REVISTA DE ODONTOLOGIA DA UNESP

ORIGINAL ARTICLE

Rev Odontol UNESP. 2017 Sept-Oct; 46(5): 249-254
Doi: http://dx.doi.org/10.1590/1807-2577.04917

Ultramorphology of pre-treated adhesive interfaces between self-adhesive resin cement and tooth structures

Ultramorfologia de interfaces adesivas entre cimento resinoso auto-adesivo e estruturas dentárias pré-tratadas

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Resumo

Introdução: Restaurações estéticas indiretas são preferencialmente cimentadas utilizando-se cimentos resinosos convencionais e sistema adesivo de condicionamento total ou cimentos resinosos autoadesivos. Estes últimos são tecnicamente menos sensíveis e aderem aos tecidos dentários sem tratamento prévio ou aplicação de adesivo, com um único passo para sua aplicação aos tecidos dentários. **Objetivo:** Comparar qualitativamente as interfaces adesivas de dois cimentos resinosos autoadesivos e um cimento resinoso convencional, sob microscopia eletrônica de varredura. **Material e método:** 42 coroas de incisivos bovinos foram seccionadas e as faces vestibulares planificadas expondo esmalte (E) ou dentina (D). Os subgrupos foram definidos de acordo com o tipo e tempo de condicionamento: E1-sem tratamento, E2-37% de ácido fosfórico por 15 segundos, E3-37% de ácido fosfórico por 30 segundos; D1-sem tratamento, D2-37% de ácido fosfórico durante 5 segundos; D3-11,5% de ácido poliacrílico durante 15 segundos. Um bloco de resina foi unido a cada substrato usando os cimentos resinosos autoadesivos RelyX U100 e RelyX U200 (3M ESPE) (n=3). Como referência de camada híbrida, foram cimentados seis blocos de resina com RelyX ARC e o sistema adesivo Scotchbond Multi-Purpose(esmalte-EA, dentina-DA). Após armazenamento (7 dias, umidade, 37±1°C), as amostras foram preparadas para análise microscópica. **Resultado:** Nos espécimes ARC, houve formação de camada híbrida em EA e DA. U100 E1 mostrou lacunas na interface adesiva, enquanto E2 e E3 apresentaram boa interação para ambos os cimentos autoadesivos. Houve interação superficial com U100 e U200 em D1, enquanto em D2 e D3, foram observadas tags de resina apenas para U100. **Conclusão:** Concluiu-se que o condicionamento do substrato pode aumentar a interação entre cimentos resinosos autoadesivos e os tecidos dentários, embora este não seja o caso do RelyX U200 e da dentina.

Descritores: Dentina; condicionamento dentário; cimento resinoso; microscopia eletrônica de varredura.

Abstract

Introduction: Convencional resin cements can be used in combination with a total-etch system in a conventional mode or as self-adhesive resin cements. The latter are less technique sensitive and able to bond to dental tissues without previous treatment or adhesive layer and requires only a single step to be applied to dental structures. **Objective:** To compare qualitatively the adhesive interfaces of two self-adhesive resin cements and one conventional resin cement after different tooth surface treatments under scanning electron microscopy. **Material and method:** 42 crowns of bovine incisors were sectioned and flattened exposing enamel (E) or dentine (D) substrate. Subgroups were defined according to conditioning type and time: E1—no treatment, E2—37% phosphoric acid for 15 seconds, E3—37% phosphoric acid for 30 seconds; D1—no treatment, D2—37% phosphoric acid for 5 seconds; D3—11.5% polyacrylic acid for 15 seconds. A resin block was bonded to each substrate using the self-adhesive resin cements RelyX U100 (3M ESPE) and RelyX U200 (3M ESPE). As a reference hybrid layer, six resin blocks were luted with RelyX ARC and Scotchbond Multi-Purpose adhesive system (3M ESPE) (enamel—EA; dentine—DA). After aging for 7 days in a moist environment at 37±1°C, samples were prepared for microscopy analysis. **Result and Discussion:** In the ARC specimens, there was hybrid layer formation in both EA and DA. U100 E1 showed gaps at the adhesive interface, while

E2 and E3 showed interaction for both self-adhesive cements. There was superficial interaction with bothU100 and U200 in D1, while in D2 and D3, resin tags were only observed in the case of U100. **Conclusion:** It was concluded that substrate conditioning may enhance the interaction between self-adhesive resin cements and dental tissues, although this is not the case for RelyX U200 and dentine.

Descriptors: Dentin; dental etching; resin cement; scanning electron microscopy.

INTRODUCTION

Resin-based cements are widely used for luting inlays, onlays, and veneer restorations. Conventional resin cements are based on etch-and-rinse technique, which require treatment of the dental structure before application of the low-viscosity composite resin[1]. Thus, clinicians must be competent in the highly sensitive technique of luting, as well as in the use of different materials and procedures, which vary depending on the adhesive system chosen. To minimize these problems and reduce the sensitivity of the technique, self-adhesive luting material, which involves only one step, has been introduced. The manufacturers of self-adhesive resin cements claim that they are suitable for all restorative materials[2]. However, despite significant improvements in adhesive dental materials, the bonding interface remains the main weakness of dental restorations[3].

Self-adhesive cements undergo a micromechanical bonding with dental substrate and chemical reaction with the calcium ions in hydroxyapatite[4]. Additionally, their simplified application have shown bond strength to dentine, but not to enamel, that is similar to those of conventional resin cements[1,2]. However, six self-adhesive resin cements had lower dentine bond strength values than conventional resin cements with etch-and-rinse adhesives[5]. Notwithstanding the technical simplicity of self-adhesive resin cement application, the absence of conditioning may create a limited decalcifying substrate, harming the diffusion of resin monomers into the dentine[6]. It was suggested dental substrate treatment that can enhance both the hybrid layer and bond strength results. Furthermore, it is not yet clear whether enamel conditioning with phosphoric acid is clinically required before luting with self-adhesive cements[3,6-12].

Previous formulations of self-adhesive resin cements (RelyX Unicem and RelyX U100, 3M ESPE) have a high viscosity and therefore require a greater cementation pressure[1,13]. To compensate this important limitation, rheological properties were changed in the new cement (RelyX U200, 3M ESPE) maintaining the original chemical properties while decreasing the viscosity.

The aim of this study was to carry out an ultramorphological characterization of the different resin cements adhesive interfaces with enamel and dentine. The adhesive interfaces of two self-adhesive resin cements and a conventional resin cement after different surface treatments were evaluated under scanning electron microscopy (SEM). The hypothesis suggested was that substrate treatments affect the dentine and enamel hybrid layer.

MATERIAL AND METHOD

Crowns of 42 bovine incisors were sectioned using a diamond disc under air–water cooling. The incisors were then split into two groups: the enamel group (E), in which the buccal faces of the incisors were flattened and wet polished with 200, 320, 400, and 600-grit SiC paper (Norton S.A., São Paulo, SP, Brazil), and in the dentine group (D) the buccal surrounding enamel was removed using diamond burs (#2214; KG Sorensen, Cotia, SP, Brasil) and the dentine surfaces were flattened and polished as described above. Within the enamel group, three subgroups were created for each cement (n=3): E1—no treatment; E2—etched with 37% phosphoric acid (Condac, FGM) for 15 seconds; E3—etched with 37% phosphoric acid for 30seconds. For the dentine group, three subgroups (n=3) were created for each cement: D1—no treatment; D2—etched with 37% phosphoric acid for 5 seconds; D3—conditioned with 11.5% polyacrylic acid solution (dentine conditioner; Vidrion, SS White, RJ, Brazil) for 15 seconds under friction. As a reference, one enamel subgroup (E/ARC) and one dentine subgroup (D/ARC) (n=3) were treated using a conventional resin cement RelyX ARC/Scotchbond Multi-Purpose Plus (ARC/SBMP; 3M ESPE; St. Paul, MN, USA).

Forty-two composite resin blocks (5 × 5 × 2 mm) were prepared with a microhybrid composite resin layered into a silicon mold. Photoactivation was performed using a light-emitting diode (1300mW/cm^2; Bluephase, IvoclarVivadent) for 60 seconds. One side of the resin blocks was abraded with 600-grit SiC paper under water cooling to create a flat surface with standardized roughness. The blocks were then thoroughly rinsed with distilled water and dried at room temperature. A thin SBMP adhesive layer was applied.

Two self-adhesive resin luting agents—RelyX U100 (U100) (3M ESPE, St, Paul, MN, USA) and RelyX U200 (U200) (3M ESPE, St. Paul, MN, USA) were mixed according to the manufacturer's instructions and applied to the dental surface. The pre-cured resin block was positioned and pressed onto the cement using5N load and excess cement was removed with microbrush. The resin cement was light cured for 20 seconds on each block side, and then for 120 seconds through the resin block, (1300mW/cm^2; Bluephase, IvoclarVivadent). In RelyX ARC groups (ARC), the resin blocks were cemented using SBMP according to the manufacturer's instructions. All specimens were stored in distilled water (37°C, 7 days) and sectioned across the adhesive interface on a cut machine fitted with a double-sided diamond disc (Isomet 1000; Buhler, Lake Bluff, IL, USA). Samples were embedded in resin (Crystal Orthophthalic Resin; Belo Horizonte, Brazil), and after 24 hours the surfaces were polished under water irrigation (Polisher APL-4; Arotec, Cotia, SP, Brazil) with 600- and 1000-grit silicon carbide sandpaper, 1200-, 2000-, and 2500-grit Al_2O_3 sandpaper (Carborundum Abrasives, Pernambuco, PE, Brazil). The samples were underwent superficial demineralization with 50% phosphoric acid for 3 seconds, rinsed in running water for 1 minute, and deproteinated by immersion in 2.5% NaOCl for 10 minutes. Subsequently, they were rinsed three times with distilled water and sequentially immersed in ethanol solutions (25%, 50%, 75%, 95%, and 100%) for approximately 20 minutes per solution. The immersion in 100% solution was repeated three times for 10 minutes each. After drying at room temperature for 10 minutes, the samples were placed into a hermetically sealed container with silica gel for at least 24 hours before carbon-sputtering under a low-vacuum (Balzers SCD 050). Analysis was performed using a low-vacuum scanning electron microscope (FEG–FEI; FEG Quanta 200F) at an accelerating voltage between 15 and 30 kV. The images were obtained in increasing magnifications to highlight the morphological characteristics of the surfaces.

RESULT

RelyX ARC resin cement samples showed an interaction between the adhesive system and the enamel–dentine hybrid layer (Figure 1). Analysis of RelyX U100 specimens showed gaps in the adhesive interface between the resin cement and the unetched enamel (Figure 2: U100-E1). There was interaction between the cement and the enamel etched for 15 seconds (Figure 2: U100-E2) and 30 seconds (Figure 2: U100-E3). Analysis of RelyX U200 specimens showed that the unetched enamel lacked a full adhesive interface (Figure 2:U200-E1). The interaction was more effective when the phosphoric acid was applied for 30 seconds compared with 15 seconds (Figure 2: U200-E2 and U200-E3). The unconditioned dentine surface bonded with RelyX U100 resin cement had a superficial interaction (Figure 3: U100-D1). Furthermore, the images of the etched dentine showed that both treatments yielded cement tags in the dentine tubules (Figure 3: U100-D2 and U100-D3). The interface analysis showed a superficial interaction between RelyX U200 resin cement and unetcheddentine (Figure 3: U200-D1). RelyX

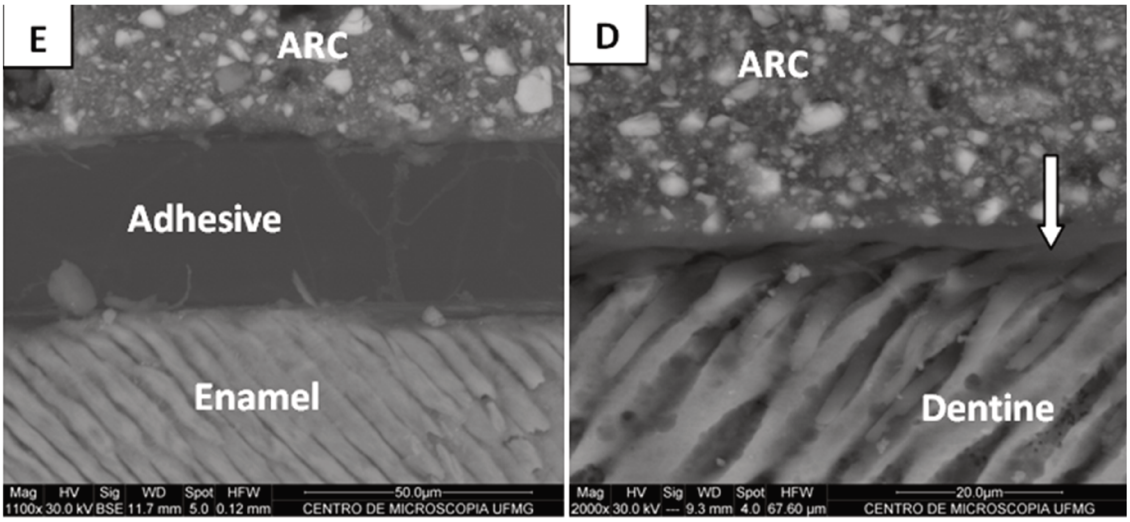

Figure 1. RelyX ARC interface after total-etch (phosphoric acid 37%) and Scotchbond Multipurpose Plus: (E) enamel, (D) dentin. Arrow shows adhesive tag in hybrid layer area.

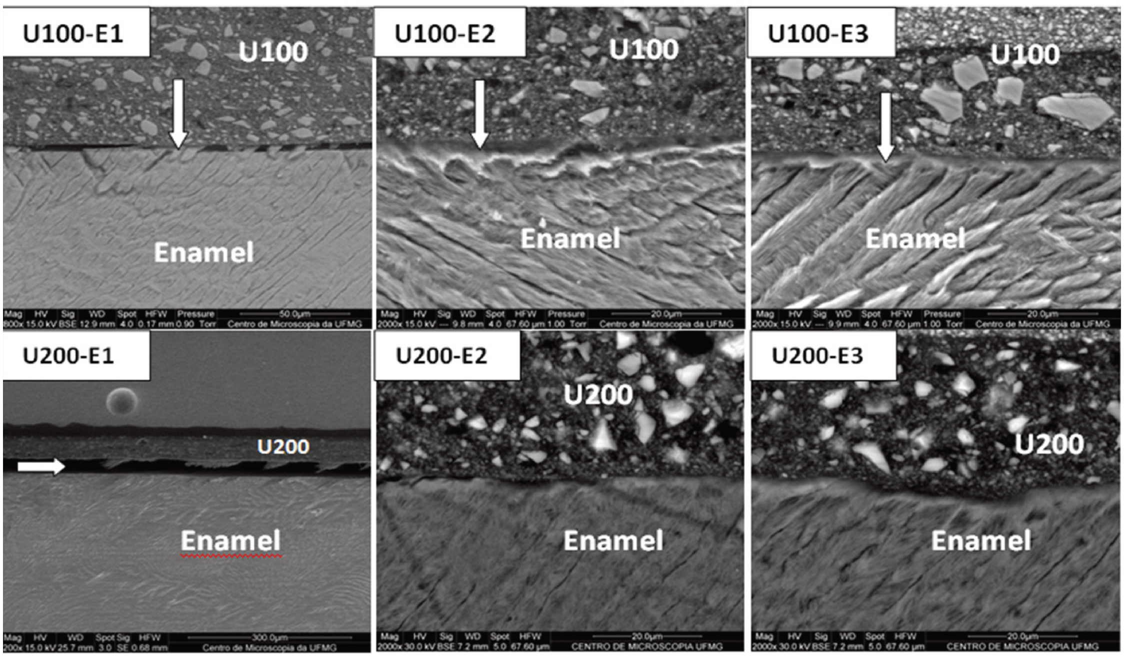

Figure 2. RelyX U100 / enamel interface: (U100-E1) no treatment; (U100-E2) phosphoric acid 37% - 15 s; (U100-E3) phosphoric acid 37% - 30 s. Vertical arrows show interaction areas. RelyX U200 / enamel interface: (U200-E1) no treatment; (U200-E2) phosphoric acid 37% - 15 s; (U200-E3) phosphoric acid 37% - 30 s. Horizontal arrow shows gap, lack of interaction.

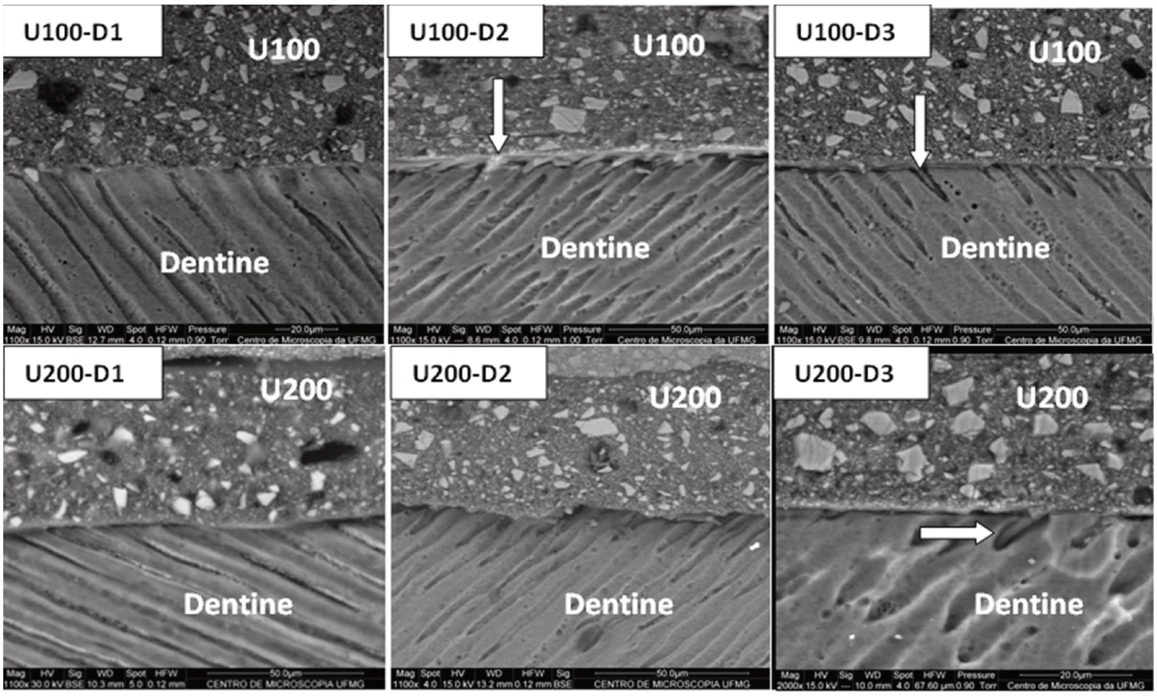

Figure 3. RelyX U100 / dentin interface: (U100-D1) no treatment, (U100-D2) phosphoric acid 37% - 15 s; (U100-D3) polyacrilic acid 11,5% - 15 s. Vertical arrows show small resin tags. RelyX U200 / dentin interface: (U200-D1) no treatment, (U200-D2) phosphoric acid 37% - 5 s; (U200-D3) polyacrilic acid 11,5% - 15 s. Horizontal arrow shows small resin tag.

U200 interacted more with dentine treated with polyacrylic acid (Figure 3: U200-D3) than phosphoric acid (Figure 3: U200-D2).

DISCUSSION

Self-adhesive cements were introduced to simplify the cementation technique without any pre-treatment of the enamel or dentine, reducing chairside, clinical steps, and operator errors. However, it has been questioned whether treatment of the tooth surface would improve the interaction between these cements and dental tissues. Several studies have reported that self-adhesive cements have limited interaction with enamel, as well as low bond strength to dentine[1,2,7,9,14,15]. In the present study, there were gaps at the interface between RelyXU100 and unetched enamel, suggesting that the interaction was not effective. In contrast, when the enamel was conditioned with phosphoric acid for 15 seconds or 30 seconds, no gaps were found. This result reinforces previous findings suggesting prior enamel conditioning to improve self-adhesive cement interaction with substrate.

A clinical trial concluded that the self-adhesive resin cement RelyX Unicem can be used for ceramic inlay luting in conjunction with selective enamel etching. In fact, they found that survival rates were even better than those without enamel etching[16]. A similar pattern was found in the present analysis of U200 cement, whereby cement interacted more with etched enamel than with unconditioned enamel. Therefore, it is likely that, in the case of unconditioned enamel, there is an irregular interaction between the cement and the substrate[12]. Besides that, cracks between the substrates in the adhesive interface, observed by Cantoro et al.[15] as well as in the present study, maybe exacerbated by sample dehydration procedures during SEM preparation, as discussed in a previous study[17]. Thus, the hypothesis suggested could be accepted once dentine or enamel hybrid layer ultramorphology was affected, depending on substrate treatment.

The resin cement RelyX Unicem has particles of glass ionomer cement, which bonds to dentine when polyacrylic acid modifies the smear layer[1]. This acid has numerous carbonyl ions that form hydrogen bonds, which promote substrate wettability. After a microtensile test, RelyX Unicem was found to confer greater bond strength after dentine treatment with polyacrylic acid, suggesting that bonding is enhanced after substrate conditioning[18]. Furthermore, the interaction between self-adhesive cement and dentine is only superficial, showing that it has a reduced capacity to completely dissolve the smear layer and interact with the underlying dentine[1,13], as observed in D1 subgroup in the present study.

The present study showed RelyX U100 tags in etched dentine samples. However, demineralization by phosphoric acid etching for 5 seconds produced a greater degradation of peritubular dentine and the presence of wider and shorter tags. However, polyacrylic acid showed a selective dissolution pattern, preserving peritubular dentine and allowing the formation of longer and narrower tags; this suggests that the cement interacts with the dentine. In contrast, Hikita et al.[14] demonstrated that the bond strength of RelyX Unicem to phosphoric acid-etched dentine was significantly lower due to inadequate infiltration of the viscous cement on the thick and compact collagen mesh, which was exposed by the phosphoric acid for 15 seconds.

In the present study, it was not possible to obtain images from whole interface between dentine etched by phosphoric acid and RelyX U200, suggesting that the dentine matrix had degraded further in this experimental subgroup, leading to disruption of the total interface. Polyacrylic-acid etched dentine generated a full interface, with the modified smear layer interacting less with U200 than withU100, and without tag formation.

The adhesive potential of RelyX Unicem to enamel and dentine may be due to different interfacial microstructures and regional dental tissues[1,2,19]. In particular, the smear layer and underlying dentine are regarded as solid structures that probably rapidly counteract the acidity of viscous solutions, thereby limiting the etching ability of acidic monomers in creating an evident hybrid layer[19]. In this regard, Al-Assaf et al.[20] reported that RelyX Unicem has the lowest bond strength values of all conventional resin cements, and that it provides no visible hybrid layer with the methodology used. Conversely, another study demonstrated that RelyX U100 confers less nanoleakage than conventional resin cement, and that it produces adhesive interfaces that are better sealed[3]. Furthermore, dentine pre-treatment with polyacrylic acid improves the microtensile bond strength of RelyX Unicem[18,21], decreasing the surface energy and enhancing adhesion to dentine[22]. Selective etching of dentine with phosphoric acid prior to luting results in the most effective bonding of all self-adhesive resin cements, suggesting that bonding can be achieved with self-adhesive resin cements without any pre-treatment steps, such as etching, priming, or bonding, which, according to the manufacturers, can compromise bonding ability[10]. Although the bonding strength of RelyX Unicem to dentine was lower than that of conventional resin cements it was more reliable less sensitive to variations in handling and aging[23].

Bond strength and restoration adaptation to the dental structure using self-etching and self-adhesive dual-curing cements are enhanced if a seating force greater than finger pressure is maintained throughout the initial self-curing period; such a force decreases the porosity of the cement[13]. According to the manufacturers, low viscosity is an advantage of RelyX U200 over U100, because it means that less pressure during cementation, and that the product can be in "automix" form. Since that cementing pressure was equal for the cements used, our results suggest that increasing the flowability did not ensure greater interaction of U200 with dentine as tags were not formed.

It was concluded that enamel etched with phosphoric acid and dentine etched with polyacrylic acid improved RelyX U100 and U200 cement interaction. Polyacrylic acid etching was more effective in the interaction between dentine and U200.

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CONFLICTS OF INTERESTS

The authors declare no conflicts of interest.

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Received: June 25, 2017
Accepted: August 17, 2017