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Bond strength and chemical interaction of self-adhesive resin cements according to the dentin region

Wolney Sério Vieira-Filho, Roberta Caroline Bruschi Alonso, Alejandra Hortencia Miranda González, Paulo Henrique Perlatti D'Alpino, Vinicius Di Hipólito*

Biomaterials Research Group, School of Dentistry, Universidade Anhanguera de São Paulo (UNIAN – SP), São Paulo, SP, Brazil

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ABSTRACT

The aim of the study is to evaluate the effectiveness and level of chemical interaction of self-adhesive resin cements (SRCs) according to the dentin region. One hundred eight sound human third molars and three SRCs were selected: Bifix SE (Voco), Maxcem Elite (Kerr), and RelyX U200 (3M ESPE). Ninety human molars were used for the bond strength test and 18 teeth for the X-ray diffraction (XRD) characterization. A flat surface of superficial, deep, or axial dentin was exposed. For bond strength evaluation, 90 indirect composite resin restorations (10 mm in diameter, 2.0 mm-thick) were built and cemented with one of the SRCs according to the manufacturer's instructions. The restored teeth were then cut into sticks with cross-sectional areas of 0.8 mm^2 and tested in tensile at a speed of 0.5 mm/min (n=10). The results of bond strength were statistically analyzed by two-way ANOVA and Tukey's test (α=0.05). The fractured specimens were classified under SEM. The remaining teeth were further sectioned in order to build dentin fragments with 2.0 mm^2 of area and 0.2 mm in thickness for XRD analysis. In general, significantly higher bond strength was found when bonding to axial and deep dentin compared to superficial dentin. Comparing the bonding effectiveness of the SRCs, taking into account the mean bond strength obtained in the 3 dentin regions, the study found no significant difference ($p > 0.05$). Although RelyX U200 showed similar bond strength irrespective of the dentin region ($p > 0.05$), the bonding results of the other 2 SRCs varied significantly ($p < 0.05$). There was a higher incidence of cohesive failure in the SRCs for all groups. The XRD analysis detected different perceptual reductions of hydroxyapatite crystallinity for all SRCs, indicating a particular chemical interaction in each experimental condition. Thus, it can be concluded that the bond strength and chemical interaction of the SRCs can vary significantly according to the dentin region.

1. Introduction

Over the last 2 decades, adhesive dentistry has undergone remarkable progress, thanks to continuous and rapidly evolving tooth-bonding technology. This evolution process has led to the development of an innovative category of resinous restorative materials, self-adhesive materials, which are able to directly interact with the dental hard tissues for bonding [1–5].

By using such particular adhesion approach, the self-adhesive resin cements (SRCs) has been outstanding. They are able to greatly simplify the cementation procedure by eliminating the need to pretreat the tooth structure, due to the incorporation of functional monomers that demineralize and infiltrate the tooth substrate, resulting in micromechanical retention [1,6]. Such functional monomers are generally esters originating from the reaction of a bivalent alcohol with methacrylic acid and phosphoric/carboxylic acid derivatives. It has been suggested that such functional monomers also are able to provide chemical adhesion to a tooth via secondary reactions with the calcium present in the hydroxyapatite (HAp), contributing to the SRCs' performance in addition to micromechanical hybridization [1,6,7].

Since SRCs interact directly with the tooth for bonding, it is speculated that the inherent characteristics of the dentin, mainly considering their chemical and morphological regional variation, could influence adhesion effectiveness. The dentinal tubule's variability in terms of number, diameter, and changes in its orientation from the surface to the pulp chamber create a severe discrepancy in dentin within the same tooth [8–10]. As a consequence of these regional morphologic dentin particularities, the level of mineralization, and thus the calcium available for chemical interaction with the SRCs, is also variable [8].

* Correspondence to: Universidade Anhanguera de São Paulo, UNIAN – SP Programa de Mestrado em Biomateriais em Odontologia, Avenida Raimundo Pereira de Magalhães, 3305 - Vila Pirituba, São Paulo, SP CEP 05145-200, Brazil.
E-mail address: vdhipolito@yahoo.com.br (V. Di Hipólito).

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Therefore, the purpose of the present study is to investigate the bond strength and level of chemical interaction of SRCs in 3 different dentin regions: superficial, deep, or axial. The research hypotheses of the study are that the (I) bond strength and (II) chemical interaction of the SRCs will be negatively influenced by the dentin region.

2. Materials and methods

This study used 108 caries-free human third molars. Teeth were obtained and used in accordance with the local IRB (# 218/11) and with the informed consent of the donors. Teeth were stored in 0.5% chloramine-T solution at 4 °C and used within 1 month following extraction.

2.1. Microtensile bond strength evaluation and SEM fractographic analysis

Ninety human molars were selected. The teeth were x-rayed using a millimeter adhesive scale fixed on periapical X-ray film (X-Ray Mesh; Hager & Werken GmbH & Co. KG – Germany) in order to estimate the dimensions of the dental structures and guide the dentin's regional exposition. After that, flat dentin surfaces were produced on each tooth using a diamond-impregnated disc (Extec, Enfield, CT, USA) under water cooling in a specific cutter machine (Isomet 1000, Buehler, Lake Bluff, IL, USA). The sectioned teeth were then randomly assigned into 3 groups according to the exposed dentin region: superficial (1 mm below the dentine-enamel junction at the occlusal surface), deep (1 mm above the highest pulp horn), or axial (1 mm below the dentine-enamel junction at the buccal or lingual surface).

Composite overlays were constructed with a resin composite (3 M ESPE Filtek Z250, St. Paul, MN, USA) using Teflon molds (12 mm in diameter, 2 mm in thickness). After the composite overlay fabrication, both sides of the restoration were sandblasted with 50 μm aluminum oxide glass spheres (Sandblaster Micro Etcher, Buffalo Dental, San Ramon, CA) for 10 s on each side. The composite overlays were then ultrasonically cleaned in distilled water for 3 min. After that, silane primer (3 M ESPE RelyX Ceramic Primer, St. Paul, MN, USA) was applied to the sandblasted surfaces with a minisponge for 1 min and air dried.

The sectioned teeth of each group were assigned into 3 sub-groups according to the 3 SRCs used (n=10). The composition of the cements are listed in Table 1. Prior to cementation, the exposed dentin surfaces were abraded with #600-grit SiC paper for 15 s in order to standardize the smear layer [11]. The SLCs were manipulated according to the manufacturers' instructions and applied to the composite overlays, which were gently seated on the prepared dentin surfaces using finger pressure. Thereafter, the restored teeth were placed under a constant seating pressure of 3.0 kg for 3 min [12]. Excess cement was removed after setting and then light cured for 60 s on 4 different regions at the tooth/restoration margin and on the top of overlay using an LED light unit (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein) with a radiant emittance of 1000 mW/cm^2.

Table 1
Description of the materials used in this study.

Resin cements	Manufacturer	Lot #	Composition
RelyX U200	3 M ESPE, St. Paul, Minnesota, USA	5355	Silane treated glass powder, substituted dimethacrylate 1-benzyl-5-phenyl-barbic-acid, calcium salt, silane treated silica, sodium p-toluenesulfinate, 1,12-dodecane dimethycrylate calcium hydroxide methacrylated aliphatic amine methacrylated aliphatic amine titanium dioxide
Maxcem Elite	Kerr Italia, Scafati, Italy	4791075	GPDM, co-monomers (methacrylate ester monomers), inert mineral fillers, Ytterbium Fluoride, activators, stabilizers and colorants
Bifix SE	Voco, GmbH, luxhaven, Germany	1420440	Aliphatic (UDMA), aromatic (BisGMA), and acid methacrylate, benzoyl peroxide (Initiator), amines (cat) and BHT (stabilizer).

GPDM, glycerol dimethacrylate dihydrogen phosphate; UDMA, urethane dimethacrylate; Bis-GMA, bisphenol A diglycidyl methacrylate; BHT, butylhydroxytoluene.

The restored teeth were then stored in distilled water at 37 °C for 24 h. After storage, the specimens were sectioned to obtain bonded, stick-shaped specimens with a cross-sectional area of 0.8 mm^2 (± 0.2) using the "nontrimming" specimens and tested in tensile at a speed of 0.5 mm/min [13]. Statistical differences among the mean bond strengths of the experimental groups were investigated by two-way ANOVA (factors: *resin cements* and *dentin region*) and Tukey's test at a preset alpha of 0.05.

The fractured specimens were mounted on stubs with double-face carbon tape and desiccated in silica gel for 2 h [13]. The specimens were then sputtered (SCD 050; Balzers, Schaan, Liechtenstein) with a thin palladium-gold film (25 nm) for 100 s at 40 mA and examined by a scanning electron microscope (SEM; JEOL-5600 LV, JEOL Ltd., Tokyo, Japan) operating at 15 kV. The failure modes were classified according to the following categories [13]: Type I – cohesive failure in the resin cement; Type II – adhesive failure; and Type III – mixed failure: adhesive and in the resin cement. The schematic representation of the technique used for the microtensile bond-strength test is illustrated in Fig. 1.

2.2. X-ray diffraction (XRD) analysis

Eighteen human teeth were selected and sectioned as previously described for the microtensile test, exposing superficial, deep, and axial dentin. Then, a square area (2.0×2.0 mm) of each sample was obtained using high-speed and cylindrical medium-grit diamond bur. The surface area of the specimens was previously standardized by using abrasive discs (Sof-lex, 3 M, ESPE, USA) mounted in a hand piece at low speed.

The dentin slices were then manually abraded by using a sequence of SiC sandpaper with decreasing grits (#A80, #150, and #600) until reaching a thickness of 0.2 mm. After that, the dentin samples were placed in an ultrasound apparatus in 90% ethanol solution for 5 min in order to eliminate waste sanding and other possible contaminants that could interfere in the XRD analysis.

The specimens were then evaluated in an X-ray diffractometer (DMAX Ultima+ Rigaku International Corporation, Tokyo, Japan) using CuKα radiation operating at 40 kV and 20 mA. Scans were performed from 10° to 80° (2θ) at a step size of 0.02° and a scan speed of 2°/min. Qualitative phase analysis was performed by using the Joint Committee on Powder Diffraction – International Center for Diffraction Data (JCPDS– ICDD) databases. The dentin samples were initially tested in order to guarantee the absence of cement to establish the individual chemical composition of each specimen. After this initial characterization, the specimens received a 1 mm thick cement layer opposite the side initially evaluated. After 3 min of chemical reaction, the SRCs were photoactivated for 20 s (LED light unit - Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein; 1000 mw/cm^2). The specimens were then tested again on the same side of the first characterization. Thus, it was possible to verify the chemical interaction of each SRC with the different regions of dentin by comparing the hydroxyapatite peak intensity at (211) between the first and second characterization of the same sample. The results were expressed in the reduction of peak intensity percentage.