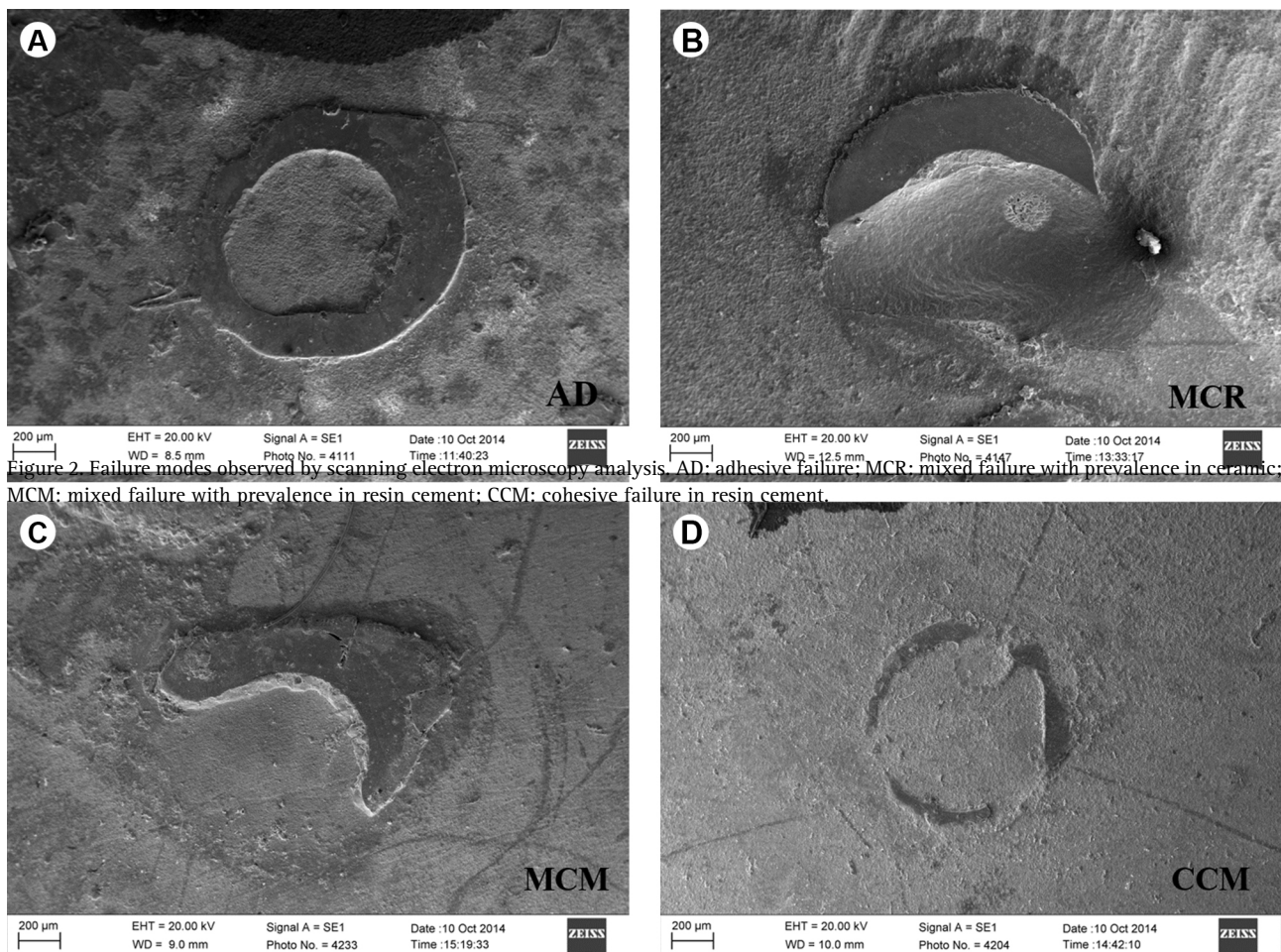


Figure 2. Failure modes observed by scanning electron microscopy analysis. AD: adhesive failure; MCR: mixed failure with prevalence in ceramic; MCM: mixed failure with prevalence in resin cement; CCM: cohesive failure in resin cement.

of adhesive failures was verified for the contaminated groups, followed by mixed failure predominantly in cement subjected to increased storage time in artificial saliva. This can be explained by a negative influence of hemostatic agents in the body of the resin cement over time. However, variations in the failure mode can be reflected by several factors, including the storage media, the test used for bond strength analysis, and the different contents of the restorative materials tested. Other laboratory tests and clinical trials are therefore needed to confirm the data obtained in this in vitro study.

The color stability of restorative materials is critical to meet suitable esthetic requirements. The aging process was used to examine the changes in the physical characteristics (color) of the materials tested over time. With respect to L^* (Fig. 1), which is responsible for the coordinated brightness between white and black, the VC group had higher values of L^* in the immediate analysis (toward white), while the VS group showed lower values (related to darkening of the specimens). These results were expected, since the specimens in the VC group were visibly lighter than the specimens of the other groups, and the VS group was already visibly more yellow, probably due to the presence of the iron element (Fe). The 6-month storage time favored the darkening of specimens of all groups, with a decrease of L^* . The analysis of coordinated a^* showed that the VS group had higher values of a^* (toward red). Nonetheless, after 6 months' storage, the high level of saturation presented by this group was reduced. The b^* coordinate analysis showed that the VS group has the darkest color (in the form of yellow), while the other groups have not shown significant differences initially. The storage time produced increased saturation toward yellow for all tested groups. The VC group showed greater instability of brightness and chromaticity (high values of ΔL , Δa , and Δb), HS also presented light instability (high values of ΔL), and VS changed by chromaticity (high

values of Δa). The total variation analysis of all parameters represented by ΔE revealed that the VC group, followed by the HS group, had more color instability after 6 months storage because ΔE had higher values. This result can be explained by the higher oxidation potential presented by aluminum, since, in both groups, contamination was created with agents containing aluminum chloride. Aluminum presents a low ΔG value (Gibbs free energy), which in fact, makes it more susceptible to rust easily (24). Furthermore, these substances contain hydroxyquinoline, which provided oxygen and light quinones, and thus may have caused darkening of the material.

According to Ruyter et al., (25) ΔE values smaller than 3.3 are considered acceptable for dental composites, while results higher than that are noticeable to the human eye. In this study, after 6 months of storage in artificial saliva, all groups except the Control group, which was considered a borderline case, showed ΔE values higher than 3.3, which means that the color change would be visible to the human eye. However, there is still controversy in the literature on determination of the visibility of the ΔE threshold (25).

Dental restorations are composed of complex color with various degrees of translucency, so color change considered clinically acceptable may also vary in relation to in vitro observations. Since we studied only one type of resin cement, further studies evaluating different cements and different ceramic materials contaminated with hemostatic agents should be conducted. The influence on the color stability of the resin cement should be analyzed carefully because we simulate the worst clinical condition that is the mixing of the hemostatic solution with resin cement. It is difficult to occur into the center of the buccal surface, however it is clearly possible to occur on the cervical margin. The marginal stain is a clinical relevant problem and may be caused by contamination between hemostatic solutions and resin cement during ceramic cementation. If the use of the hemostatic solution is essential to prevent the

blood contamination the adequate removal procedure of hemostatic solutions after the cords insertion on the gingival sulcus is fundamental to minimize the contamination preserve the bond strength between resin cement and ceramic restoration and the color stability of the ceramic restoration on the cervical margin.

Within the limitations of this study, the following may be concluded. The contaminating hemostatic agents reduced the bond strength, and altered the color stability of the tested light-cured resin cement. The contamination with HS and VC did not reduce the DC of the resin cement,

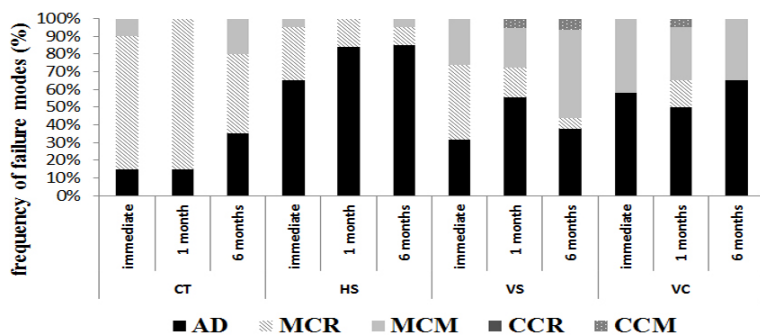


Figure 3. Frequency distribution of failure modes (%): AD, adhesive failure; MCR, mixed failure with prevalence in ceramic; MCM, mixed failure with prevalence in resin cement; CCR, cohesive failure in ceramic; CCM, cohesive failure in resin cement. *Statistically significant difference for hemostatic agents ($p < 0.001$) and time ($p = 0.021$).

and the contamination with VS increased the DC compared with control group. The control group presented higher bonding strength compared with that of the experimental groups, and the storage time in artificial saliva for 1 month and 6 months did not influence the results. The color stability of light-cured resin cement was influenced by storage time and interaction between contamination and storage time, with the agents containing aluminum chloride (VC and HS) presenting higher ΔE values after 6 months.

Resumo

O objetivo desse estudo foi avaliar o grau de conversão, estabilidade de cor, composição química e resistência de união de um cimento resinoso fotoativado contaminado com três soluções hemostáticas diferentes. Foram preparadas amostras para o grupo controle (cimento não contaminado) e grupos experimentais (cimento contaminado com uma das soluções hemostáticas) de acordo com os testes. Para o grau de conversão e análise de cor ($n=10$), as amostras (3 mm de diâmetro e 2 mm de espessura) foram avaliadas por espectroscopia de infravermelho com transformação de Fourier (FTIR) e espectrofotometria CIELAB (L^* , A^* , B^*), respectivamente. Para a análise química elementar ($n=1$), os espécimes (2 mm de espessura e 6 mm de diâmetro) foram avaliados por espectroscopia de energia dispersiva de raios-x (EDS). As resistências de união dos grupos foram avaliadas pelo ensaio de microcisalhamento ($n=20$) em um substrato cerâmico de vidro reforçado com leucita, seguida da análise de modo de falha por microscopia eletrônica de varredura (MEV). Os valores médios, com exceção da avaliação química e do modo de falha, foram avaliados por ANOVA e pelo teste de Tukey. A estabilidade de cor foi influenciada pelo tempo de armazenagem ($p<0,001$) e interação entre a contaminação e o tempo ($p<0,001$). A contaminação pelo Hemostat e Viscostat Clear não influenciaram no GC, porém a contaminação com Viscostat aumentou o GC. A resistência de união do cimento a cerâmica foi negativamente afetada pelos contaminantes ($p<0,001$). A contaminação por agentes hemostáticos afetou a resistência de união, o grau de conversão e a estabilidade de cor do cimento resinoso fotoativado testado.

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