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Micromorphology of resin–dentin interfaces using self-adhesive and conventional resin cements: A confocal laser and scanning electron microscope analysis

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ABSTRACT

Purpose: The aim of this study was to evaluate the resin–dentin morphology created by four dual-cured resin cements. Materials and Methods: Two self-adhesive resin cements (RelyX Unicem, 3 M ESPE and Clearfil SA Luting, Kuraray Med.) and two conventional resin cementing systems (RelyX ARC, 3 M ESPE and Clearfil Esthetic Cement, Kuraray Med.) were evaluated. Occlusal dentin surfaces of 32 extracted human third molars were flattened to expose coronal dentin. Teeth were assigned to 8 groups (n=4), according to resin cement products and microscope analysis (SEM: scanning electron microscope or CLSM: confocal laser scanning microscopy). For CLSM, two different fluorescent dyes, fluorescein isothiocyanate–dextran and rhodamine B, were incorporated into the adhesive system and resin cement, respectively. The resin cements were applied to indirect composite resin disks, which were cemented to dentin surface according to manufacturer's instructions. After 24 h, all restored teeth were vertically sectioned into 1-mm-thick slabs for SEM or CLSM analyses. Results: Scotchbond Multi-Purpose Plus/RelyX ARC cementing system formed a thin hybrid layer and resin tags penetration into the dentin tubules. Clearfil DC Bond/Clearfil Esthetic Cement showed only short resin tags. Neither hybrid layer nor resin tags were detected for self-adhesive resin cements. Conclusion: Representative SEM and CLSM images provided resin–dentin interfaces variability among resin cements studied.

1. Introduction

Resin cements are the most indicated as luting materials in the cementation of indirect composite resins and ceramic restorations to tooth structures [1,2]. The increased demand of esthetic treatment with metal-free restorations and the evolution of bonding/adhesive techniques are responsible for the widespread use of resin cements. These luting systems can be classified according to the bonding strategies: self-adhesive resin cements, which do not require a bonding agent and the conventional resin cements, which are used after an adhesive application [1,2,3].

The conventional cementing systems are used with etch-and-rise adhesives or self-etching primers. For the etch-and-rinse technique, a 30–40% phosphoric acid conditioner demineralizes the dentin surface and totally removes the smear layer and smear plugs to allow the monomer infiltration into the intertubular dentin and dentin tubules, and create the hybrid layer [4]. The resin cements that combine self-etching primers application prior to luting procedure do not require the conditioning and rinsing steps, because the self-etching primers contain acidic monomers [5]. The etching aggressiveness of each self-etching adhesive depends on the type of acidic functional monomers, such as carboxyl or phosphate groups [6,7]. On the other hand, the self-adhesive resin cement has been recently developed which does not require any dentin pretreatment [1,3,6].

Studies have focused in micromorphological analyses of bonded interfaces between tooth structures and adhesive systems [8,9,10] or resin cements [11,12] to provide further information about the correct use of bonding agents and resin cements in order to improve bonding efficiency and durability. The confocal laser scanning (CLSM) and scanning electron (SEM) microscopies have been routinely used to evaluate resin–dentin bonded interfaces and can be a important tool to study the interaction between self-adhesive resin cements and dentin surface [9,12], generating new information for this category of resin cements.

The purpose of this study was to evaluate by SEM and CLSM the features of resin–dentin interfaces of indirect resin composite restorations created by self-adhesive resin cements or bonding

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agents combined with their respective dual-curing cementing systems. The null hypothesis tested was that interfacial structures with hybrid layer and resin tags formation is not influenced by the type of resin cements regardless of the use of bonding agents for conventional resin cements.

2. Materials and methods

2.1. Specimen Preparation and Experimental Groups

This research protocol was approved by the Institutional Review Board of the Piracicaba School of Dentistry, Campinas State University (089/2009). Thirty-two freshly extracted, erupted, human third molars were stored in a saturated thymol solution for no longer than 3 months. The teeth were then transversally sectioned in the middle of the crown using a diamond blade saw (Buehler Ltd., Lake Bluff, IL) on an automated sectioning device (Isomet 2000; Buehler Ltd.) under water irrigation to expose the dentin. The exposed dentin surfaces were wet polished by machine (APL-4, Arotec, Cotia, SP, Brazil) using 600-grit SiC paper to create a flat surface with standardized smear layer formation before application of bonding agents. The prepared teeth were then randomly assigned to four groups according to products, so four teeth were prepared for SEM analysis and four teeth were prepared for CLSM analysis.

Four commercial dual-curing resin cements were used in this study: two self-adhesive resin cements (RelyX Unicem, 3M ESPE, Seefeld, Germany and Clearfil SA Luting, Kuraray Medical Inc., Kurashiki, Japan) and two conventional resin cements (RelyX ARC, 3M ESPE, St. Paul, MN, USA and Clearfil Esthetic Cement, Kuraray Med.), which were combined with a three-step, etch-and-rise adhesive (Adper Scotchbond Multi-Purpose Plus, 3M ESPE) and a single-step, self-etching adhesive (Clearfil DC Bond, Kuraray Med.), respectively. The composition of resin cement and adhesive system, classification, manufacturers, shade and lot number are described in Table 1.

Thirty-two pre-polymerized, light-cured composite resin disks (B2D shade, Sinfony; 3M ESPE) were prepared to simulate overlying laboratory-processed composite resin restorations [13]. The composite was placed into silicon molds (vinyl polysiloxane impression material, Express, 3M ESPE, St. Paul, MN, USA) to create resin disks with 2 mm thick and 10 mm in diameter. The Visio Alfa light curing unit (3M ESPE, St. Paul, MN, USA) was used for initial prepolymerization of the composite disk for 5 s and the Visio Beta Vario Light Unit (3M ESPE, St. Paul, MN, USA) was used during 15 min for final polymerization.

The surface of each disk was airborne-particle abraded with 50 μm aluminum oxide (Danville Engineering, Danville, VA, USA) for 10 s (air pressure: 0.552 MPa; distance from the tip: 1.5 cm) and silanated using coupling agents according to manufacturer directions (RelyX Ceramic Primer, 3M ESPE or Clearfil Ceramic Primer, Kuraray Med.).

When RelyX ARC was used, Adper Scotchbond Multi-Purpose Plus was previously applied to the dentin surface. According to

Table 1
Cementing systems, manufacturer, classification, shade, compositions and batch number of resin cements/adhesive systems used in this study (Information Supplied by the Manufacturer).

Cementing systems (manufacturer)	Classification	Shade	Composition (batch number)
RelyX ARC/Adper™ Scotchbond™ Multi-Purpose Plus (3M ESPE, St. Paul, MN, USA)	Dual-cured resin cement/3-step etch-and-rise adhesive system	A1	Scotchbond Multi-Purpose Plus: Activator: ethyl alcohol, sodium benzenesulfinate (N123538); Primer: water, HEMA, copolymer of acrylic and itaconic acids (N124894); Catalyst: bis-GMA, HEMA, benzoyl peroxide (N130621); RelyX ARC Paste A: silane treated ceramic, TEGDMA, bis-GMA, silane-treated silica, functionalized dimethacrylate polymer; 2-benzotriazolyl-4-methylphenol, 4-(dimethylamino)-benzeneethanol; Paste B: silane treated ceramic, TEGDMA, bis-GMA, silane-treated silica, functionalized dimethacrylate polymer, 2-benzotriazolyl-4-methylphenol, benzoyl peroxide (N149151).
Clearfil Esthetic Cement/Clearfil DC Bond (Kuraray Medical, Kurashiki, Japan)	Dual-cured resin cement/1-step self-etching adhesive system	Clear	Clearfil DC Bond: Liquid A: HEMA, bis-GMA, dibenzoyl peroxide, 10-MDP, colloidal silica, dl-camphorquinone, initiators, others (00271A); Liquid B: ethanol, water, accelerators, catalysts (00146B); Clearfil Esthetic Cement: Paste A & B: bis-GMA, TEGDMA, hydrophobic aromatic dimethacryate, hydrophilic aliphatic dimethacrylate, silanated silica filler, silanated barium glass filler, colloidal silica, dl-camphorquinone, catalysts, accelerators, pigments (0015AB).
RelyX Unicem (3M ESPE, St. Paul, MN, USA)	Dual-cured self-adhesive resin cement	A2	Base: glass power, methacrylated phosphoric esters, TEGDMA, silane-treated silica, sodium persulfate. Catalyst: glass power, dimethacrylate, silane-treated silica, TEGDMA, dental glass (394291)
Clearfil SA Cement (Kuraray Medical, Kurashiki, Japan)	Dual-cured self-adhesive resin cement	A2	Paste A & B: bis-GMA, sodium fluoride, TEGDMA, 10-MDP, hydrophobic aromatic dimethacryate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, silanated colloidal silica, dl-camphorquinone, initiators, catalysts, pigments, others (0005AB).

Abbreviations: HEMA: 2-hydroxyethyl methacrylate; TEGDMA: triethylene glycol dimethacrylate; bis-GMA: bisphenol A diglycidyl ether methacrylate; 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate.

3M ESPE manufacturer, dentin was etched with 37% phosphoric acid (Scothbond Etchant) for 15 s, followed by rinsing and surface moist control. The etched surfaces were rinsed with air/water spray for 10 s, followed by air-drying for 3 s and the water excess removed with absorbent paper (Kleenex, Kimberly-Clark, Mogi das Cruzes, SP, Brazil).

Afterwards, the Activator, Primer, and Catalyst were consecutively applied to the moist dentin surface. For Clearfil DC Bond/ Clearfil Esthetic Cement, equal amounts of DC Bond Liquid A & B were mixed for 10 s. The mixed solution was applied to the dentin surface with a microbrush and the mixture was left undisturbed for 20 s. After air blowing (10 s), the self-etching adhesive was light-cured for 20 s prior to resin cement application.

RelyX Unicem self-adhesive resin cement was applied in the resin disk after base and catalyst pastes were dispensed on the mixing pad and mixed during for 20 s. For Clearfil SA Luting, base and catalyst pastes were dispensed with the automix syringe and applied to the indirect resin composite. The self-adhesive resin cements do not require any dentin pre-treatment. For the self-adhesive resin cements, the dentin surfaces were wet. After the exposed dentin were wet polished, the flat surfaces to be bonded were rinsed with air/water spray for 10 s, followed by air-drying for 3 s and the water excess removed with absorbent paper (Kleenex, Kimberly–Clark, Mogi das Cruzes, SP, Brazil).

The mixed resin cements pastes were applied to the resin composite pre-polymerized disk, which was placed on the dentin surface with 500 g load, the excess was removed and the restoration was light-cured from their buccal and lingual aspects for 40 s (XL 3000; 3M ESPE, St. Paul, MN, USA). A minimal output intensity of 580 mW/cm^2 was used and was constantly measured with a radiometer Curing Radiometer, Model 100, Kerr Corp Orange, CA, USA). After removal of the load, additional 40 s of light exposure was performed also on mesial and distal surfaces. The restored teeth was dark stored in relative humidity for 24 h at 37 °C.

2.2. Scanning Electron Microscopy Analysis

To observe dentin interface by SEM, restored teeth were sectioned in the mesial-distal direction under water cooling into four 1.2-mm thick slabs with a slow-speed diamond saw (Isomet 1000, Buehler Ltd., Lake Bluff, IL, USA). Each slab was wet polished with 600-, 1200- and 2000-grit SiC paper. Afterwards, the specimens were polished using soft cloths and diamond pastes of decreasing abrasiveness (6, 3, 1 and ¼ μm). Slabs were etched with 37% phosphoric acid solution for 10 s to remove the mineral content, washed with water and immersed in 5% sodium hypochlorite for 5 min to remove exposed collagen from the dentin surface, respectively. Finally, specimens were subjected to ultrasonic bath with distillated water for 10 min and allowed to dry overnight at 37 °C. Specimens were then sputter coated with gold (MED 010, Balzers Union, Balzers, Liechtenstein) and observed using a scanning electron microscope (VP 435, Leo, Cambridge, England) at x1.000 magnification.

2.3. Confocal Laser Scanning Microscopy Analysis

For this analysis, two different fluorescent dyes, fluorescein isothiocyanate–dextran (Fluorescein–Isothiocyanate–Dextran, Sigma, St. Louis, MO, USA) and rhodamine B (Rhodamine B, Sigma), were incorporated into the adhesive systems and resin cements, respectively. Fluorescein was incorporated into adhesive systems tested (40 μg/mL) [11]. This amount of fluorescein was added to each bottle of Adper Scotchbond Multi-Purpose Plus Adhesive (Activator, Primer and Catalyst) and to Clearfil DC Bond (Liquid A & B). The dye was mixed directly in the supplied bottle using a mixing device (Vortex Machine, Scientific Industries, New York, NY, USA) during 6 h to measure the complete dye dissolution. Rhodamine was added to the base resin cement paste of all resin cements and mixed to obtain a paste with uniform shade (0.32 μg/mg) [11].

The teeth were restored as previously described. Restored teeth were stored in vegetable oil for 24 h and were vertically (mesial-distal direction) sectioned under vegetable oil lubrification (Liza Pure Vegetable Oil, Cargill Agrícola S.A., Mairinque, SP, Brazil) into four 1.2-mm thick slabs with a slow-speed diamond saw (IsoMet 1110, Buehler Ltd.). Afterwards, slabs were wet polished with 600-, 1200- and 2000-grit SiC paper, were storage in vegetable oil and were analyzed under CLSM (LSM 510 Meta Confocal Microscope, Zeiss; Göttingen, Germany). An argon laser at 488 nm and He–Ne laser at 543 nm provided excitation energies, so images were obtained in dual fluorescence mode using a 25X objective and were analyzed by AxioVision LE software (Zeiss).

2.4. Data Analysis of Dentin–Resin Cements Interfaces

The four sections from each tooth were analyzed using SEM and Confocal Laser Scanning Microscopy. Several pictures were taken from the mesial side of dentin–resin cement interfaces until distal side of each slab. Thus, entire interfaces of all slabs from each tooth were observed under scanning electron and confocal laser microscopies. All figures obtained from SEM and Confocal Laser Scanning Microscopy were analyzed by 3 observers in order to avoid individual bias, but only representative images for each group were selected to represent the characteristic of each dentin–resin cement interfaces formed by the resin cement tested.

3. Results

Representative SEM and CLSM images are shown in Figs. 1–4. Once this study was based on a qualitative analysis, only visual differences among groups were considered and described as findings. For Adper Scotchbond Multi-Purpose Plus/RelyX ARC, a thin hybrid layer and resin tag formation was clearly visible (Fig. 1d), while short resin tags were observed for Clearfil DC Bond self-etching system (Fig. 2d). No dentin hybridization process and no resin tags inside the dentinal tubules were noted for self-adhesive resin cements. Also, the intimate and uniform contact was not seen between dentin and resin cement, because some bubbles or voids were presented at the interfaces (Figs. 3a and 4a).

CLSM images allowed visualization the adhesive system (Figs. 1a and 2a) and resin cements (Figs. 1b and 2b). The adhesive, which was labeled with fluorescein (Figs. 1a and 2a), showed green fluorescence, while the resin cement, that was stained with rhodamine B, exhibited red fluorescence (Figs. 1b, 2b, 3b and 4b). For the Adper Scotchbond Multi-Purpose Plus/RelyX ARC, it was not possible to distinguish the adhesive and resin cement layers, when the fluorescence dyes were added to the bonding agent and resin cement and when they were analyzed together (Fig. 1c). The resin cement diffusion into adhesive layer and its mixture with labeled adhesive produced orange color fluorescence at dentin–resin cement bonded interface.

Clearfil DC Bond/Clearfil Esthetic Cement showed monomer diffusion into the dentinal tubules (Fig. 2a,c) and into the resin cement layer (layer above) with green color predominance (Fig. 2c). For this cementing system, it was possible to detect the bonding between adhesive and resin cement, but in some figures it was observed dark gaps between dentin and resin cement as artifacts. A thin orange line can be seen, which

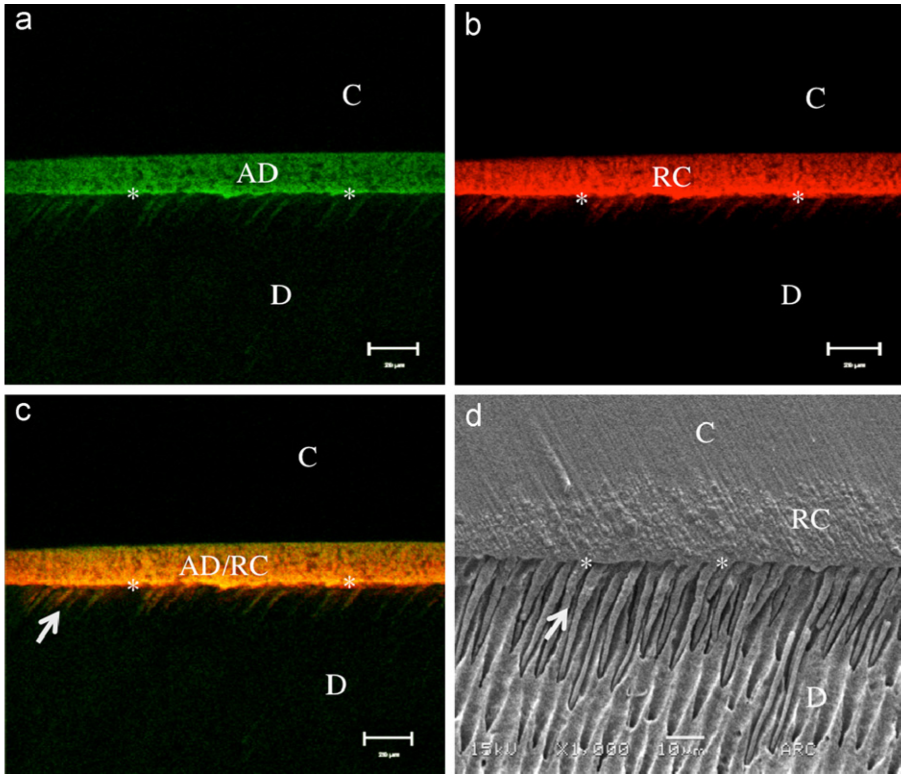

Fig. 1. Bonding of indirect composite (C) to dentin (D) using Adper Scotchbond Multi-Purpose Plus adhesive system (AD) and RelyX ARC resin cement (RC). The hybrid layer (*) and tag formation (arrows) can be clearly observed (c and d). For CLSM images, the adhesive was labeled with fluorescein (a) and the resin cement was stained with rhodamina B (b), showing green and red fluorescence colors, respectively. The c demonstrates an orange layer, which correponded to the mixture bewteen the adhesive (green/AD) and resin cement (red/RC), showing no distinction between adhesive and resin cement (AD/RC). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

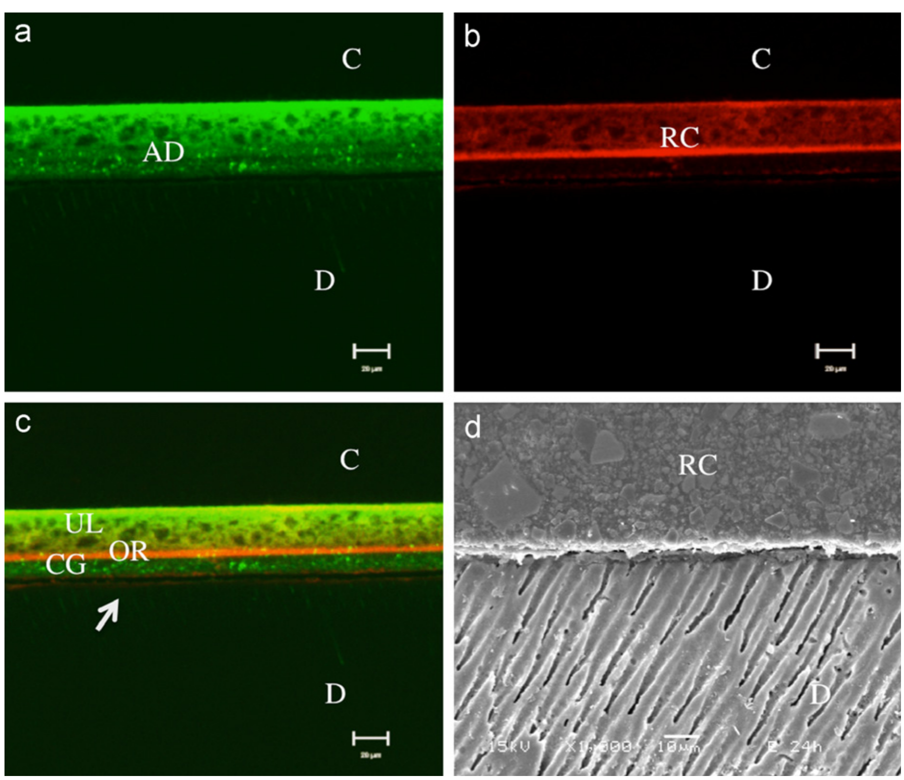

Fig. 2. Bonding of indirect composite (C) to dentin (D) using Clearfil DC Bond/Clearfil Esthetic Cement (RC). For CLSM images, the adhesive was labeled with fluorescein (a) and the resin cement was stained with rhodamina B (b), showing green and red fluorescence color respectively. (a) and (c) demonstrate short resin tags (arrows) formation of the green-labeled adhesive. Also, it is possible to verify three layers, according to the green intensity: upper green layer (UL) correspond to the resin cement infiltrated by adhesive, meddium layer (orange line—OR) is the mixture bewteen adhesive (green) and red-labeled resin cement, and the clear green layer (CG) (botton) that is the self-etch in contact with dentin. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

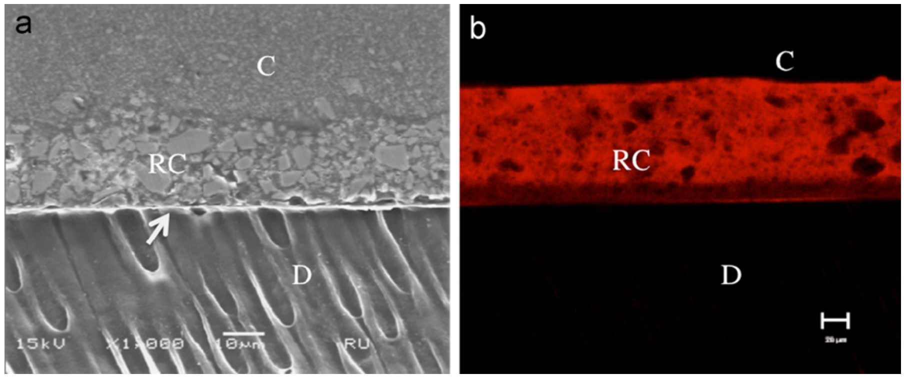

Fig. 3. Bonding of indirect composite (C) to dentin (D) using RelyX Unicem self-adhesive resin cement (RC). No distinction hybrid layer and resin tags were detected. The arrow show the contact between the dentin and resin cement (a).

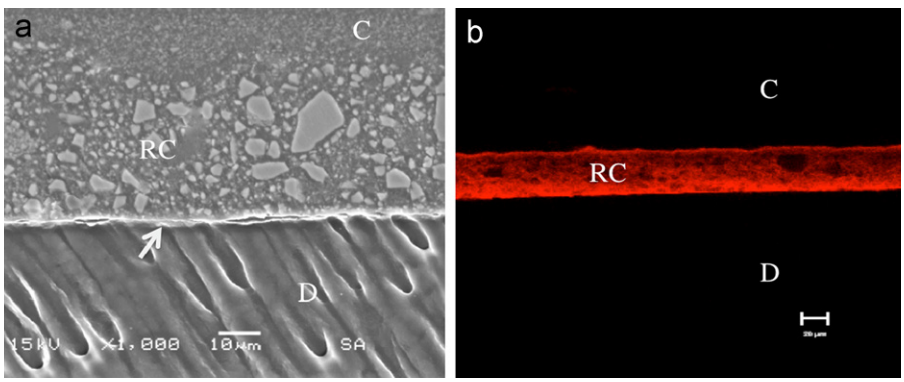

Fig. 4. Bonding of indirect composite (C) to dentin (D) using Clearfil SA Luting resin cement (RC). No distinction hybrid layer and resin tags were detected. The arrow show the contact between the dentin and resin cement (a).

corresponded to an overlap (adhesive and resin cement mixture) (Fig. 2c).

For self-adhesive resin cements, SEM and CLSM images showed the interaction between the dentin and the luting materials (Figs. 3 and 4). No diffusion of rhodamine (and resin cement) into the intertubular dentin or dentinal tubules were observed. Thus, the formation of hybrid layer and resin tags was not detected for RelyX Unicem (Fig. 3a and b) and Clearfil SA Luting (Fig. 4a and b).

4. Discussion

The CLSM is a valuable technique to visualize interfacial structures [9], once it provides more detailed information than SEM analysis [12]. This method does not require elaborate specimen preparation, decreasing the risk of dehydration, shrinking and other artifacts, such as gap formation during coating or under SEM observation [14]. In addition, it allows researchers to capture images from the specimen subsurface, reducing the effects of surface contamination [15].

Fluorescent agents promote specific emission wavelength for each resinous component when they are excited by laser with specific wavelengths [11]. Rhodamine B is frequently used as fluorochrome for the analysis of bonded interfaces [9,11,12,15], it is stable under various pH conditions, soluble in organic solutions [9] and displays a red-color characteristic [15]. On the other hand, fluorescein isothiocyanate–dextran was added to the adhesive composition emitted a green color [11].

The type of bonding agent is determinant for the formation of interfacial structures. While self-etching etching adhesives promote the formation of a thin hybrid layer (0.5–2 μm), etch-and-rinse systems produced hybridization higher than 2 μm [16,17]. Representative SEM images of the Adper Scotchbond Multi-Purpose Plus Adhesive/RelyX ARC showed thin hybrid layer (3–5 μm thickness) and longer resin tags than those created by DC Bond/Clearfil Esthetic Cement. In general, this hybrid layer is equivalent to the demineralization depth promoted by phosphoric acid etching. On the other hand, the thickness of the interaction zone for self-etching primers depends on etching aggressiveness of acidic monomers from bonding agents within this category, which range from mild to strong [5,6,7]. The interfacial morphology of different cementing systems observed in SEM and CSLM images corroborates with the data of previous published articles [11,16,18,19,20].

The orange layer observed in Fig. 1c was created by the mixture of uncured bonding agent (Adper ScotchBond Multi-Purpose Plus) and resin cement (Rely X ARC). According to 3M ESPE manufacturer, adhesive components must be applied and left in the uncured state prior to the RelyX ARC application, consequently the mixture of all agents occurs prior to light activation. As rhodamine penetrated into dentinal tubules, the cured of the mixture of these materials formed the resin tags. Also, some components of the resin cement seemed to form the hybrid layer, since the red fluorescence dye was presented in this interface area. Arrais et al. (2009) [11] also reported resin cement penetration within the hybrid layer and into the dentin tubules, depending on the category of cement system.

Conversely, the light-activation of Clearfil DC Bond self-etching bonding agent did not allow a massive penetration of the components of the Clearfil Esthetic Cement into dentin. This self-etching forms short resin tags (Figs. 2a and 2c) and a thin hybrid layer (less than 0.5 μm), which was not identified by microscopy methods used in this study. However, the adhesive was able to infiltrate and blend with the resin cement, which is the main function of this type of bonding agent. This mixture resulted in predominance of green fluorescence (Fig. 2c). The self-etching adhesive contains self-curing components or co-initiators, such as the dibenzoyl peroxide, which are important to increase the degree of conversion of resin cements [21]. A thin orange line represents the mixture between adhesive from oxygen-inhibited layer and resin cement (Fig. 2c).

No diffusion of the rhodamine B dye into the dentin was observed using CLSM methodology. Also, no hybrid layer formation and resin tags were detected for RelyX Unicem (Fig. 3a and b) and Clearfil SA Luting (Fig. 4a and b). These findings are in agreement with previous investigations regardless of the self-adhesive resin cements [6,7,18,19,20].

Studies have suggested that RelyX Unicem show the best performance when used in wet dentin [22] and when a strong seating load of restoration is applied during the luting procedure [6,7]. However, the seating load used in this study (500 g) was not enough to improve superficial contact between dentin and resin cements, since some voids or bubbles were seen at the interface.

The bonding mechanism of self-adhesive resin cements to mineralized dental tissues is related to chemical reaction with hydroxyapatite. The Clearfil SA Luting contains 10-MDP monomer (10-methacryloyloxydecyl dihydrogen phosphate), which has the ability to form strong ionic bond with calcium of enamel and dentin [23]. For RelyX Unicem, the bonding mechanism of self-adhesive resin cements involves phosphoric acidic methacrylates that react with the basic components, i.e., filler particles in the luting cement and hydroxyapatite in tooth tissue. After initial mixing, the cement is very acidic, however, the pH-value tends to increase according to the changes from hydrophilic to a hydrophobic features of resin cement [2]. Thus, the interfacial structure of resin cements depends on the approach of each luting material and can vary from micromechanical retention provided by hybridization to superficial interaction with chemical reaction between resin-based material and mineralized dental tissues. Therefore, the hypothesis tested in the current study must be rejected.

5. Conclusion

The SEM and CLSM images identified the interfacial structures of self-adhesive resin cements and conventional cementing systems involved in the bonding to dentin. Only the conventional resin cement combined with the three-step, etch-and-rise adhesive showed typical hybrid layer and resin tags formation, while neither resin tags nor hybridization were detected for self-adhesive resin cements.

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