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Universal cements: dual activated and chemically activated

Eliane de Lima, Ricardo Santos, Márcia Durão, Armiliana Nascimento and Rodivan Braz

University of Pernambuco, Camaragibe, Recife, Brazil

ABSTRACT

Objective: The aim of the present study was to assess the bond strength of universal cements cured either dually or chemically only.

Methods: Three cements were assessed using different types of application: dual activated (DA) or chemically activated (CA). In total 80 dentin blocks were used, obtained through the enamel wear of the lingual and buccal surfaces of bovine incisors. Standard cone-shaped cavity preparations were created using diamond burs. Subsequently, indirect restoration blocks were designed with Filtek Z350 (3M ESPE) composite resin. The teeth were divided into two groups (DA and CA) and then subdivided into four subgroups ($n = 10$) prior to cementation with the respective products: Duo-Link (Bisco); RelyX Ultimate (3M ESPE); Nexus 3 (Kerr) and conventional RelyX ARC (3M ESPE) as the control. The cementation in the PA group was applied following the manufacturer's instructions. The CA group was cemented in a darkroom to avoid exposure to light. They were stored in distilled water at 37 °C for 24 h and submitted to the push-out test. Data were analyzed by two-way ANOVA and Tukey's *post-hoc* test ($p < .05$).

Results: The greatest bond strength results were obtained for photoactivated universal cements.

Conclusion: Chemical activation is not sufficient to ensure acceptable bond strength.

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Introduction

The clinical success of indirect restoration procedures depends among others on the cementation technique used to create a stable bond between the restoration and the different dental structures. Resin cements have been widely used in the fixation of inlays, onlays, crowns and pins due to their improved mechanical properties, ease of use and esthetic qualities.[1] However, many new products have appeared in the dental market, making the choice of materials a difficult task for professionals.

Resin cements are composed of a matrix of Bis-GMA or UEDMA, monomers with a low molecular weight, such as TEGDMA, and functional hydrophilic groups that promote adhesion to dentin, such as HEMA and 4-META.[2] Resin-based cements may contain the monomer MDP (10-methacryloyloxydecyl dihydrogen phosphate), which also promotes adhesion.

Adhesive cementation is a critical phase that involves the application of an adhesive system and a cementation agent.[3] Consequently, this slow, sensitive and complex procedure requires multiple steps.[4]

Universal cements, which involve a process of dual polymerization, have recently been launched in the market. However, the use of a multimode adhesive system is recommended. These adhesives can be applied to enamel and/or dentin, offering the possibility of using them as a conventional (complete conditioning technique) or self-etching system. These systems differ in relation to the availability of the monomer MDP, which interacts chemically with the hydroxyapatite of dental tissue.[5,6]

Resin cements are also classified based on the form of activation, which can be one of the following: photoactivation; chemical activation or dual activation. Chemical activation is still used in places with poor lighting, whereas photoactivation (dual) is more commonly used due to the following advantages: storage stability; less air bubbles during handling and better physical properties.[7]

Monomer infiltration of demineralized substrates and a high degree of conversion are crucial factors when seeking to establish a long-lasting bond.[5] Under clinical conditions, it seems probable that light cannot reach the entire coronal cavity with similar irradiance values during photoactivation. Previous studies have shown that a large distance between the point of the photopolymerizer and the surface of the material tends to cause a loss of irradiance, which can

CONTACT Eliane Alves de Lima eliane_alveslima@hotmail.com Ataulfo Alves-130. Camaragibe–PE CEP: 54753–080, Brazil

have a negative effect on the adhesive bond to dentin.[8]

For dual resin cements, the degree of final conversion also depends on the quantity of light that reach the material. Theoretically, these cements combine the beneficial qualities of auto-cured and light-cured cements, given that the portions submitted to an insufficient quantity of light exhibit delayed polymerization. Although chemical and light-induced polymerization are present in these cements, they are complementary and independent.[9]

Concerning indirect restorations, a portion of the light emitted by the photopolymerization unit is absorbed by the restoration material and does not reach the cement.[10] In these cases, the areas of cement that have received an insufficient quantity of light will depend on chemical activation to ensure complete polymerization. However, previous studies [11,12,13] have indicated that chemical activation in isolation is ineffective in terms of dual resin cements attaining maximal monomer conversion. According to McComb,[14] the complete polymerization of resin cements is essential for satisfactory bond strength and the longevity of restorations. Thus, these materials in their dual cure form should be able to attain an elevated degree of conversion with or without light.

Therefore, the aim of the present study was to assess the bond strength of universal cements cured either dually or chemically only. The null hypothesis suggested that there would be no difference in the bond strength between dual activated (DA) and chemically activated (CA) cements.

Materials and methods

Three resin cements were studied: Duo-Link (Bisco); Ultimate RelyX (3M/ESPE) and Nexus 3 (Kerr). RelyX ARC (3M ESPE) was used as control (Table 1).

Preparation of the specimens

A total of 80 bovine incisors, free from cracks and structural defects, were selected. The teeth were disinfected in a 0.1% aqueous solution of thymol at 4 °C for no longer than 1 week. The roots were removed with a water-cooled diamond saw (KG Sorensen, Barueri, SP, Brazil). The buccal and lingual surfaces of the crown were wet-ground with 400- and 600-grit SiC abrasive papers in a polishing machine (Resistec São Paulo, Brazil) in order to obtain flat dentin surfaces. Standardized conical cavities (2 mm top diameter; 1.5 mm bottom diameter; 2 mm height) were prepared with conical diamond burs at high-speed, under air–water cooling. A custom-made preparation device allowed the cavity dimensions to be standardized. The burs were replaced after every five preparations. In this manner, a cavity with a C-factor magnitude of 2.2 was obtained.[15] Samples were prepared according to a previously described method for the push-out bond strength test.[16]

Design of the indirect restorations

Indirect restorations were obtained using the direct/indirect technique. The preparations were isolated with KY gel (Johnson&Johnson). Subsequently, the resin compound Filtek Z350 XT A2 (3M/ESPE) was inserted in a single increment, adapted and photoactivated by LED light (Optilight Plus 600mW/cm^2, Gnatus, SP, Brazil) for 40 s on each face. The restorations were covered with polyester strips so that the light could be positioned directly above them. The restorations were concluded with Sof-Lex (3M/ESPE) finishing discs on the vestibular and lingual faces, using a low rotation to remove irregularities of the resin compound. The restorations were removed and cleaned in an ultrasonic cube (Ultracleaner 1400, São Paulo, Brazil) for 10 min.

Cementation of the restorations

The prepared teeth were randomly divided into two experimental groups depending on the type of activation: DA group and CA group:

Table 1. Cements used, manufacturers and chemical composition.

Products	Manufacturer	Product composition
RelyX Ultimate Clicker	3M ESPE, St. Paul, MN	Base: methacrylate monomers; Radiopaque; salinized fillings; initiator and stabilizer components; rheological additives. Catalyst: methacrylate monomers; alkaline radiopaque; initiator and stabilizer components; pigments; fluorescent dyes; rheological additives, cure in the dark; activator of the Scotchbond Universal adhesive.
Duo-Link	Bisco	Base: Bis-GMA; Triethylene glycol dimethacrylate; urethane dimethacrylate; fill the glass; catalyst; Bis-GMA. Triethylene glycol dimethacrylate; fiberglass.
Nexus 3	Kerr, Australia Pty. Ltd.	The manufacturer did not specify the composition.
RelyX ARC	3M/ESPE St. Paul, MN	Paste A: TEGDMA, Bis-GMA, zirconia silica, pigments, amine and a photoinitiator system. Paste B: Bis-GMA, TEGDMA, zirconia silica, benzoyl peroxide.

- *DA group* – The cement agent was applied in the cavities as recommended by the manufacturer (Table 2). The indirect restoration block was cemented under pressure. The samples were stored in distilled water at a temperature of 37 °C for 24 h and then submitted to the push-out test.
- *CA group* – The cements were manipulated and applied in the cavities in a dark room used to process X-ray film (Odontológica Indústria e Comércio Ltda., São Paulo, Brazil), protecting the materials from exposure to ambient light. Cementation was conducted as previously described.

Mechanical trial of bond strength through 'push-out' extrusion

The samples were positioned in the center of a steel support with a central opening of 3 mm, coupled to a universal testing machine (KRATOS, São Paulo, Brazil). The coronary face with the largest diameter was positioned on the lower device push-out. A stainless steel rod of 1 mm in diameter was adapted to the load cell, exerting a compression force in the central area of the restoration. The machine operated at a rate of 0.5 mm/min until complete extrusion of the restoration occurred. The results were obtained in kilogram-force and converted into megapascal. Later, they were submitted to statistical analysis.

Results

Table 3 displays the different types of activation and compares the DA groups with the CA groups. A statistically significant difference was found between all of the cements. The greatest bond strength values were associated with dual activation. No statistically significant differences were found between the three universal cements in the DA group or in relation to the control group. However, when chemically activated, Universal RelyX Ultimate cement exhibited a significantly lower bonding strength than the other materials studied (Table 3).

Discussion

In the present study, adhesion to dentin was the main theme of discussion since the morphological complexity of dentin substrate means the optimization of bonds to dentin still represents one of the greatest challenges in adhesive dentistry today.[17]

In the experimental conditions of the present study, the null hypothesis suggested that there would be no difference in the bond strength of DA or CA cements. This null hypothesis was rejected. All of the resin cements assessed exhibited significantly higher results when dual activated.

Previous studies of cements that exist in the market have reported the best bond strength results in association with dual activation. These results show the inefficiency of chemical activation, compared to dual activation, when seeking to attain the maximal conversion of monomers and consequently improve the bond strength of these cementing agents.[18] These findings were corroborated by the results in the present study, indicating that chemical activation without light activation of universal resin cements is not sufficient to attain an acceptable bond strength.

When the universal resin cements in the present study were chemically cured only, the bond strength of RelyX Ultimate was significantly lower than those

Table 3. Means (standard deviations) of tensile bond strength (MPa) according to the cementing agent and the activation technique.

Cementing agents	Photoactivated (DA)	Chemically activated (CA)	
Duo Link Universal	11.67 ± 2.82	8.49 ± 2.18^a	$p^1 = .011^*$
RelyX Ultimate	10.15 ± 3.20	1.43 ± 0.34^b	$p^{(1)} < .001^*$
Nexus 3	10.50 ± 2.13	6.86 ± 1.27^a	$p^{(1)} < .001^*$
Rely ARC	10.56 ± 4.29	7.14 ± 2.64^a	$p^{(1)} = .046^*$

$p = .517$ $p = .001^*$.
[a,b]Different letters in the same column indicate statistically significant differences between the cements and by group – DA or CA – ($p < .05$).
*Indicates a significant difference between the activation techniques per cement.

Table 2. Application according to the manufacturer's instructions in the photoactivated groups.

Cement	Form of application
RelyX Ultimate Clicker Universal	After the application of the self-etching adhesive system Adper Scotchbond Universal (3M/ESPE), mix the paste base and the catalyst base until obtaining a homogenous paste in 20 s using a spatula. Apply to all cavity walls, position and stabilize the indirect restoration and photoactivate each face of the restoration for 20 s.
Duo-Link Universal	Apply the self-etching adhesive system All Bond. Mix the two pastes, applying directly in the preparation, position the indirect restoration by holding it firmly in place for between 20 and 30 s, remove the excess and photoactivate for 40 s.
Nexus 3 Universal	After the application of the self-etching adhesive system OptiBond All In One, apply the cement and remove the excess and photoactivate for 40 s.
RelyX ARC Conventional	After acid conditioning and the use of the adhesive system (Adper Single Bond), dispense equal proportions of cement on a disposable mixing block, mix with a spatula for 10 s, apply to the cavity and wait 5 min. Then, photoactivate for 40 s.

of the other cements tested. According to Gauthier et al.,[19] the reduction in bond strength is caused by the chemical interaction between tertiary amine, which is present in the composite and accelerates the reaction of chemical activation, and unreacted acid monomers from the surface layer of the adhesive inhibited by oxygen. This interaction would impede the participation of amines in the redox reaction, resulting in failures in the polymerization process of the layer of resin cement in contact with the adhesive, possibly leading to the failure of the entire adhesion complex.[20,21]

The reasons for this result may not be restricted to the negative interaction between simplified adhesives and systems that initiate the chemical polymerization of resin cement. A number of aspects related to adhesion to dentin should be considered, including the great hydrophilicity of self-etching systems, enabling them to behave like semipermeable membranes; the high concentration of solvents among these products, hindering the creation of a solvent-free environment; the susceptibility of single-step adhesive systems to separate phases and form air bubbles, leading to abnormalities in the monomer/water ratio.[22]

According to Gauthier et al., [19] universal self-etching adhesive systems can result in aqueous dentin permeability and osmotic bubbles that may affect the durability of the bond. Multimode universal adhesives contain similar levels of water to single-step self-etching adhesives in their solutions, which can degrade the bond interface. However, these observations require further study, validation and scientific evidence.

The DA universal cementing agents exhibited satisfactory results when compared with results in the literature.[18] In most of these systems, methacrylate monomers are partially substituted by phosphate monomers, such as MDP, which can promote chemical adhesion between the adhesive and the substrate. This appears to be a determining factor in the stability and durability of bonds.

Based on the results of the present study, it is possible to deduce that the increase in bond strength associated with universal cements could have been caused by the chemical interaction between MDP and hydroxyapatite.[23,5] The RelyX Ultimate system associated with the Single Bond Universal adhesive system exhibited MDP in its composition and contained copolymers of glass ionomer. The latter cause a reaction between free carboxylic groups and calcium through polyacids. The deposition of MDP salts and calcium could explain the elevated bond strength promoted by these universal systems.

In the present study, the single bond used together with RelyX ARC as a control exhibited similar results to the new universal cement agents associated with the self-etching, universal and multimode adhesive systems used in the self-etching technique.

Concerning adhesive techniques, conventional adhesive systems require acid conditioning using phosphoric acid at concentrations of 30% to 40%, which exposes an extensive network of collagen fibers that is not completely involved in the adhesive system.[24] These exposed collagen fibers are susceptible to degradation due to the hydrolysis caused by the infiltration of oral liquids in the adhesive interface or even by the outbreak of liquids from dental pulp.[25] In addition, inactive collagenolytic enzymes intrinsic to dentin, the matrix metalloproteinases (MMPs), are activated after acid conditioning and cause greater collagenolytic activity.[26] MMP's are activated by the application of adhesive systems, regardless of the strategy of application. The acid pH of the adhesive systems is also involved in this activation. However, self-etching systems are less intense than conventional systems.[27]

Based on the discussion and the results obtained, it seems reasonable to state that self-etching adhesive systems are the most favorable selection when dealing with dentin structure. This type of system offers satisfactory results with no significant differences to the controls, as well as the benefit of less MMP activation.

Conclusion

It is possible to conclude that:

*DA universal resin cements exhibit a satisfactory adhesive performance.

*The cementing agents showed much lower bond strengths when activated chemically only than when they were dual activated.

Disclosure statement

The authors report no conflicts of interest. The authors alone are responsible for the content and writing of this article.

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