










Comparative evaluation of the strength of adhesion by push-out bond strength tests on endodontic posts cemented with different resinous types of luting cements: An in vitro study

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Abstract

INTRODUCTION. Endodontic posts play a vital role in strengthening debilitated teeth. They are luted into the radicular space using dental cements that are generally resinous. Given that the forces faced by human teeth are loading in nature and the stresses are accumulative, the bond strength of the interfaces at both the radicular dentin and luting agent and the endodontic post and the luting agent needs to be evaluated. Micro pushout bond strength (POBS) testing is done to determine these strengths. This experimental, in vitro study evaluated the adhesion strength of endodontic posts cemented with different resin-based luting agents using micro-POBS.

MATERIALS AND METHODS. A total of 30 human lower incisors were selected and divided into three groups ($n=10$), based on the type of adhesive resin cement used for luting. Prefabricated endodontic fiber posts were inserted into the root canals of each tooth, and different resin types of cement – Estecem II, Multilink Automix, and Relyx Ultimate. were used to cement the fiber posts. The micro-POBS test measured the bond strength of the cemented pins to the root canal dentin.

RESULTS. The results showed that the mean POBS of Group B was higher than that of Group A and Group C. The difference between the mean POBSs of Group A and Group C was not statistically significant ($p > 0.05$).

CONCLUSION. The resin adhesive cement used can affect the POBS of endodontic posts to root canal dentin. Clinicians should choose the appropriate resin cement for cementing endodontic posts to ensure a strong and durable bond for a good clinical prognosis.

Keywords: endodontic posts, resin-based luting agents, micro pushout bond strength, resin cement

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Сравнительная оценка прочности адгезии с помощью тестов на выталкивание эндодонтических штифтов, цементированных различными типами композитных цемента: лабораторное исследование

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Резюме

ВВЕДЕНИЕ. Эндодонтические штифты играют важную роль в укреплении ослабленных зубов. Они фиксируются в корневом канале с помощью стоматологических цементов, которые обычно являются смолистыми. Учитывая, что на зубы человека воздействуют постоянные нагрузки и накапливаются

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стрессы, необходимо оценивать прочность сцепления как между корневым дентином и фиксирующим агентом, так и между эндодонтическим штифтом и фиксирующим агентом. Для определения этих показателей проводится тест на микровыталкивание (POBS). Это экспериментальное *in vitro* исследование оценивало прочность адгезии эндодонтических штифтов, зацементированных различными смоляными фиксирующими агентами, с помощью теста на микровыталкивание.

МАТЕРИАЛЫ И МЕТОДЫ. Было выбрано 30 нижних резцов человека и разделено на три группы ($n = 10$), в зависимости от типа использованного адгезивного смоляного цемента. В корневые каналы каждого зуба были установлены предварительно изготовленные эндодонтические стекловолоконные штифты, которые фиксировались с использованием различных смолистых цементов: Estecem II, Multilink Automix и Relyx Ultimate. С помощью теста на микровыталкивание оценивалась прочность сцепления зацементированных штифтов с дентином корневого канала.

РЕЗУЛЬТАТЫ. Результаты показали, что среднее значение POBS в группе В было выше, чем в группах А и С. Различие между средними значениями POBS в группах А и С не было статистически значимым ($p > 0.05$).

ЗАКЛЮЧЕНИЕ. Используемый смоляной адгезивный цемент может влиять на POBS эндодонтических штифтов к дентину корневого канала. Врачи должны выбирать подходящий смоляной цемент для цементирования эндодонтических штифтов, чтобы обеспечить прочное и долговечное соединение для хорошего клинического прогноза.

Ключевые слова: эндодонтические штифты, композитные фиксирующие агенты, прочность адгезии при микровыталкивании, композитный цемент

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INTRODUCTION

Endodontic treatment may become necessary due to extensive caries, trauma, fractures, and periodontal pathologies, which have led to the development of an irreversible pulpal pathology, such as irreversible pulpitis or necrosis or even in vital healthy teeth that require intentional endodontic treatment even in the absence of any pulpal or periapical pathology [1–3]. When there is extensive loss of tooth structure, and the tooth is debilitated at both the macro (visible tooth loss) and the micro (depletion of inorganic content) levels, and in order to restore its form and function, the tooth needs to be reinforced using an endodontic post. The most commonly used pre-fabricated endodontic posts in restorative dentistry, today, are fibre posts [3; 4]. These posts are cemented into the radicular space using luting agents which could be – GIC-based, zinc phosphate-based, or resin-based luting systems. Using a system that provides a seamless interface – a monobloc, with the root canal dentin – luting agent-post system, would be ideal, as it would be biomimetic. The endodontic post on its own is incapable of binding to root dentin, and requires a luting agent, which on the one side bonds to the dentin and on the other side bonds to the endodontic post [5–7]

Research has shown that adequate post-endodontic reconstruction gives the devitalized tooth, a functional capacity almost equal to that of the vital tooth, and the long-term success of endodontic treatment depends on the execution of a suitable coronal restoration, which guarantees a coronal seal and mechanical reinforcement to the treated dental biostructures [8].

Over the years there has been a slow but steady shift away from luting agents like GIC and $ZnPO_4$, which are used primarily to cement metal-based post systems, to the adhesive resin systems and calcium silicate material-based cements for the luting of the fibre and

fibre-reinforced posts. The adhesive luting agents are categorized as per generations and newer and newer systems enter the market periodically. The clinician is hard-pressed to choose these and it is generally the ready availability and the cost of the luting agent which dominates the selection of the luting system [9].

Achieving a “monoblock” within the radicular space is a desirable but intangible goal. Given the varied materials involved in the tertiary kind of monoblock which is typically created in the root canal space, the best that could happen would be to have retention of the root canal filling material or posts within the radicular space and the transmission and eventual dissipation of forces via the luting cement [10–12].

Using a micro pushout BS test on luting agents mimics the dynamic forces that a tooth faces during mastication and it impacts the integrity of the bond of the luting agent with the post and the radicular dentin [13; 14].

AIM

This experimental analysis aimed to compare the retentive capacity of quartz fibre posts, when luted into the root canal using three different resin-based types of cement – Estecem II, Multilink Automix, and Relyx Ultimate, using an *in vitro* experimental test model – the micro-POBS, to highlight their efficacy, predictability, and differences and to take stock of the current technological and product situation. It would thus allow the clinician to make an informed choice about the choice of a luting agent based upon the moduli of elasticity rather than an adhesive strength test. Further the evaluation of the mode of failure in the samples allowed for identification of the adhesive interface that displayed the most instances of failure – radicular dentin – luting agent interface vs. post – luting agent, which meant that the bonding procedure could be improved at that interface.

MATERIALS AND METHODS

Study design

The present experimental study used three types of cement for cementing endodontic pins are, as described below:

- Estecem II, used for group A cementation – Bis-GMA, TEGDMA, Bis-MPEPP, Silica-Zirconia Filler;
- Multilink Automix, used for group B cementation – Dimethacrylate and HEMA;
- Relyx Ultimate, used for group C cementation – Methacrylate monomers.

Sample size and sample preparation

The sample size was calculated using G-Power 3.1.9.7. A power analysis was performed where $\alpha=0.05$, Power = 0.80 and Effect size 0.91. A total of 30 sample size was calculated, divided into ($n=10$) in each group.

30 human lower incisors without caries were selected, which had been extracted due to periodontal causes, and were subjected to photographic and radiographic examination, to verify the presence of only one non-calcified straight canal and to exclude the presence of fractures. The narrow diameters of the mandibular incisors are consistent across all the teeth and are thus good to use in the micro-POBS, giving standardized results.

After extraction, the teeth were autoclaved and stored at room temperature in 2% glutaraldehyde to maintain disinfection and maintain hydration [15].

The teeth were cut 1 mm above the cemento-enamel junction with a diamond bur. 856 012 Komet (Komet, Lemgo, Germany), under abundant irrigation. The canal patency was established by using 8 and 10 number K-Files. Cleaning and shaping were done for all the teeth with the M-two system, and the apex was enlarged up to MTwo Apical A3 25/45. Irrigation was done with EDTA 17% (EDTA canal cleaner, Dentalica, Milan, Italy) and hypochlorite 5.25%. Canals were obturated with Touch and heat and the compaction of the obturation was radiographically assessed, and repeated when voids were detected in the apical one-third. The post space was prepared with a TokuDrill (Tokuyama Dental Corporation, Tokyo, Japan) at 15000 rpm under a microscope, by the same operator. VersaBrush Endobrush (Vista, Dental Products, Racine, WI, USA) with 10,000 rpm pumice was used to remove any gutta perch residue from the post-space, which was then confirmed under a microscope. The teeth were then divided into 3 groups of 10 each, depending upon the type of luting agent being used.

- Group A ($n=10$), cementation with Estecem II;
- Group B ($n=10$), cementing with Multilink Automix;

- Group C ($n=10$), cementing with Relyx Ultimate.

The posts were treated with alcohol and air-dried before use. Each luting agent was manipulated as per the manufacturer's instructions and posts were luted to the radicular dentin. After curing the luting agent, the fit was confirmed radiographically and the extra post length was allowed to remain for ease of slicing the samples. A high translucency, variable taper quartz fiber radiopaque post system called TokuPost was used. This is pre-silanized and was used for all the samples. The slices were prepared by making horizontal cuts with a diamond saw (Isomet, Buehler; Lake Bluff, IL, USA) at 250 rpm under abundant irrigation, obtaining 5–6 samples from each tooth having an approximate thickness of 1 mm. Each slice was marked with an indelible marker on the apical side. The thickness of each slice was confirmed with an electronic gauge (accuracy 0.01 mm). 48 samples were obtained from group A, of which 27 were from the coronal third of the tooth, 21 were from the middle third of the tooth, 58 were from group B, of which 30 were from the coronal one-third, 28 were from the middle-third, 57 from group C, of which 30 from the coronal one third, 27 from the middle third (Fig. 2, 3).

Push-out test

The micro push-out test was performed at the University of Siena (Siena, Italy). The machine used is a Universal Testing machine on which a cylindrical piston (Triax Digital, Controls, Milan) has been mounted, which directs the pressure load on the pin at a speed of 0.5 mm/min (Fig. 4, 5).

Statistical analysis

The data was entered and analyzed using the Statistical Package for Social Sciences (SPSS) for Windows, Version 28.0 (Armonk, NY: IBM Corp) Confidence intervals were set at 95%, and a p-value \leq of 0.05 was considered statistically significant. One-way ANOVA was applied to compare the three luting agents and post hoc test was applied for intragroup significance.

RESULTS

The statistical analysis shows higher force values for group B (Relyx Ultimate 16,74 2,48 MPa), compared to groups A (Estecem II 11,06 2,76 MPa) and C (Multilink Automix 10,73 2,73 MPa), which report no statistically significant differences (Table 1, 2). An analysis of the modes of fractures shows a prevalence of adhesion failure between cement and dentine compared to adhesion between pin and dentine.

(MPa) Standard deviation (DS) for the group, Median, and fracture (Fig. 6).



Fig. 1. Materials used for cementation in Group A, B, and C and samples

Рис. 1. Материалы, использованные для цементирования в группах А, В и С, и образцы

TokuPOST h15r	
Total length	15 mm
Length on capacity part (cylindric)	7 mm
Length on capacity part	8 mm
Capacity diameters	Ø1.05–Ø1.25–Ø1.45
Apical diameters	Ø0.55–Ø0.75–Ø1.95
TokuPOST h19S	
Total length	18 mm
Length on capacity part (cylindric)	7 mm
Length on capacity part	11 mm
Capacity diameters	Ø1.25–Ø1.45–Ø1.65
Apical diameters	Ø0.55–Ø0.75–Ø1.95

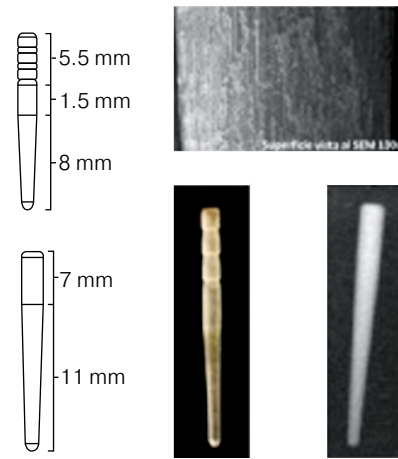


Fig. 2. Description of TokuPOST endodontic pins

Рис. 2. Описание эндодонтических штифтов TokuPOST

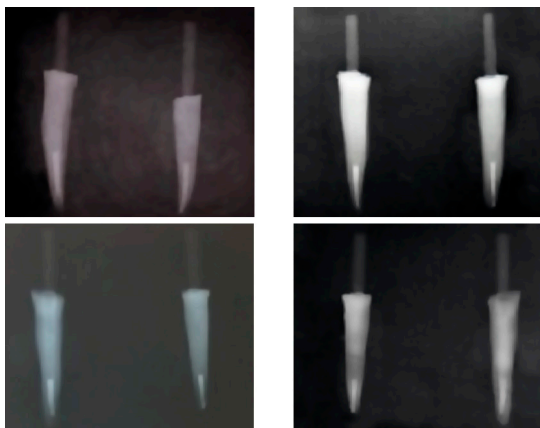


Fig. 3. Radiographical examination of some samples after hardening of the pins

Рис. 3. Рентгенологическое обследование некоторых образцов после затвердевания штифтов

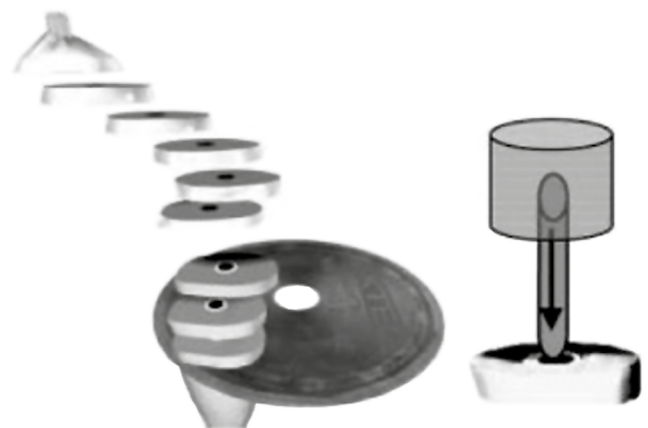


Fig. 4. Sample preparation for push-out testing

Рис. 4. Подготовка образцов для тестирования на выталкивание

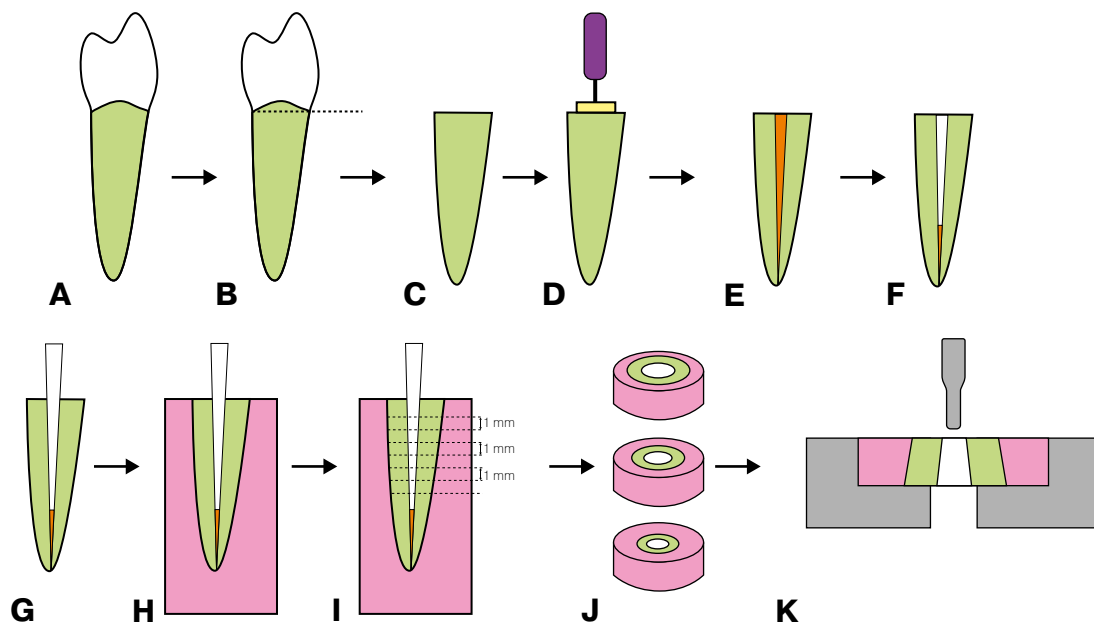


Fig. 5. The root is cut into slices 1 mm thick after the pin is cemented, a force is applied to each slice until the pin is dislocated

Рис. 5. Корень разрезается на срезы толщиной 1 мм после цементирования штифта, к каждому срезу прикладывается сила до тех пор, пока штифт не сместится

Table 1. Statistical analysis for individual root regions for each group**Таблица 1.** Статистический анализ отдельных областей корня для каждой группы

Group	Root region	Mean \pm DS	Mean \pm DS	Median	DC	PC	DF
EST	Cervical	11.76 \pm 2.78	11.06a \pm 2.76	10.73	16	8	24
	Middle	10.40 \pm 3.85					
REL	Cervical	17.79 \pm 2.96	16.74b \pm 2.48	16.62	17	8	33
	Middle	15.56 \pm 3.35					
MUL	Cervical	11.24 \pm 2.20	10.73a \pm 2.73	10.64	36	2	19
	Middle	10.20 \pm 3.28					

Note. EST: Estecem II, REL: Relyx Ultimate, MUL: Multilink Automix, DC: failure adhesion between dentine and cement, PC: failure adhesion between post and cement, DF: fracture of the element. Values with the same letters indicate non-significant differences ($p > 0.05$).

Примечание. EST: Estecem II, REL: Relyx Ultimate, MUL: Multilink Automix, DC: отказ адгезии между дентином и цементом, PC: отказ адгезии между штифтом и цементом, DF: перелом элемента. Значения с одинаковыми буквами указывают на незначительные различия ($p > 0.05$).

Table 2. Mean and (MPa) standard deviation (DS) for individual root regions for each group**Таблица 2.** Средние значения и стандартное отклонение (МПа) для отдельных областей корня для каждой группы

Group	Coronal Region	Medial Region
	Mean (MPa) \pm DS	Mean (MPa) \pm DS
EST	11.76 ^a (MPa) \pm 2.78	10.4 ^a (MPa) \pm 3.85
REL	17.79 ^b (MPa) \pm 2.96	15.56 ^b (MPa) \pm 3.35
MUL	11.24 ^a (MPa) \pm 2.21	10.2 ^a (MPa) \pm 3.23

Note. EST: Estecem II, REL: Relyx Ultimate, MUL: Multilink Automix, Values with the same letters indicate non-significant differences ($p > 0.05$).

Примечание. EST: Estecem II, REL: Relyx Ultimate, MUL: Multilink Automix. Значения с одинаковыми буквами указывают на незначительные различия ($p > 0.05$).

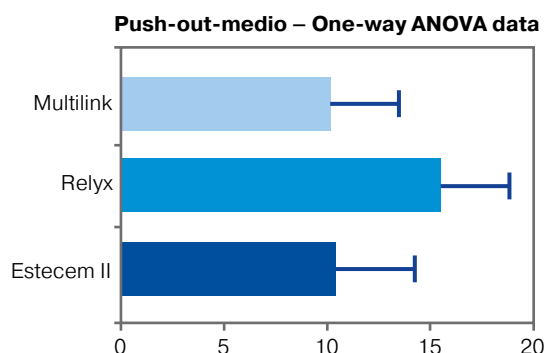
**Fig. 6.** Statistical analysis One-way ANOVA third medium results

Рис. 6. Статистический анализ: результаты однофакторного дисперсионного анализа (ANOVA) для третьей среды

DISCUSSION

Non-vital dentin demonstrates singularly distinct properties from that of vital dentin. The major differences are:

- reduction in the modulus of elasticity;
- reduction in the hydration of the dentin (loss of freely bound water);
- loss of mineral content from the dentin owing to chelating agents and irrigating solutions;
- actual loss of dentin due to the shaping with root canal instruments;
- crack initiation and propagation in the intertubular dentin.

This makes it vulnerable as an adhesive substrate, particularly in the radicular space where removing the smear layer is also difficult. This study thus sought to examine the micro push-out bond strengths of three commercially available adhesive systems – Estecem II, Multilink Automix, Relyx Ultimate for the luting of endodontic posts, commercially available as Tokuposts [3; 9].

The adhesives on the market are either etch-and-rinse or self-etch, and in the past, while the etch-and-rinse systems were used in the radicular space, the self-etch type of adhesive was indicated for use in the coronal area, and its use was not indicated in the radicular space [16]. Recently though the advances in the science of self-etch bonding agents have seen a rise in the usage of self-etch in the radicular space.

This type of adhesive is intended to simplify the clinical procedure, as they include acids and hydrophilic monomers, in the same bottle, which are intended to demineralize and infiltrate enamel and/or dentine and do not require pretreatment of the substrate.

Estecem II cement has been chosen because it is a material recently introduced in the market, of which data do not yet exist in the literature. In contrast, the cement Multilink Automix and Relyx Ultimate have both been chosen as they are widely used in clinical practice, and have the same type of polymerization mechanism and adhesion as that of Estecem II, but different monomers. The evaluation of the luting agent's efficacy is done by micro-POBS, along with an assessment of the mode of failure [17–21].

The push-out test is the most reliable and most used test for evaluating the bonding force between endodontic fiber posts and root canal dentin, as it closely approximates the dynamic masticatory forces that a functional tooth is subject to. Micro POBS also shows far fewer premature failures [22].

In this study, a load was applied via a piston only on the luted post, without contacting the root canal walls. As opposed to push-out tests, the micro push-out tests allow for the distribution of forces across the interface more uniformly and permit microscopic evaluation of the various regions of the sample separately to determine the fracture modes of individual samples [23–26].

The fibre posts cemented with Relyx Ultimate cement (16.74–2.48 MPa) showed a higher retention than those cemented with Multilink Automix (10.73–2.73 MPa) and Estecem II (11.06–2.76 MPa) ($p < 0.05$), which do not show statistically significant differences between them ($p < 0.05$) [27].

The bond strengths achieved with the etch and rinse adhesive decrease coronally similar to the self-

etch adhesives. Since the bonding mechanism in this group is based on micromechanical retention following elimination of the smear layer and exposure of dentinal tubules, a decrease in bond strength from the coronal part towards the apical segment can be attributed to the partial removal of the smear layer and various other debris including remnants of gutta-percha, preventing unilateral, seamless contact of the adhesive cement with the radicular wall [28].

The same result was seen in this study when the middle third and coronal third of the radicular dentin were examined and compared. No literature is available on Estecem II cement, due to the recent marketing and therefore it is not possible to compare our results with those of other studies. According to information provided by the manufacturer, Multilink Automix cement has been used in conjunction with the self-etching, self-curing Multilink Primer A&B adhesive system. The combination of Multilink cement with the indicated adhesive system accelerates the curing of cement, making it more efficient. Relyx Ultimate cement is a combination with Scotchbond Universal adhesive, and contains 10-methacryloyloxydecyl dihydrogen phosphate (MDP) monomer, which increases the chemical bond established between phosphate groups and residual hydroxyapatite crystals on the structure of dentine collagen fibres [28–30]. This chemical bond reduces the degradation of the hybrid layer and shows greater water stability than other functional monomers. The different chemical properties peculiar to the various types of cement could justify the results obtained.

An analysis of the results for the two root regions shows that the values obtained are slightly higher for the third coronal relative to the middle third, but the difference is not statistically relevant ($p < 0.05$) (Table 2). This can be justified by an incomplete polymerization even if the cement is dual, a lack of photoactivation can make their self-polymerization only partially efficient,

especially at the apical level. It is advisable always to carry out photopolymerization because studies have shown an incomplete hardening in the absence of it. Dual cure cement is recommended for the cementation of fiber posts because photopolymerization is not effective at the most apical levels of post-space. The adhesion strength in the middle third is generally lower than in the coronal one-third, which could be due to the unfavorable C factor [12]. However, reduced bonding in the procedures we have used, which is normally most effective in the third coronal canal, could justify the results obtained, which show a reduction in micro push-out bond strength from the coronal to the apical segments.

CONCLUSION

This study has analysed by micro push-out test the adhesion strength obtained by luting Tokupost quartz fibre pins using Estecem II, Relyx Ultimate, and Multilink Automix cement. All the luting cements used are dual polymerization and have been used with a self-etch technique.

The difference in bond strengths in the different root regions was assessed and based on the limitations imposed by the experimental design, the following conclusions can be drawn:

1. Relyx Ultimate cement showed higher adhesive strength than Multilink Automix cement and Estecem II cement which showed no statistically significant differences between them.

2. Concerning the third coronal and the third middle, the total statistics confirm that the cement Relyx Ultimate demonstrates greater adhesion strength than the other two groups in both zones of the root.

3. The adhesion force obtained in the middle third is lower in absolute value, although not reaching the level of significance, than that obtained in the coronal third, this demonstrates a homogenous adhesion efficiency at all levels of the post-space.

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