24 HOURS AND 3-MONTHS BOND STRENGTH BETWEEN DUAL-CURED RESIN CEMENTS AND SIMPLIFIED ADHESIVE SYSTEMS

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ABSTRACT
This study evaluated the bonding compatibility between dual-cured resin cements and simplified adhesive systems (one-step self-etch and two-step etch & rinse), measured after 24 hours and 3 months. The occlusal dentin surfaces of 24 human third molars were exposed and flattened. Teeth were randomly assigned to 3 groups and treated with different combinations of adhesive system and resin cement [G1 - Single Bond/Rely X ARC (SB/RX); G2 - Excite DSC/Variolink II (EX/VR); G3 - Adper Prompt/Rely X ARC (AD/RX)]. Indirect composite restorations were cemented on flattened surfaces, and sectioned to obtain multiple bonded beams for the microtensile bond strength test. The beams from each tooth were tested under tension after 24 hours and 3 months (ANOVA/ Tukey's test, α=5%). Failure patterns were evaluated with scanning electron microscopy. After 24h, AD/RX presented the lowest bond strength mean values. AD/RX specimens did not withstand three months storage. SB/RX and EX/VR presented similar bond strengths in both periods tested. The association AD/RX resulted in low bond strength mean values, especially after storage. Cementing indirect restorations using one-step self-etch adhesive systems and dual-cured resin cements would be clinically unreliable.

Key words: dentin bonding, resin cements, system adhesive, bond strength.

AVALIAÇÃO DA RESISTÊNCIA DE UNIÃO DE CIMENTOS RESINOSOS DE DUPLA POLIMERIZAÇÃO ASSOCIADOS A SISTEMAS ADESIVOS SIMPLIFICADOS APÓS 24 HORAS E 3 MESES

RESUMO
Este estudo avaliou a compatibilidade de união entre cimentos resinosos de dupla polimerização e sistemas adesivos simplificados (adesivo autocondicionante - 1 passo- e adesivo total-each - 2 passos), após 24h e 3 meses. A superfície oclusal de 24 terceiros molares humanos foi exposta e planificada. Os dentes foram aleatoriamente divididos em 3 grupos usados de acordo a seguinte combinação entre sistema adesivo/cimento resinoso [G1 - Single Bond/Rely X ARC (SB/RX); G2 - Excite DSC/Variolink II (EX/VR); G3-Adper Prompt/Rely X ARC (AD/RX)]. Restaurações indiretas em resina composta foram cimentadas na superfície dentinária, e seccionados em múltiplos palitos para a realização do teste de resistência de união através do ensaio de microtração. Os palitos obtidos por dente foram submetidos ao teste após 24 horas e 3 meses (ANOVA/ Tukey test, α=5%). O padrão de fratura foi observado no microscópio eletrônico de varredura. Após 24 horas, AD/RX apresentou menor valor médio de resistência de união. Os corpos-de-prova do grupo AD/RX não resistiram ao armazenamento de 3 meses. SB/RX e EX/VR demonstraram similar resistência de união em ambos os períodos avaliados. A associação AD/RX resultou em menores valores médios de resistência de união, especialmente após o armazenamento. A cimentação de restaurações indiretas utilizando sistemas autocondicionantes (1 passo) e cimentos resinosos de dupla polimerização não foi considerada clinicamente confiável.

Palavras chaves: dentina, cimentos resinosos, sistemas adesivos, resistência de união.

INTRODUCTION

The effectiveness of adhesive techniques has expanded the range of clinical applications[1]. For example, the positive results obtained using adhesive cementation in indirect restorations have increased the frequency of metal-free restoration procedures, allowing sound dental tissue to be preserved with more conservative tooth preparations[2,3]. However, the clinical success of adhesive cementation is highly dependent on the bonding properties of adhesive systems and resin cements [4].

Adhesive systems can be classified according to their adhesion strategies, i.e. etch & rinse or self-etch techniques[5]. The simplified etch & rinse technique involves two steps: acid conditioning followed by application of a mixture of primer and adhesive monomers [6]. When bonding to dentin, the rinsing and drying steps make this technique more susceptible to

Table 1: Materials used in the present study, with the corresponding manufacturers, batch numbers and composition.

Product (batch number)	Composition*
Single Bond (3M ESPE, 4JY)	Etchant: 35% H3PO4
	Bis-GMA, HEMA, PAA, ethanol, water, initiators
Excite (Ivoclar Vivadent, F66811)	Etchant: 35% H3PO4
	DMA, HEMA, alcohol, phosphoric acid acrylate, SiO2, initiators, stabilizers
Adper Prompt (3M ESPE, L29064)	Liquid-A: Methacrylated phosphoric esters, Bis-GMA, initiators, stabilizers. Liquid-B Water, HEMA, stabilizers, polyalkenoic acid
Rely X ARC (3M ESPE, FL-316)	Bis-GMA, TEGDMA, Zirconia/silica filler, dimethacrylate polymer, pigments, initiators
Variolink II(Ivoclar Vivadent, F51866/F56391)	Bis-GMA, UDMA, TEGDMA, fillers, catalysts, stabilizers, pigments

*Bis-GMA, bisphenol-glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; PAA, polyalkenoic acid copolymer; DMA, dimethacrylates; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

operator error, reducing adhesive performance[7]. One-step self-etch or all-in-one systems, as they are commonly referred to, involve only one procedure[5]. These systems use acidic monomer solutions that etch, prime, and bond simultaneously[8]. They eliminate critical steps, such as rinsing and drying[9] that decrease the technique's sensitivity[8]. Consequently, these systems are being increasingly used[6], even for adhesive cementation in indirect restorations.

Adverse interactions between simplified adhesives and chemical/dual-cured resin cements have been reported[2,6,10,11]. An incompatible acid-base reaction occurs between acidic monomers present in simplified systems and the tertiary amines present in the catalyst paste of resin cements, resulting in incomplete polymerization of resin cements. The higher concentration of ionic and hydrophilic groups in adhesive systems may allow the movement of water and ions from the underlying dentin even after polymerization, compromising the hermetic sealing of the dentin interface[2]. Moreover, one-step self-etch systems usually have a pH of 1.0 or lower, resulting in considerably deep demineralization effects, and thus more hydrophilic interfaces that are more prone to hydrolytic degradation[12].

In vitro bond strength evaluations are usually performed 24h after specimen preparation[9,13]. This period of time may not be long enough to verify the efficiency of restorative and adhesive materials, both in terms of bond stability and durability[13,14]. Thus, the aim of this study was to evaluate the compatibility, after 24h and 3 months, between simplified adhesives systems (two etch & rinse systems and one one-step self-etch system) and dual-cured resin cements using a microtensile bond strength test (μTBS). Additionally, the morphological characteristics of the fractured surfaces were evaluated using scanning electron microscopy (SEM). The null hypothesis tested was that combinations of simplified adhesive systems and resin cements present similar bond strength to dentin and long-term performance.

MATERIAL AND METHODS

This study was approved by the Ethical Committee in Research at the Federal University of Bahia. Twenty-four non-carious human third molars were disinfected with 0.5% chloramine T solution. Their roots were mounted in self-curing acrylic resin cylinders (2.5 cm diameter). The occlusal enamel was removed using 180-grit silicon carbide paper under running water, and the smear layer was standardized with 600-grit silicon carbide papers under water cooling during 1 minute.

Teeth were randomly allocated to 3 groups according to the following treatments (n=8): SB/RX (Single Bond (3M ESPE, St. Paul, MN, USA)/ Rely X ARC (3M ESPE, St. Paul, MN, USA); EX/VR (Excite DSC (Ivoclar Vivadent, Schaan, Liechtenstein)/Variolink II (Ivoclar Vivadent, Schaan, Liechtenstein); AD/RX (Adper Prompt (3M ESPE, St. Paul, MN, USA)/ RelyX ARC). The composition of the materials and manufacturers are shown in Table 1. The adhesive systems and resin cements were manipulated and applied according to manufacturers' instructions. The same manufacturer was used for all groups to avoid chemical bias.

Rectangular indirect restorations (4x4x6 mm) were fabricated using composite resin (Tetric Ceram, Ivoclar Vivadent, Schaan, Liechtenstein). The composite was inserted in 2.0 mm thick increments and each increment was light-activated for 20 seconds. The light output of the light curing unit (Optilight 600, Gnatus, Ribeirão Preto, SP, Brazil) was tested (500 to 600 mW/cm^2) with a radiometer (Curing Radiometer, model 100, Kerr Corporation, Orange, USA).

The indirect composite was bonded to dentin with adhesive systems and resin cements in accordance with the manufacturer's instructions. Visible-light activation of each surface was applied for 20 s (Optilight 600). After cementation, specimens were stored in artificial saliva at 37°C for 24h. Next, they were sectioned on the mesio-distal and bucco-lingual planes using a low-speed, water-cooled diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). The boundaries of the restoration were discarded, six beams were obtained per tooth and the cross-sectioned area was measured with a digital caliper (727, Starrett Ind., Itu, SP, Brazil) to the nearest 0.01 mm. Beams obtained from each tooth were divided into 2 groups: half specimens were tested after 24h; the other half specimens were stored in artificial saliva (37°C) and tested after 3 months. Throughout the storage process, the artificial saliva was changed every five days.

To carry out microtensile testing, specimens were attached to the flat grips of a µTBS device with cyanoacrylate glue (Super Bonder; Henckel Loctite, Itapevi, SP, Brazil) and tested under tension in a Universal Testing Machine (EMIC DL 500, São José dos Pinhais, SC, Brazil) at a cross-head speed of 0.5 mm/min, until failure. Means and standard deviations were calculated and expressed in MPa. No bond strength value was attributed for premature debonding during specimen preparation. The µTBS data were analyzed using Repeated Measures ANOVA and Tukey's test (α=5%).

Table 2: Mean values (standard deviations) of µTBS (MPa).

Experimental groups	Storage	
	24 hours	*3 months*
Single Bond + RelyX ARC	27.76 (5.20) Aa	26.61 (5.73) 7 Aa
Excite + Variolink II	22.60 (9.68) Aa	22.87 (8.30) Aa
Adper Prompt + RelyX ARC	13.59 (5.58) B	-

Means followed by the same letters are not statistically different (ANOVA/Tukey's test, =0.05). Upper case letters compare adhesive systems within storage periods. Lower case letters compare storage periods within adhesive system.

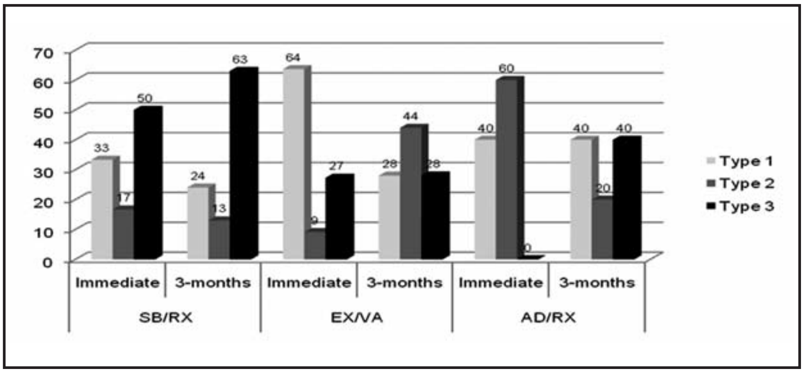

Fig. 1: Failure mode distribution according to the combination between adhesive system and resin cement.

After testing, each specimen was gold-sputter coated (Balzers-SCD 050 Sputter Coater, Germany) and observed by SEM (JSM 2900, JEOL, Scanning Electron Microscopy Peabody, MA, USA) to assess the failure pattern. Failure modes were classified in three types: type 1 - failure between adhesive system and dentin, and partial cohesive failure in adhesive resin; type 2 - total cohesive failure in the adhesive resin; type 3 - cohesive failure in the cement resin.

RESULTS

Bond strength results are shown in Table 2. All specimens from the AD/RX group aged for 3 months debonded at the cement/dentin interface during specimen storage. Therefore, this combination could not be included in the statistical analysis. SB/RX and EX/VR presented similar bond strength in both periods and were not significantly affected by storage. The failure mode distribution is described in Fig. 1. In both periods tested, SB/RX presented mainly type 3 failures (Fig. 2: SEM photomicrograph illustrating a cohesive failure in the resin cement). The most frequent type of failure in EX/VR was type 1 after 24 hours

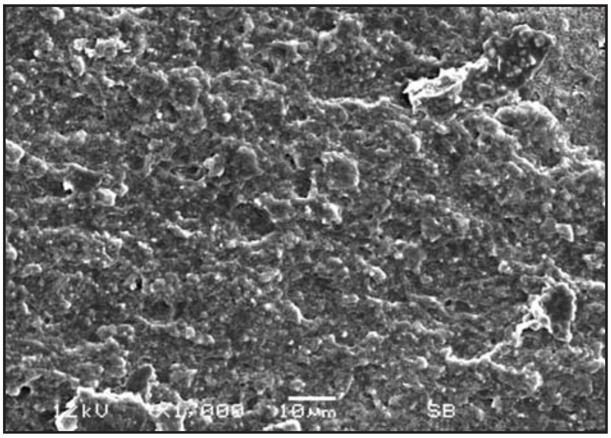

Fig. 2: SEM photomicrograph illustrating a cohesive failure in the resin cement.

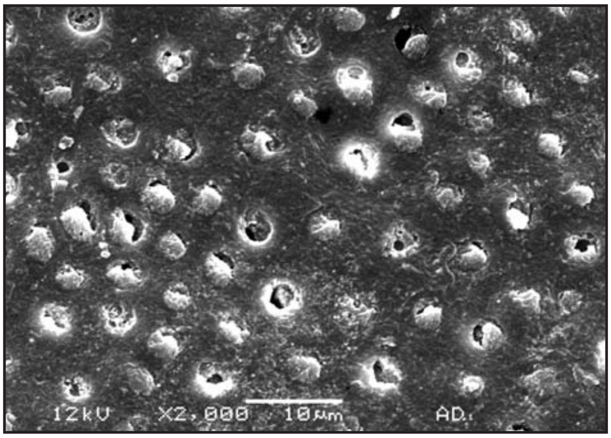

Fig. 3: SEM photomicrograph illustrating the dentin side of a fractured specimen presenting an adhesive failure between adhesive resin and dentin.

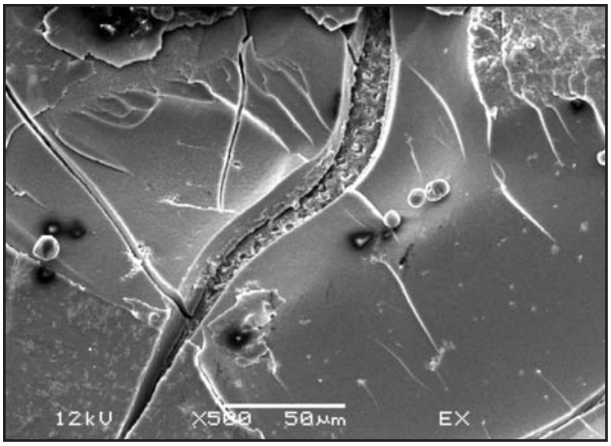

Fig. 4: SEM photomicrograph illustrating the dentin side of a fractured specimen with a total cohesive failure in the adhesive resin.

(Fig. 3: SEM photomicrograph illustrating the dentin side of a fractured specimen presenting an adhesive failure between adhesive resin and dentin); and type 2 after the 3-months period (Fig. 4: SEM photomicrograph illustrating the dentin side of a fractured specimen with a total cohesive failure in the adhesive resin). AD/RX presented mainly cohesive failures within the adhesive after 24 hours. After 3-months, both type 1 and type 3 failures were observed.

DISCUSSION

The present study was conducted to evaluate the µTBS of dual-cured resin cements bonded to simplified adhesive systems, after 24 hours and 3 months storage. Early bond strength measurements might provide an important reference regarding the bonding capability of adhesive materials[9]. Nevertheless, it is widely accepted that long-term clinical studies provide more realistic results with respect to the efficiency of restorative materials[14], but the drawbacks of such studies are the high cost and extensive length of time[15]. To overcome these limitations, *in vitro* methods, such as artificial aging, were developed to simulate aging conditions in an effort to provide information about bonding durability. In this study, specimens were stored in artificial saliva to simulate aging. However, other clinically-related factors, such as chewing stress, pH and temperature variations, were not simulated in this study.

The storage method and the solution used are important variables in this study. Storage solutions have a significant effect on the leachability of filler particles, and artificial saliva might promote a greater degradation of resin-based materials than distilled water[16]. Thus, it can better simulate the intraoral conditions. The interfacial bonding area exposed to the storage period may also play an important role in specimen degradation[14]. In the present study, specimens with 1 mm^2 interfacial bonding areas were stored in artificial saliva. This method was chosen to accelerate the degradation of the interfaces between resin cements, adhesive systems and dentin.

The etch & rinse systems (SB/RX and EX/VR) showed statistically similar bond strength values, irrespective of length of storage period. Therefore, the 3-months period may not be long enough to age restorative materials with improved bonding performance, such as the two-step etch & rinse systems. On the other hand, the bond created using the one-step self-etch system was incapable of withstanding 3 months of storage, suggesting that this adhesive system does not present a reliable long-term capacity for cementing indirect restorations. The relatively high dentin permeability found in simplified self-etch systems results from the increased concentration of acidic monomers [6,17]. The faster hydrolytic degradation of these systems in humid environments might justify the incidence of premature failures in the AD/RX group [12,18]. Similar incidences of premature failure have previously been reported[1].

Type 2 failure mode was frequently noted in the non-aged group AD/RX. It can be hypothesized that the higher permeability and incomplete light activation of the one-step self-etch system promoted a faster hydrolytic degradation, premature failure and, consequently, the loss of specimens. On the other hand, SB/RX groups demonstrated fewer failures involving the hybrid layer, since mainly cohesive failures in the cement resin were observed. In addition to hydrolytic degradation, the chemical incompatibility between chemical/dual-cured resin cements and simplified adhesive systems can negatively influence the adhesive cementation of indirect restorations[6,8,19]. The result of this adverse reaction is the incomplete polymerization of resin-based materials[2]. Since the degree of conversion of resin-based materials is directly related to their mechanical properties[11], an adequate polymerization of the resin cement is a fundamental prerequisite for restoration stability[20]. Although simplified etch & rinse systems might also present a chemical incompatibility with dual cure cements, the results of this study indicate that this effect might be less pronounced than with one-step self-etch systems (AD/RX group).

Within the limitations of this study, it could be concluded that there is no satisfactory evidence to justify the use of simplified self-etch systems and dual-cured composites for cementing indirect restorations. Findings showed that this association resulted in low bond strength mean values, especially after storage. However, bonding of indirect composite using two-step etch & rinse adhesive systems and chemical/dual-cured resin cement was not significantly affected by storage.

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