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Effects of curing mode and moisture on nanoindentation mechanical properties and bonding of a self-adhesive resin cement to pulp chamber floor

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ABSTRACT

Objective. This study aimed to investigate the effect of dentin surface moisture and curing mode on microtensile bond strength (MTBS) and nanoindentation characteristics of a self-adhesive resin cement.

Methods. Forty-four extracted human molars were distributed into four groups according to dentin surface moisture (dry or wet) and curing mode of the resin cement (light or chemical). Clearfil SA Cement (Kuraray Noritake Dental, Japan) was used for cementation of composite cores to the pulp chamber dentin. The specimens were sectioned into beams for MTBS test at the pulpal floor. Nanoindentation hardness and creep of the cement layer were measured under 100 mN load with 30 s hold segment. Data were statistically analyzed using two-way ANOVA and Weibull distribution of MTBS ($\alpha = 0.05$).

Results. Moisture, curing mode or their interaction did not significantly affect mean MTBS values that ranged 17.6–22.6 MPa ($p > 0.05$); however, the lowest characteristic strength was found in moist chemically cured group. Hardness ranged 437–512 MPa, and was not affected by the experimental factors ($p > 0.05$). Nanoindentation creep ranged 9.3–10.9% with the chemically cured groups showing the highest values, indicating lower cross-linking and deformation resistance of their polymer network.

Significance. Additional moisture on dentin surface did not contribute to adhesion of the anhydrous self-adhesive resin cement to dentin. Light-curing, despite attenuation through the composite core, was beneficial and improved nanoindentation creep resistance of the cement. The difference was not, however, reflected in the mean bond strength or hardness values.

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1. Introduction

The success of an endodontically treated tooth relies not only on the apical sealing but is also highly dependent on coronal sealing. The adhesives used for bonding act as durable barriers hampering coronal microleakage, thus achieving an effective coronal seal, which is a fundamental step in prevention of bacterial invasion, secondary caries and decementation. It is also suggested that bonding techniques might have significant potential in enhancing the fracture strength of endodontically treated teeth and preventing vertical root fracture [1–3].

Indirect adhesive procedures represent a substantial portion of esthetic restorative procedures, especially for restorations where a large amount of the natural tooth substance is lacking. However, bonding to intracanal and pulp chamber dentin and coronal tissues should still be optimized [4]. Variation in the structure of the pulp chamber makes it a challenger bonding substrate; since it is not prepared during the endodontic procedures, this area does not have a dominant smear layer. Therefore, the effectiveness of the adhesive systems depends mostly on collagen-rich predentin, enlarged tubules, and the small amount of intertubular dentin [5]. The bond strength values reported for pulp chamber dentinal surfaces were usually lower than the bond strength values of flat coronal–occlusal dentin [6]. Until recently, all luting agents required some pretreatment of the dentin; either acid-etching or application of a self-etching primer to prepare the tooth prior to cementation [7], which resulted in complex and technique sensitive application procedure. A new type of luting material has been developed that does not require any pretreatment of the tooth surface, the so-called self-adhesive resin cement (SARC) [8]. Some of these materials rapidly gain popularity among clinicians due to their simplified application technique and favorable early results [9]. In order to become widely accepted, the newly introduced SARCs are expected to offer good esthetics, optimal mechanical properties, dimensional stability, and good adhesion [8].

Dentin is composed of about 50 vol% mineral in the form of a carbonate rich apatite; 30 vol% organic matter which is largely type I collagen; and about 20 vol% water [10]. Based on the intrinsically moist structure of dentin, bonding has been more complicated compared to enamel, especially with adhesive systems that require moisture control for optimal adhesion [4,8]. On the other hand, while SARCs contain no water in their composition, water is crucial for their mechanism of action. The mechanism involves ionization of the acidic functional monomer to demineralize, penetrate, and establish a chemical bond with calcium ions form dentin apatite, simultaneously allowing for two-fold (i.e., micro-mechanical and chemical) bonding mechanism [11]. SARCs have a limited ability to infiltrate dentin substrate due to their high viscosity compared to adhesive materials for direct bonding resulting in a relatively thin hybrid layer [12]. Therefore, the role of chemical interactions such as those between the acidic functional monomers and the substrate [13], should be highlighted as adhesion mechanism for an SARC. Apart from the formation of complex compounds with calcium ions, different kinds of physical interactions, such as hydrogen bonding or dipole-to-dipole interactions were assumed to favorably influence self-adhesion [14].

The formulations of SARC are expected be moisture tolerant, as claimed by the manufacturers [15]. SARCs rely on the intrinsic dentinal water and perhaps, water remaining on dentin surface after washing to establish adhesion. However, water content also affects the polymerization efficacy of resin blends depending on their chemical formulations and initiator mechanisms [16]. Residual water may compromise integrity of the interface, where excessive moisture on the bonding substrate has been regarded as unfavorable [17]. A previous report indicated that adhesion of an SARC was compromised to air-dried dentin surface and extra moisture resulted in better adhesion [18]; however, few other studies have investigated the effect of dentin moisture on the bond strength of other SARCs.

The SARCs are categorized as dual-cured resin cements, in which both light-activating and chemical-activating mechanisms are provided. Light activation of the resin cements may increase the degree of conversion when compared to chemical-activation alone and enhance physical properties [19]. On the other hand, chemical-activation is expected to provide a uniform polymerization at the bottom of deep cavities where access for curing light is limited. Polymerization contraction of composite resin luting materials is known to produce high stresses in the interface, specially under constrained setup such as a in a cavity; with chemical activation alone, resin cements produced lower shrinkage and stress rates than the light-cured resins [20]. The effectiveness of chemical activation without photo-irradiation of the resin cements has been variable among different resin cements, with some products exhibiting inadequate polymerization and poor adhesion when no light was applied [21–23]. The acidic nature of an SARC has been mentioned as one factor hampering its effective polymerization specially under chemical mode [24].

Several techniques have been introduced to evaluate the mechanical performance of resins. More recently, nanoindentation technique has enabled investigation of local mechanical properties of materials under various loading regimes and allows the investigation of selected material properties on small amounts of materials, based on the load–displacement data of indentations on a submicron scale. Measurement of mechanical properties by nanoindentation has been suggested as advantageous over the conventional methods for its high resolution of force and accurate indent positioning [25]. Hardness is known is as an indirect indication of its conversion degree [26]. Resin-based dental adhesives exhibit time-dependent creep, which is likely to be related to the polymer network integrity and affect their performance [27]. The time-dependent viscoelastic response was shown to be an important feature of both natural and synthetic biomaterials [27,28]. Some previous studies have reported on the effect of curing strategy on mechanical properties of resin cements, but few studies have looked into indentation creep.

Thus, the aim of this laboratory study was to investigate the MTBS, nanoindentation mechanical properties of an SARC used with two curing modes of bonding to pulp chamber dentin under different surface moisture conditions. The null hypotheses proposed were that [1] the moisture of dentin