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Degree of conversion and adhesion of methacrylate-based resin cements with phosphonic or phosphoric acid acrylate to glass fiber posts at different regions of intraradicular dentin

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Degree of conversion and adhesion of methacrylate-based resin cements with phosphonic or phosphoric acid acrylate to glass fiber posts at different regions of intraradicular dentin

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Short title: *Resin cement adhesion on glass-fiber post to root dentin.*

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Abstract: This study evaluated the degree of conversion (DC) and adhesion of methacrylate-based resin cements to glass fiber posts at different regions of intraradicular dentin. Single-rooted teeth (N=24, n=12 per group) were cut at the cement-enamel junction (CEJ), endodontically treated and post space (depth= 8 mm) was prepared. Teeth were randomly divided into two groups according to the resin cements: a) Group ML: Methacrylate-based cement with phosphonic acid acrylate (Multilink Automix, Ivoclar Vivadent); b) Group RXU: Methacrylate-based cement with phosphoric acid acrylate (RelyX Unicem 2 Automix, 3M ESPE). Fiber reinforced composite root posts (RelyX Fiber Post, 3M ESPE) were cemented according to the manufacturers' instructions of the resin cements. Root slices of 2 mm thick (n=3 per tooth) were cut below the CEJ 1, 3, 5 mm apically. The DC of each section was analyzed with micro-Raman spectrometer and push-out test was performed in the Universal Testing Machine (0.5 mm/min). After debonding, all specimens were analyzed using optical microscope to categorize the failure modes. While data (MPa) were statistically evaluated using Kruskal Wallis, Mann-Whitney U tests, for DC data 3-way ANOVA and Tukey`s tests were used (α=0.05). Regardless of the resin cement type, the mean push out bond strength results (MPa), were significantly higher for the coronal slices (ML: 9.1±2.7; RXU: 7.3±4.1) than those of the most apical ones (ML: 7±4.9; RXU: 2.89±1.5) (P=0.002). Resin cement type and (P<0.001) root level (P=0.002) significantly affected the DC values, while the interaction terms were not significant (P=0.606). Overall, DC was significantly higher for ML (67±8.2%) than RXU (26±8.8%) (P<0.001). Adhesive failures at the cement-dentin interface were more commonly experienced in RXU than in ML, whereas ML presented more incidences of adhesive failures at the cement-post interface. Considering the push-out bond strength, degree of conversion and failure types, methacrylate-based cement with phosphonic acid acrylate should be preferred to those containing phosphoric acid to adhere glass fiber posts in the root canal.

Keywords: Adhesion, degree of conversion, fiber post, fiber-reinforced-composites, self-adhesive cement

Introduction

The use of translucent root posts supporting the core material especially under the all-ceramic crowns in the anterior region of the mouth is desirable for improved aesthetics as opposed to the metal posts. The favourable physical properties of some fiber reinforced composite (FRC) posts enable the light transmission through their structure, allowing the light to polymerize resin-based materials in the root dowel space [1]. Additionally, the clinical success of FRC posts has been mainly credited to their mechanical features, such as lower elasticity modulus than those of metal or ceramic posts [2,3], thereby, reducing the incidence of root fractures. Due to their matrix composition, FRC posts are chemically compatible with methacrylate based adhesive systems and can be adhesively cemented in the root canal [4-7]. Unfortunately, microscopy analysis often showed incidence of voids and bubbles at the interface between the resin cement and root dentin [4] that resulted in reduced adhesion of the post in the root canal [8].

Recently, prospective clinical studies investigating the performance of translucent fiber posts suggest that the cementation step is one of the main reasons of clinical failure [9,10]. The failures in the form of voids were partially attributed to multiple steps in cementing the post starting from conditioning the dentin with phosphoric acid, primer and adhesive resin application to condition the post surface, silanization and adhesive resin application. In order to save time and reduce procedural errors, self-adhesive resin cements were introduced, reducing the cementation procedure to only one step [11]. Such cements contain bifunctional monomers containing phosphonic or phosphoric acid acrylates, 10-Methacryloyloxydecyl dihydrogen phosphate (MDP), bis-HEMA-phosphate, glycerolphosphate dimethacrylate and do not necessitate conditioning neither the root dentin nor the post surface. The reliability of self-adhesive cements has yet to be validated on the post cementation procedures.

The objectives of this study therefore were a) to evaluate the degree of conversion and push-out bond strength of glass fiber posts cemented with methacrylate-based resin cements with either phosphonic or phosphoric acid acrylates at different regions of the intraradicular dentin and b) to analyze the failure types after debonding. The null hypotheses tested were that there would be no difference between degree of conversion and adhesive properties of the two cements in all depths of the root canal.

Materials and Methods

Specimen preparation

Single rooted (N=24) human teeth extracted for periodontal reasons not longer than 6 months were collected and cleaned of all debris with a curette. They were then cut at the cement-enamel junction (CEJ) using a cylindrical diamond-coated bur (diameter: 0.14 mm) driven by a turbine, under constant water-cooling. Root canals were firstly shaped with K-file from number 8 to 25 and, later, with rotary Ni-Ti files (Protaper[®] series S1, S2, F1, F2, Dentsply Tulsa Dental, Tulsa, Oklahoma, USA). During the shaping process, teeth were cleaned with 5% NaOCl and 10% EDTA. Each root canal was dried with sterile paper points. Then, the canals were filled with standardized gutta percha points (ProTaper, Dentsply) simulating the standard endodontic treatment. Post space (depth=8 mm) was prepared in each tooth using Gates Glidden burs (size 1 to 3), largo burs (size 1 to 4) and the dedicated burs corresponding to the final size of the chosen post, in order to remove all of the residuals of gutta percha from the root canal walls. Each post space was then cleaned using the dedicated brushes driven by a low-speed hand-piece and saline rinsing.

Fiber reinforced composite posts (RelyX™ Fiber Post, 3M ESPE, Seefeld, Germany) with double-conical shape (diameter: 1.6 mm coronal, 0.8 mm apical) were cleaned in alcohol prior to cementation.

Teeth were randomly divided into two groups according to the resin cements (n=12 per group): a) Group ML: Methacrylate-based cement with phosphonic acid (Multilink Automix, Ivoclar Vivadent, Schaan, Liechtenstein); b) Group RXU: Methacrylate-based cement with phosphoric acid acrylates (RelyX Unicem 2 Automix, 3M ESPE) (Table 1).

In Group ML, after etching the root dentin with 37% H_3PO_4 for 30 s, rinsing for 30 s, and drying with paper points, a thin layer of adhesive (Multilink Primer A/B) was applied using an extra-fine microbrush (Microbrush applicator, 3M ESPE) according to manufacturer`s instructions.

Both cements were mixed according to the manufacturer's instructions and applied in the canal using a special applicator (Centrix[®] tip) in order to avoid the possible void formation. Subsequently, the posts were inserted gently into the post space. The cements were polymerized accordingly with an LED polymerization device (Bluephase G2[®], Ivoclar Vivadent; output: 1200 mW/cm[2]) keeping the light tip

orthogonal to the post at a 2 mm constant distance. One operator performed all cementation procedures. The specimens were stored in distilled water in the dark for 24 hours at 37°C.

The apical one third of the specimens (3 mm) were embedded in acrylic resin. Root slices of 2 mm thick were cut from the CEJ apically 1 (coronal), 3 (middle), 5 (apical) mm distant to CEJ, perpendicular to the long axis of the tooth using a diamond-coated saw a under water-cooling (Isomet Buhler, Buffalo, NY, USA) at slow speed (Fig.1).

Degree of conversion measurement

The surface of the slices corresponding to 1, 3 and 5 mm distant to the CEJ were analyzed with the micro-Raman spectrometer (LabRam HR, Horiba, Kyoto, Japan) using Helium/Neon laser (wavelength: 632.8 nm; acquision time: 100 s). Two spectra were collected for each specimen and then a baseline correction was applied (Figs. 2a-c).

Push-out bond strength test

The thickness of the specimens was measured using a digital caliper, and then they were fixed to the loading jig. The push-out test was performed in the Universal Testing Machine (Triax 50, Controls SPA, Milano, Italy) where a cylindrical stainless steel punch applied compressive load proportional to the diameter of the post material only, without contact with dentin (0.5 mm/min).

The push out bond strength result (R) (MPa) was calculated dividing the load to failure (F) (Newton) by the area (A) of the interfacial area (mm^2):

$$R = F/A, \text{ where}$$

$$F = \text{load for rupture of the specimen (N)}$$

$$A = \text{interfacial area (mm}^2\text{)}$$

Interfacial area (A) of the specimens were calculated using the formula employing the lateral area of the frustum of a right circular cone with parallel bases (Fig. 3a) $\underline{A=\pi \times g \times (R_1+R_2)}$, where, A=interfacial area, π=3.14, g=slant height of the cone or generatrix of the frustum, R_1=radius of the smaller base, and R_2=radius of the larger base. For calculation of the generatrix of the cone frustum g (Fig. 3b), the Pythagoras' theorem was employed: $g_2=h_2+[R_2-R_1]^2$, where g=generatrix of the frustum, R_1=radius of the smaller base, R_2=radius of the larger base and h=section's height [12].

Failure analysis

After debonding, the slices were analyzed under an optical microscope (Nikon Eclipse I150, Tokyo, Japan) at x30 and the failures were classified as follows: AD: Adhesively (A) debonded from dentin (D), complete detachment of cement from the dentin, AP: Adhesively (A) debonded from post (P), complete detachment of the cement from the post; C: Cohesive (C) failure in the dentin; M: Mixed (M) failure, a combination of adhesive and cohesive failures.

Statistical analysis

Statistical analysis was performed using SPSS 12.00 (SPSS, Chicago, IL, USA). Kolmogorov-Smirnov and Shapiro-Wilk tests were used to test normal distribution of the data. As the data (MPa) were not normally distributed (p=0.042), Kruskal Wallis, Mann-Whitney U and Wilcoxon Signed Ranks non-parametric tests were adopted. The means of DC (%) each group were analyzed by two-way analysis of variance (2-way ANOVA) where DC (%) was the dependent variable and cement types (2 levels: ML vs RXU) and root levels (3 levels: coronal, middle, apical) independent variables. Due to significant group factor ($P<0.05$), multiple comparisons were made by Tukey-Kramer adjustment test. P values less than 0.05 were considered to be statistically significant in all tests.

Results

Significant difference was observed between the groups ($P<0.05$). Regardless of the resin cement type, the mean push out bond strength results (MPa), were significantly higher for the coronal slices (ML: 9.1±2.7; RXU: 7.3±4.1) than those of the most apical ones (ML: 7±4.9; RXU: 2.89±1.5) ($P=0.002$) (Fig. 4).

   Overall, DC was significantly higher for ML (67±8.2%) than RXU (26±8.8%) ($P<0.001$) (Fig. 5). Resin cement type and ($P<0.001$) root level (p=0.002) significantly affected the DC values, while the interactions terms were not significant ($P=0.606$) (Fig. 6).

   Adhesive failures at the cement-dentin interface (AD) were more commonly experienced in RXU than in ML, whereas ML presented more incidences of adhesive failures at the cement-post interface (AP) (Fig. 7). Fewer M and C type of failures were observed.

Discussion

Based on the results of this study, DC was significantly different for the two cements tested, with ML presenting higher values of DC. Thus, the first hypothesis is rejected. Likewise, ML cement type showed significantly higher push-out bond strength results compared to RXU but as in deeper portions of the canal the results decreased with both cements, the second part of the hypothesis could be rejected.

The retention of posts in different areas of the post space can be measured by means of either using microtensile or push-out tests. Due to the small size (ca. 1 mm^2) of the bonded area of the specimens, microtensile test allows more uniform distribution of the stresses along the bonded joints. However, particularly for testing post-cement-dentin assemblies push-out test was reported to be more reliable than microtensile test as in the latter specimen preparation results in high number of pre-test failures and the wide distribution of data relative to the microtensile test [13].

Although the push-out bond strength of ML was significantly higher than that of RXU, the results in general could still be considered low compared to adhesion to sound dentin or even caries-affected dentin [14]. Adhesion of root posts to intraradicular dentin presents several clinical challenges due to heterogeneity of the substrate and exposure of root dentin to irrigation solutions or contamination with the canal filling materials [15]. In this study, in order to simulate the clinical conditions, the root canals were filled with gutta percha that was then removed prior to cementation of the post. The accessibility of the root canal during handling of the materials and the peculiar conditions of moisture in the root canal are other factors that can possibly influence the quality of adhesion. Furthermore, the poor match of circular pre-fabricated posts to the corresponding irregular post spaces may affect the non-uniform adaptation of the luting cement to root canal walls. Finally, high and therefore unfavourable C-factor for the root canal contributes maximizing the polymerization stress of luting cements [16]. The lower bond results with both resin cements could be attributed to these reasons.

Micro-raman spectroscopy is a precise and non-destructive method used for measuring the DC of resin composites where the measurement of non-reacted methacrylate groups allows calculating the percentage

of Carbon double bond that transform into single bond. The advantages of both chemical and photo-polymerized components of the luting cements tested, characterized by extended working time and capability to convert independent from the presence of light, allows reaching a more stable bond even in regions of the post space that cannot be easily reached by the blue light from the polymerization device. The employment of translucent posts help to improve the light transmission from the polymerization device through the post and increase stiffness of the photo-polymerized part of the resin cements in the root canal [1,17]. The results of this study support this statement at least for the tested FRC post in that DC did not significantly change from coronal to apical parts of the root. Yet, the push-out bond strengths in the most apical parts of the root were significantly lower with both cements. One reason for this could be due to insufficient cleaning of the intraradicular dentin walls in the apical part impairing the adhesion even though all steps of the experiment was performed by one experienced operator.

The most common failure type was adhesive detachment of the cement from the dentin surface in RXU and less in ML. Both cements were self-etch or self-adhesive cements that did not necessitate acid etching of the root. However, this type of failure indicates that the adhesion was not sufficient to intraradicular dentin with both cements. In only a few cases, in ML (2) and RXU (1), cohesive failure in dentin was observed.

The concentration of the acidic monomers in such self-adhesive cements is low enough to avoid excessive hydrophilicity but high enough to etch dentin or enamel [18]. These types of cements become more hydrophobic as the acid functionality is consumed through reaction with calcium in tooth. During endodontic treatment, mineralized tissues are disintegrated during instrumentation, producing considerable quantities of mineral debris and generating a thick smear layer in intraradicular dentin being denser than that observed in coronal dentin [19]. The presence of such a layer impairs a proper contact between the acidic methacrylates of self-adhesive cements during adhesive procedures, interfering with its bond strength to dentin. The negligible incidence of cohesive failures in dentin apparently indicates that etching capacity of ML (pH=4.2) and RXU (pH=2.1) with acidic monomers was not sufficient.

Even though, the FRC post surfaces were not conditioned, AP type of failures occurred with the ML cement but not with the RXU. Expansion of the RXU [20], which is about 0.63% in one month according to

the manufacturer, could fill the root canal space and compensating for the lack of adhesion to the post surface and the inferior DC. In fact in this study, aging was not performed. Thus, it cannot be stated how much expansion has taken place within 24 h of water storage, which needs to be further investigated.

It has to be noted that FRC post surfaces have not been conditioned physico-chemically in this study that may increase the adhesion of resin cements on the FRC post [21,22]. Also, no aging was performed to simulate the possible early failures. Thus, the results of this study should additionally be verified in aged conditions and in particular under fatigue conditions in water accompanied with temperature alterations [22]. Standard endodontic treatment includes the use of a root canal sealant, which was not used in this study since gutta percha already contaminates the root canal during cleaning the canal. This could be considered a limitation of this study and should be incorporated in the protocol in similar studies in the future. Endodontic problems associated with RXU due to low DC of this cement should be monitored in clinical studies.

Conclusions

From this study, the following could be concluded:

1.   Overall, push-out bond strength of dentin-cement-fiber post assembly with methacrylate based resin cements containing phosphonic acid (ML) was significantly higher than that having phosphoric acid acrylate (RXU) in its composition.

2.   With both cements tested, push-out bond strength of the fiber posts decreased significantly 5 mm below the cement-enamel junction.

3.   Degree of conversion was almost 3-fold higher with ML compared to RXU and it did not show significant difference at 3 and 5 mm away from the cement-enamel junction.

4.   Adhesive failures at the cement-dentin interface were more commonly experienced in RXU than in ML, whereas ML presented more incidences of adhesive failures at the cement-post interface.

Clinical Relevance

Considering the push-out bond strength and degree of conversion, methacrylate-based cements with phosphonic acid should be preferred to those having phosphoric acid when cementing glass fiber posts in

the root canal, providing that with both cements the weakest link was between the dentin-cement interface at 3 and 5 mm below cemento-enamel junction.

Conflict of interest

The authors did not have any commercial interest in any of the materials used in this study.

References

1. Roberts HW, Leonard DL, Vandewalle KS, Cohen ME, Charlton DG. The effect of a translucent post on resin composite depth of cure. Dent Mater 2004;20:617-622.

2. Asmussen E, Peutzfeldt A, Heitmann T. Stiffness, elastic limit, and strength of newer types of endodontic posts. J Dent 1999;27:275-278.

3. Mannocci F, Sherriff M,Watson TF. Three-point bending test of fiber posts. J Endodont 2001;27:758-761.

4. Ferrari M, Vichi A, Grandini S, Goracci C. Efficacy of different adhesive techniques on bonding to root canal walls: an SEM investigation. Dent Mater 2001;17:422-429.

5. Freedman GA. Esthetic post-and-core treatment. Dent Clin North Am 2001;45:103-116.

6. Boschian Pest L, Cavalli G, Bertani P, Gagliani M. Adhesive postendodontic restorations with fiber posts: push-out tests and SEM observations. Dent Mater 2002;18:596-602.

7. Sahmali S, Demirel F, Saygili G. Comparison of in vitro tensile bond strengths of luting cements to metallic and tooth-colored posts. Int J Periodont Restor Dent 2004;24:256-263.

8. Ferrari M, Vichi A, Grandini S, Goracci C. Efficacy of a self-curing adhesive-resin cement system on luting glass-fiber posts into root canals: an SEM investigation. Int J Prosthodont 2001;14:543-549.

9. Malferrari S, Monaco C, Scotti R. Clinical evaluation of teeth restored with quartz fiber-reinforced epoxy resin posts. Int J Prosthodont 2003;16:39-44.

10. Monticelli F, Grandini S, Goracci C, Ferrari M. Clinical behavior of translucent-fiber posts: a 2-year prospective study. Int J Prosthodont 2003;16:593-596.

11. Radovic I, Corciolani G, Magni E, Krstanovic G, Pavlovic V, Vulicevic ZR, Ferrari M. Light transmission through fiber post: the effect on adhesion, elastic modulus and hardness of dual-cure resin cement. Dent Mater 2009;25:837-844.

12. Galhano G, de Melo RM, Valandro LF, Bottino MA. Comparison of resin push-out strength to root dentin of bovine- and human-teeth. Indian J Dent Res 2009;20:332-326.

13. Goracci C, Tavares AU, Fabianelli A, Monticelli F, Raffaelli O, Cardoso PC, et al. The adhesion between fiber posts and root canal walls: comparison between microtensile and push-out bond strength measurements. Eur J Oral Sci 2004;112:353-361.

14. Cardoso PE, Sadek FT, Goracci C, Ferrari M. Adhesion testing with the microtensile method: effects of dental substrate and adhesive system on bond strength measurement. J Adhes Dent 2002;4:291-297.

15. Bouillaguet S, Troesch S, Wataha JC, Krejci I, Meyer JM, Pashley DH. Microtensile bond strength between adhesive cements and root canal dentin. Dent Mater 2003;19:199-205.

16. Aksornmuang J, Nakajima M, Senawongse P, Tagami J. Effects of C-factor and resin volume on the bonding to root canal with and without fibre post insertion. J Dent 2011;39:422-429.

17. Özcan M, Zamboni S, Valandro F, Bottino M, Bagis B. Microhardness of dual-polymerized resin cement around a translucent fiber post in the intraradicular environment. J Conserv Dent 2011;14:370-373.

18. Ferracane JL, Stansbury JW, Burke FJ. Self-adhesive resin cements - chemistry, properties and clinical considerations. J Oral Rehabil 2011;38:295-314.

19. Brunzel S, Yang B, Wolfart S, Kern M. Tensile bond strength of a so-called self-adhesive luting resin cement to dentin. J Adhes Dent 2010;12:143-150.

20. Nakamura T1, Wakabayashi K, Kinuta S, Nishida H, Miyamae M, Yatani H. Mechanical properties of new self-adhesive resin-based cement. J Prosthodont Res 2010;54:59-64.

21. Sahafi A, Peutzfeldt A, Asmussen E, Gotfredsen K. Effect of surface treatment of fabricated posts on bonding of resin cement. Oper Dent 2004;29:60-68.

22. Valandro LF, Özcan M, de Melo RM, Galhano GA, Baldissara P, Scotti R, Bottino MA. Effect of silica coating on flexural strength of fiber posts. Int J Prosthodont 2006;19:74-76.

Legends

Tables:

Table 1. Brands, manufacturers, batch numbers, chemical compositions, application system and modes of the resin cements used in this study.

Figures:

Fig. 1 Schematic drawing of the root slices obtained below the CEJ apically from each tooth which were then exposed to degree of conversion and push out tests.

Figs. 2a-c Root slices obtained from **a)** coronal (1 mm below CEJ), **b)** middle (3 mm below CEJ) and **c)** apical (5 mm below CEJ). Note the variations in root diameter and morphology from coronal to apical.

Figs. 3a-b a) Schematic drawing of the internal section of the root wall, **b)** Calculation of the generatrix of the cone frustum.

Fig. 4 Push out bond strength of the fiber posts with the two cements tested at 1 mm, 3 mm and 5 mm below the CEJ. ML: Multilink Automix; RXU: RelyX Unicem 2 Automix.

Fig. 5 Overall mean push-out bond strength results and degree of conversion of the two cements tested. ML: Multilink Automix; RXU: RelyX Unicem 2 Automix.

Fig. 6 Degree of conversion of the two cements tested at 1 mm, 3 mm and 5 mm below the CEJ. ML: Multilink Automix; RXU: RelyX Unicem 2 Automix.

Fig. 7 Distribution of failure types for the post-cement-dentin interfaces for the two cements tested at 1 mm, 3 mm and 5 mm below the CEJ. AD: Adhesively (A) debonded from dentin (D), complete detachment of cement from the dentin, AP: Adhesively (A) debonded from post (P), complete detachment of the cement from the post; C: Cohesive (C) failure in the dentin; M: Mixed (M) failure, a combination of adhesive and cohesive failures.
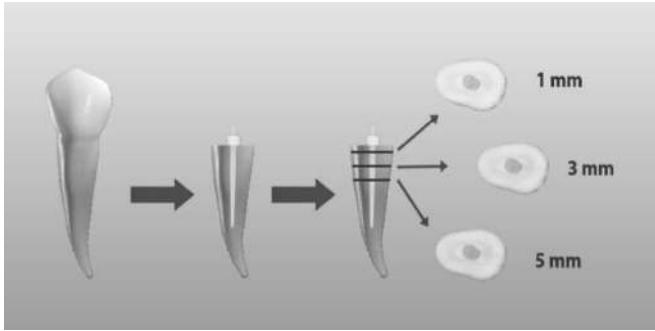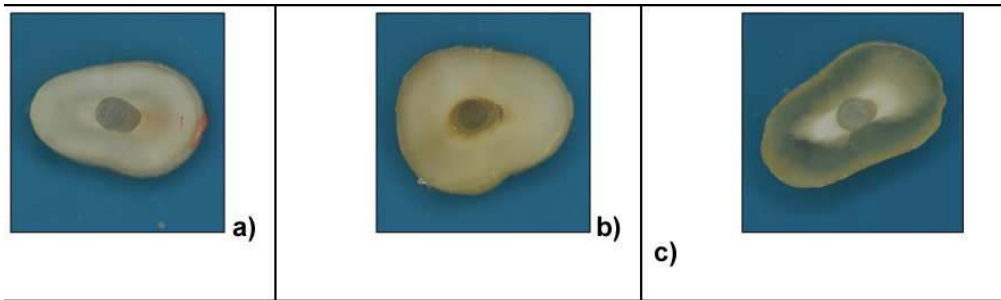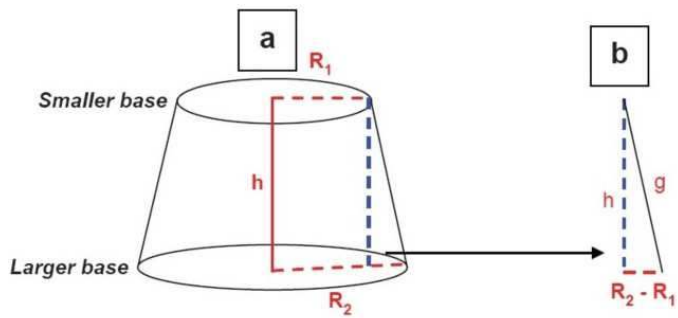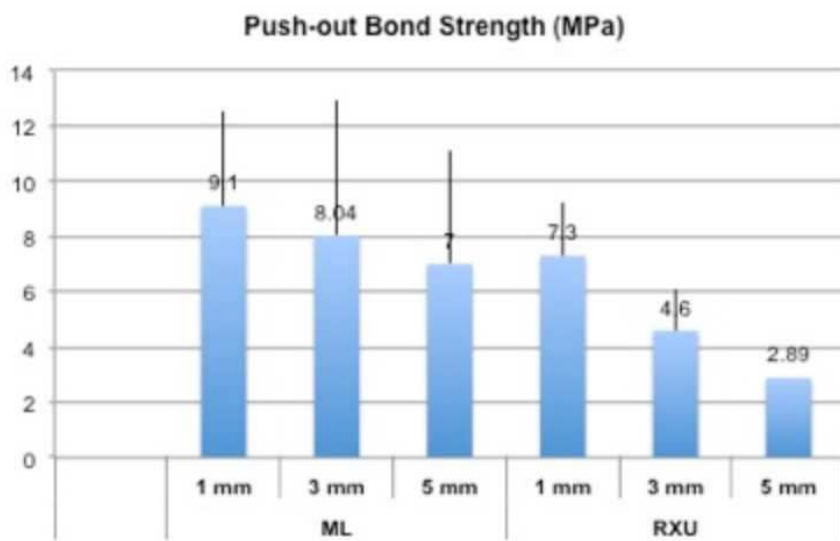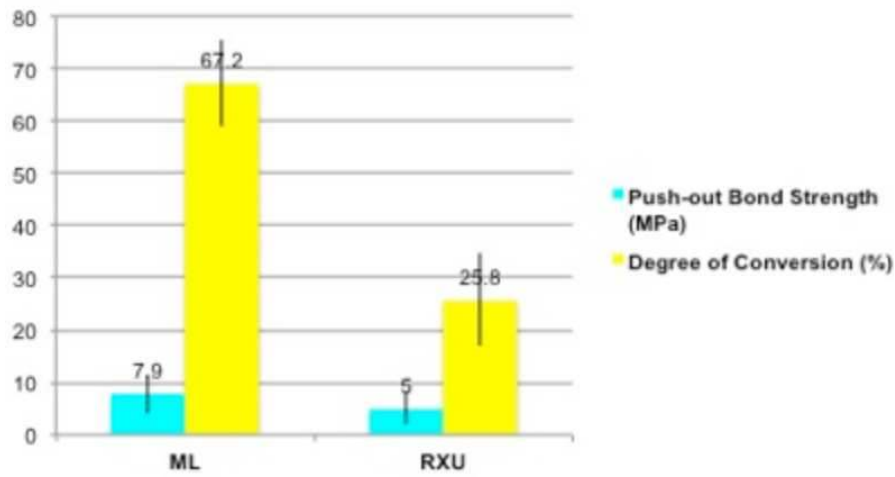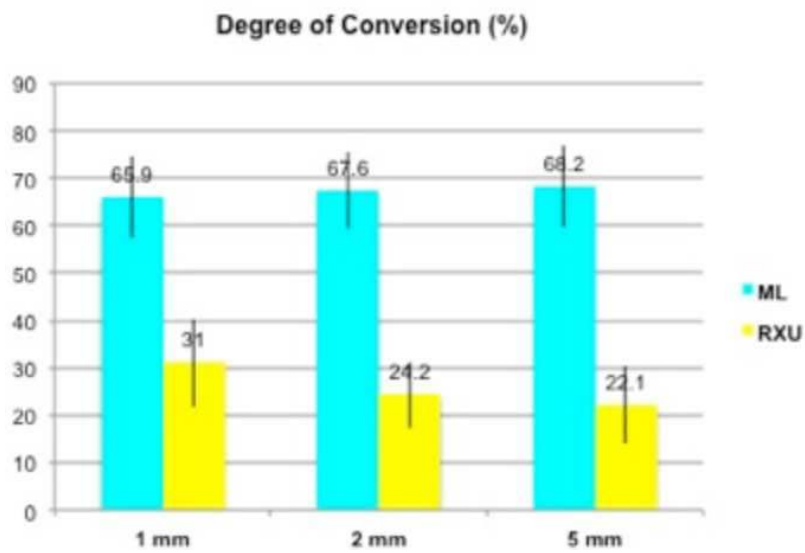
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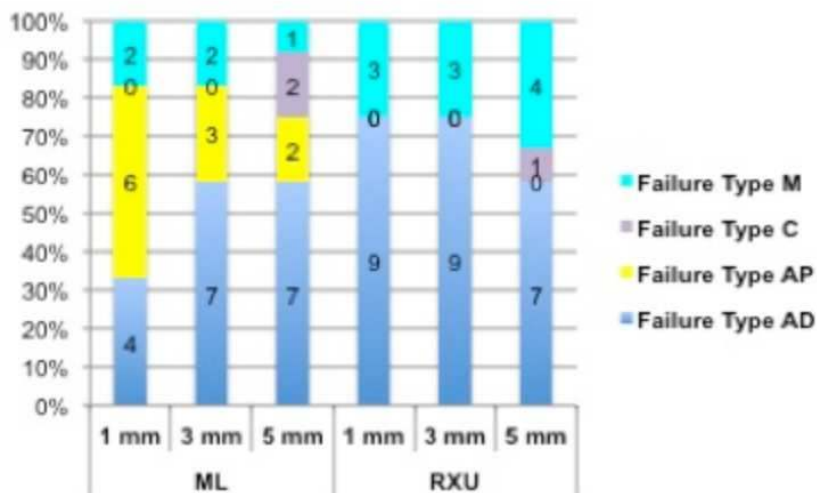

Figure 7

Tables:

Brand (Manufacturer)	Batch Number	Chemical Composition	Application System	Application Mode
Multilink Automix (Ivoclar Vivadent, Schaan, Liechtenstein)	N74236	Dimethacrylate containing phosphonic acid groups, Hydroxyethylmeth acrylate, inorganic fillers, initiators. pH = 4.2	Base paste/Catalyst paste, automixing system.	Mix the two self-curing Primer liquids A and B in a 1:1 mixing ratio, Apply the mixed cement, cure for 20 s.
RelyX Unicem 2 Automix (3M ESPE, Seefeld, Germany)	427041	Methacrylate monomer containing phosphoric acid groups, silanated and alkaline fillers, initiators, stabilizers, pigments and rheological additives. pH = 2.1	Base paste/Catalyst paste, automixing system, endo tip.	Apply the mixed cement, cure for 40 s. The cement begins to self-cure after 150 s and stops in 360 s.

Table 1. Brands, manufacturers, batch numbers, chemical compositions, application system and modes of the resin cements used in this study.