

Effect of Polyacrylic Acid Conditioning on the Longevity of Bonding of Self - Adhesive Resin Cement to Dentin

Efeito do Condicionamento com Ácido Poliacrílico na Longevidade da Adesão de Cimento Resinoso Autoadesivo à Dentina

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Abstract

Bonding of fiber posts using self-adhesive resin cement to radicular dentin is controversial. The aim was to evaluate the effect of conditioning of polyacrylic acid on the longevity of bonding of self-adhesive resin cement to dentin. Thirty six single rooted pre molars were divided into 3 groups (n=12) ARC: ScotchBond Multi Purpose + RelyXTMARC; U: RelyXTMU200; PU: 11.5% Polyacrylic acid + RelyXTMU200. After conditioning of the dentin, the resin cement and the post were inserted and light-activated for 60s (1200mw/cm²) and stored in distilled water (24h/37°C). Two discs of 1 mm thickness were obtained from each root-third and tested in push-out (0.5mm/min) after 24 hours and 6 months. For statistical purposes, the data from the root-thirds were grouped and the bond strength (in MPa) was analyzed using Two-Way Analysis of Variance and Tukey's post-hoc test at 5%. The power of the test was calculated. The bonding of the U at 6 months was higher than PU at both times (p<0.001). There were no differences for time (p=0.153) or for the interaction of treatment x time (p=0.090). Adhesive fractures between the cement and dentin were prevalent. The power test was 0.97. The bonding of the RelyXTMU200 was influenced by the dentin pre-treatment with polyacrylic acid and the bonding of all groups remained stable for 6 months.

Keywords: Dental Cements. Dentin. Shear Strength.

Resumo

A adesão de pino de fibra de vidro com cimento resinoso autoadesivo à dentina é controversa. Foi objetivo avaliar o efeito do condicionamento da dentina com ácido poliacrílico na longevidade da adesão de cimento resinoso autoadesivo. Trinta e seis raízes de pré-molares uni radiculados foram divididas em 3 grupos (n=12) ARC: ScotchBond Multi Purpose + RelyXTMARC; U: RelyXTMU200; PU: Ácido poliacrílico 11,5% + RelyXTMU200. Após condicionamento da dentina, o cimento resinoso e o pino de fibra de vidro foram inseridos, fotoativados por 60s (1200mw/cm²) e armazenados em água destilada (24h/37°C). Dois discos de dentina de 1 mm de espessura foram obtidos de cada terço radicular e testados em push-out (0.5mm/min) após 24 horas e 6 meses. Para análise estatística, os dados dos terços radiculares foram agrupados e resistência adesiva (em MPa) analisada por Análise de Variância de dois fatores e teste de Tukey a 5%. O poder do teste foi calculado. A adesão de the U após 6 meses foi maior que a de PU nos dois períodos (p<0,001). Não houve diferença para o fator tempo (p=0,153) nem para a interação tratamento x tempo (p=0,090). Fraturas adesivas prevaleceram entre cimento e dentina. O poder do teste calculado foi de 0,97. A adesão do RelyXTMU200 foi influenciada pelo pré-tratamento da dentina com ácido poliacrílico e a adesão de todos os grupos se manteve estável até 6 meses.

Palavras-chave: Cimentos Dentários. Dentina. Resistência ao Cisalhamento.

1 Introduction

The category of adhesive procedures includes the use of posts to root dentin to stabilize direct and indirect restorations in teeth that have suffered severe destruction^{1,2}, when there is remaining healthy dentin of the crown height and length³⁻⁶. The fiberglass post is compatible with the resin materials commonly used in adhesive procedures^{7,8} and so, the bonding may be enhanced when associated with adhesive cements⁹⁻¹¹. The low incidence of root fracture is advantageous, due to the similar elastic modulus between the post and the dentin^{12,13}.

The bonding of the fiberglass post to dentin using self-adhesive resin cement is user-friendly to clinics in general⁸.

These cements are said to be self-adhesive to the dental tissue using a single step application technique^{14,15}. Although the concept of self-adhesive cement does not require pre-treatment of the dental substrate, failing to remove the smear layer resulting from the preparation of the root canal is considered one of the variables which could interfere in the bonding procedure^{16,17}.

Having that in mind, there are still doubts regarding the factors that could influence the bonding fiberglass posts bonding to root dentin using self-adhesive resin cements; there is still no consensus in the literature regarding the substrate treatment which will receive the cement and the bonding

durability is still a challenge.

The aim of this study was to evaluate whether different protocols of cementing a fiberglass post to root dentin, using self-adhesive resin cement, influence the bonding over time. To achieve this, an investigation was performed to test the hypotheses that I) There is no difference in bonding whether the root dentin is conditioned with 11.5% polyacrylic acid or not; II) There is no difference in bonding at 24 hours and 6 months.

2 Material and Methods

Following the approval of the Ethical Committee on Research of the dental school (protocol № 462.477), 36 single rooted human pre molars, with completed root apex, free of caries, fractures, lacerated roots and previous endodontic treatment, with at least 14 mm ± 1 between the cement enamel junction (CEJ) and the root apex, were disinfected for 7 days in a 0.5% T chloramine solution at 4 °C.

The dental crowns were sectioned perpendicular to the long axis, at the height of the CEJ to obtain minimum root lengths of at least 14mm and create access to the root canal. The patent canal length (PCL) was the established working limit and the instrumentation performed until the apical foramen. The roots were filled using the Hybrid Tigger technique, maintaining a distance of 1mm from the apex with the Sealer 26® endodontic cement (Dentsply, Dentsply DeTrey, Konstanz, Germany). The cones were condensed by means of a McSpadden. (Easy Equipamentos Odontológicos, Belo Horizonte, Minas Gerais, Brazil).

After allowing for the setting time of the cement, the 36 roots were cleared thermoplastically using Termo Pack II equipment (Easy Equipamentos Odontológicos, Belo Horizonte, Minas Gerais, Brazil). The samples were wrapped in sterile gauze soaked with a 0.9% NaCl solution, packed in individual containers and stored at room temperature for 24 hours. For the final calibration of the passage a 1.6mm diameter Exacto drill bit number 2 was used (Angelus Indústria de Produtos Odontológicos S/A, Londrina, Paraná, Brazil), coupled to a Konzept low speed turbine (Fábrica

KaVo do Brasil Ind. Com. Ltda, Joinville, Santa Catarina, Brazil) to a maximum depth of 9mm from the CEJ.

The prepared roots were irrigated with a 0.9% NaCl solution and dried with absorbent paper towels, before being numbered from 1 to 36 and allocated randomly into three (3) experimental groups (n=12). Each specimen received a translucent fiberglass pin Exacto # 2 (Angelus Indústria de Produtos Odontológicos S/A, Londrina, Paraná, Brazil) with a conical shape, smooth surface, diameter of over 1.6mm and length of 15mm. The surface was cleaned with sterile gauze soaked in Absolute Ethyl Alcohol (F. Maia Indústria e Comércio Ltda., Cotia, São Paulo, Brazil) and dried with air blasts. A silane coupling agent (Angelus Indústria de Produtos Odontológicos S/A, Londrina, Paraná, Brazil), was applied for 60 seconds with an extra thin disposable paint brush (KG Sorensen Indústria e Comércio Ltda, Barueri, São Paulo, Brazil) and the excess removed with air blasts.

The root dentin of the ARC specimens was conditioned with 35% H₃PO₄ gel for 15 seconds; the U specimens were not conditioned, as recommended by the manufacturer; the PU specimens were conditioned with a 11.5% polyacrylic acid solution for 15 seconds. Following this the root dentin in all the groups was washed with water for 30 seconds using a triple syringe and dried with absorbent paper points. The pins in the ARC group were cemented after the application of Adper Scotchbond™ Multipurpose Plus adhesive system (3M-ESPE, St. Paul, MN, USA) with the resin cement RelyX™ARC (3M-ESPE) using a Centrix syringe. In the U and PU groups the pins were cemented with pre-mixed self-adhesive resin cement RelyX™ U200 Automix (Dental Products, 3M-ESPE, St. Paul, USA); and inserted in the canal using manual pressure; the excess cement was removed immediately. The cements activation was performed using a Radium-Call curing light (Southern Dental Industries Limited, Bayswater, Victoria, Australia) with light intensity equals to 1200mW/cm², for 60 seconds in the coronal root direction. The composition and mode of application of the materials used in this study are presented in Table 1.

Table 1 - Material, composition and application mode

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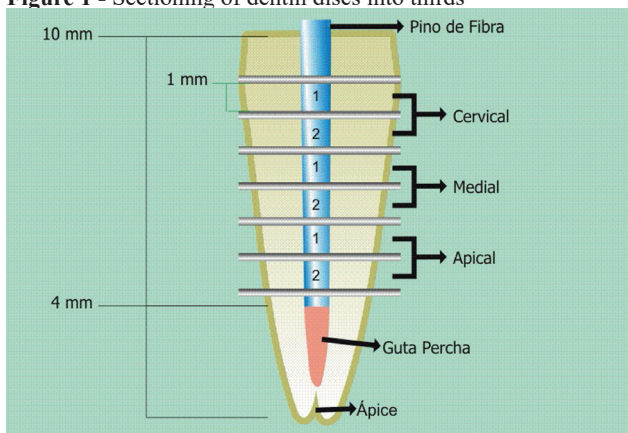
Material / Manufacturer	Composition	Application
Polyacrylic Acid Scotchbond™ / DFL	11,5 % of Polyacrylic Acid	Apply for 15 seconds on dentin and remove with plenty of water for 30 seconds and dry with absorbent paper points.
Phosphoric Acid Conditioner Scotchbond™ / 3M ESPE	35% of Phosphoric Acid.	Apply for 15 seconds on dentin and remove with plenty of water for 30 seconds and dry with absorbent paper points.
Bonding Agent Silano / ANGELUS	monofunctional γ -metacrilosipropiltrimetoxisilano (MPS) and Ethanol.	Apply for 60 seconds and dry lightly with air .
Resin cement RelyX™ U200 Automix / 3M ESPE	Paste A – Amine, bis-GMA, TEGDMA, photoinitiators, inorganic particles of silica and zirconia (68% by weight) and pigments. Paste B – TEGDMA, bis-GMA, inorganic particles of silica and zirconia (67% by weight) benzoyl peroxide.	Apply with the Automix syringe and wait the setting time.

Material / Manufacturer	Composition	Application
Resin cement RelyX ^{MR} ARC / 3M ESPE	Silane-treated ceramic, triethylene glycol dimethacrylate (TEGDMA), bisfenol A diglycidyl ether methacrylate (BISGMA), Silane-treated silica, funcionalized dimethacrylate polymer.	Mix equal parts of Paste A and Paste B (10s) Apply (Centrix syringe), wait the setting time.
ScotchBond Multi Purpose Plus (SBMP) / 3M ESPE	35% Phosphoric Acid SBMP Activator: sulfonic acid etilic solution; photoinitiators Primer: 2-hidroxymethylmetacrilate (HEMA); polialquenoico acid copolimer SBMP Catalyst: Bis-GMA; HEMA; benzoil peroxide	Acid etching (15s); Wash (30s); Dry (absorbent paper); Apply Activador (one coat); Apply Primer; Apply Catalyst
Pino Fiberglass EXACTO / Angelus	80% of glass fiber. 20% of epoxy resin.	Place the pin in the channel to verify its adaptation and integration into complete root.

Source: Authors.

After preparation of the 36 specimens according to their experimental group, the specimens were stored for 24 hours in humidified individual containers in an oven at 37°C. With the aid of an Extec 12205 diamond cutting disc (Erios, São Paulo, São Paulo, Brazil) coupled to an ISOMET 1000 cutting machine (Buhler Ltd., Lake Bluff, IL, USA), at a speed of 200rpm under constant cooling with distilled water, two 1mm slices of each root third were taken from the specimens (Figure 1).

Figure 1 - Sectioning of dentin discs into thirds

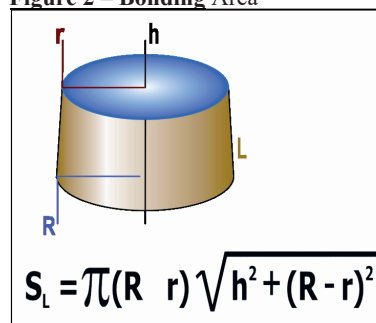


Source: Authors.

After immersion in distilled water, half the specimens were subjected to the push-out test after 24 hours and the other half after 6 months, with weekly changes of the storage medium to assess the bonding durability. Each specimen was fixed in a device and a compression load applied to the slice in the apical – coronal direction so as to push the post, respecting the taper of the root canal, by means of a 1mm diameter cylindrical punch connected to a universal testing machine (Emic DL 2000, São José dos Pinhais, Paraná, Brazil) at a speed of 0.5 mm per minute, using a load cell of 50 kilogram-force (kg_f) until the pin was displaced in the root canal.

The bond strength was obtained in Newtons (N) and converted to Mega-Pascal (MPa) by dividing the maximum load failure obtained by the area of the bonded interface according to the formula (Figure 2): $S_L = \pi [(R-r) \sqrt{h^2 + (R-r)^2}]$.

Figure 2 – Bonding Area



Source: Authors.

After being subjected to mechanical testing the dimensions of the specimens were measured using a DIGIMESS digital caliper (Digimess Instrumentos de Precisão Ltda; São Paulo, São Paulo; Brazil) with an accuracy range of 0.02 mm. The failure surfaces were examined through a Bel Photonics optical microscope (Bel Microimagre Analyser, Monza, Italy) magnified 40X to determine the type of failure, categorized as follows: (1) *adhesive failure* - between the pin and the self-adhesive cement, (2) *adhesive failure* - between the self-adhesive cement and the root dentin, (3) *cohesive failure* of the pin system, (4) *cohesive failure* of the self-adhesive cement, and (5) *mixed type*, a combination of both types of the above failures.

For statistical purposes, the data of the root thirds were grouped and the root considered as the experimental unit. Bond strength (in MPa) was analyzed using MANOVA General Linear Model and Tukey’s post-hoc test at 5%. The test power was calculated.

3 Results and Discussion

Data analysis showed no outliers at a significance level of $\alpha = 5\%$ (Grubbs test; $p = 0.299$). The Kolmogorov-Smirnov test showed normal distribution (KS = 0.079). The Test Power calculated was 0.97. A statistical difference was observed for the treatment of root dentin ($p < 0.000$). The bonding was higher in the U group at 6 months and lower in the PU group at both evaluation moments. The bonding in the U group at 24h was similar to the ARC and PU groups. The bonding at 24h

was similar to that at 6 months in all the groups. There was no difference for time ($p = 0.427$) or for the interaction between

treatment and time ($p = 0.093$). Table 2 presents the average bond strength values (MPa) according to the experimental groups.

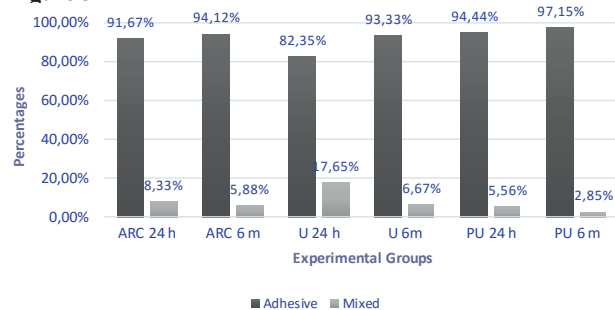
Table 2 - Mean Bond strength values (standard deviations) in MPa of experimental conditions

TIME	Surface Treatment						p %
	ARC		U		PU		
	MEAN	SD	MEAN	SD	MEAN	SD	
24 hours	4.99	(1.79) ^{AB}	5.04	(1.36) ^{AB}	3.42	(1.27) ^B	0.000
6 m	4.54	(2.41) ^{AB}	6.79	(2.41) ^A	3.19	(1.75) ^B	
p %	0.427						

Source: Authors.

Observation of the fracture pattern showed predominance of adhesive failure between the cement and dentin in all the experimental groups, followed by mixed failures at a lower percentage. There was no cohesive failure. The types of fractures are presented in Figure 3, in percentages (%) of incidences in the experimental groups, at 24 hours and 6 months.

Figure 3 - Distribution of fracture modes



Source: Authors.

The objective of this study was to evaluate the bonding of self-adhesive resin cement to root dentin, testing the effect of prior conditioning with 11.5% polyacrylic acid, over six months. The results showed that the bonding was influenced by the dentin treatment, whereby the roots to which the cement was applied as recommended by the manufacturer presented greater bonding after six months than the group conditioned with polyacrylic acid at both moments, rejecting the first hypothesis of this study.

Some authors have observed that the use of ultrasound equipment during endodontic treatment can optimize cleaning of the root canal and facilitate the interaction between the cement and the dentin, thereby increasing bonding¹⁸, particularly in the apical third. In the present study the application of polyacrylic acid was performed manually, which probably explains why this treatment did not result in greater cement bonding; since polyacrylic acid is a weak acid and may not have been sufficient to facilitate the smear layer removal. A previous study¹⁹ also did not observe increased bonding of RelyX Unicem cement to dentin after conditioning with polyacrylic acid in the same concentration as used in this study.

It has been observed that the smear layer produced on the root canal during endodontic treatment is thicker than that of

coronal dentin²⁰ and that the capacity of some self adhesive cements to pass through this and interact with the underlying dentin could also be hampered by the high viscosity of the material and by the buffering effect which happens during the material setting²¹. In a previous study²², conditioning of the coronal dentin with polyacrylic acid removed the smear layer and exposed the collagen fibrils (0.5-1.0 micrometer), and favored the ionic interaction between the carboxyl groups of the acid and calcium ions on the hydroxyapatite²³; it was thought that the same might happen with the removal of the root dentin smear layer, however, the present study did not confirm this result.

It is possible that the use of other acids such as 17% EDTA solution could be considered as it is routinely used as the final step in the instrumentation of the root canal system. However, the use of this acid prior to RelyX Unicem, the forerunner of RelyX U200, decreased adherence¹⁹, a result explained by the authors due to a possible increase in humidity in the dentin after removal of the smear plugs, which may have interfered with the polymerization of the self-adhesive cement, allied to the high viscosity of the material, which would not have interacted with the dentin. The same explanation could be applied after the conditioning with polyacrylic acid in the present study.

In the present study it was also observed that the bonding was maintained over six months in all the groups, regardless of the pre-treatment of the dentin with polyacrylic acid, verifying the second hypothesis of the study. In other words, application of the cement using the method recommended by the manufacturer was sufficient to adhere the fiberglass pin to the root dentin. Self-adhesive resin cement does not require the pretreatment of the dentin surface of the root canal due to superficial interaction with the underlying dentin²¹.

According to the manufacturer, RelyX U200 presents chemical union with the hydroxyapatite (a mechanism similar to that of glass ionomer cement modified by resin, but with the substitution of polyacrylic acid with an acid functional monomer of the type phosphoric acid ester). The manufacturer emphasizes that the cement pH ($pH = 2$) undergoes neutralization soon after application, and that this leads to the bonding longevity, as observed in the present study. Another aspect highlighted in previous studies^{17,24} was that

RelyX U200 is more hydrophobic and tolerant to moisture. The decrease in the material viscosity, in relation to RelyX Unicem, may also have facilitated the interaction of the cement with the dentin in this study.

The similarity observed between the bonding of the U group after 24 hours and the PU group at both evaluation moments suggests that the presence of the smear layer did not negatively interfere in the bonding fiberglass pin bonding to the dentin during the 6 months, disagreeing with the results of earlier studies which tested other self adhesive cements^{10,25,26}. It is known that in a systematic review of in vitro studies²⁷, self-adhesive resin cements were considered as efficient at joining fiberglass pins to dentin as those using acid conditioning and adhesive systems, with the advantage of having a simplified cementation technique, as shown in this study.

One differentiating aspect of this study was to evaluate the adherence considering the root as the experimental unit, grouping the root-thirds. This and other methodological aspects of the push-out test such as the formula used in the calculus of the adhesive area, vary among different research centers, making it difficult to compare results of different studies. Despite these limitations, the present study suggests that the use of RelyX U200 self-adhesive resin cement demonstrates promising results for cementing fiberglass pins. The realization of further clinical studies could contribute with additional clarification to the issue of intra-radicular cementation.

4 Conclusion

Taking into account the limitations of this research it can be concluded that the bonding of a fiberglass post to dentin using RelyX™ U200 does not require prior conditioning of the root dentin with polyacrylic acid. Irrespective of the dentin treatment, the bonding remained stable for 6 months.

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