International Journal of Adhesion & Adhesives 38 (2012) 69–74

Contents lists available at SciVerse ScienceDirect

International Journal of Adhesion & Adhesives

journal homepage: www.elsevier.com/locate/ijadhadh

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

T.R. Aguiar [a], C.B. Andre [a], C.A.G. Arrais [b], A.K. Bedran-Russo [c], M. Giannini [a,*]

[a] Department of Restorative Dentistry, Piracicaba School of Dentistry, Campinas State University, Piracicaba, SP, Brazil
[b] Department of Operative Dentistry, Guarulhos University, Guarulhos, SP, Brazil
[c] Department of Restorative Dentistry, College of Dentistry, University of Illinois at Chicago, Chicago, IL, USA

ARTICLE INFO

Article history:
Accepted 25 May 2012
Available online 6 June 2012

Keywords:
Resin cements
Confocal laser scanning microscopy
SEM
Hybrid layer
Dentin

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

* Corresponding author. Tel.: +55 19 21065338; fax: +55 19 21065218.
E-mail address: giannini@fop.unicamp.br (M. Giannini).

0143-7496/$ - see front matter © 2012 Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.ijadhadh.2012.05.009

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.