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Tensile Bond Strength and Flexural Modulus of Resin Cements—Influence on the Fracture Resistance of Teeth Restored with Ceramic Inlays

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Clinical Relevance

The mechanical properties of resin cement can influence the fracture resistance of teeth restored with ceramic inlays. In general, cement with a higher elastic modulus resulted in a tooth/ceramic inlay that had a higher fracture resistance.

SUMMARY

Purpose: This *in vitro* study tested tensile bond strength to enamel and dentin and the flexural modulus of three resin cements. It also determined the influence of these properties on the fracture resistance of teeth restored with ceramic inlays. Materials and Methods: Initially, 10 standard ceramic discs were bonded to enamel using the following resin cements: Enforce (E), RelyX ARC (RX) and Fill Magic Dual Cement (FM). After seven days of storage, the specimens were subjected to tensile forces at a crosshead speed of 0.5 mm/minute until fracture. The enamel was then ground and the ceramic disks were bonded to dentin. The flexural modulus of each type of resin cement was calculated based on the straight-line tension-deformation curve using the three-point flexure method. For resistance to fracture, 40 sound maxillary premolars were randomly divided into four groups (n=10). Three groups were submitted to preparations and restored with ceramic inlays bonded with the same resin cements used during the tensile test (n=10). Intact teeth were used as the control group. The specimens were subjected to compressive axial loading at 0.5 mm/minute using a 10-mm steel ball until fracture. Results: Statistical analysis revealed that, for all cements, the bond strength to enamel was significantly higher (p<0.05) than that obtained in dentin. In both substrates, RX and FM showed higher bond strengths than that obtained for E (p<0.05). In relation to flexural modulus, FM had the lowest and E the highest flexural modulus; whereas, RX

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differed from the other two ($p<0.05$). The teeth with inlays that were bonded using RX, and E had a significantly higher ($p<0.05$) fracture resistance than those where the inlays were bonded with FM but without recovering the resistance observed for the control group (intact teeth). Conclusion: The three resin cements had different mechanical properties. A higher flexural modulus usually resulted in improved resistance to fracture for the ceramically restored teeth.

INTRODUCTION

Removal of tooth structure has been considered the main reason for decreased fracture resistance of prepared teeth.[1-2] Such weakening of the tooth structure is due to removal of occlusal enamel, destruction of marginal ridges and a reduction in cusp volume, all of which lead to increased deflection and higher stress concentration at the cusp bases.[3]

With the introduction of adhesive cements, the need for dental structure removal has been reduced, allowing for the preservation of more sound tooth tissue.[3-5] Moreover, adhesive cementation has significantly increased the indications for ceramic restorations.[6-7] However, ceramics are friable materials, and restoration fracture has been considered the main reason for failure.[8]

The use of resin cement with feldspathic ceramic has been considered a paramount condition for the success of a restoration. Some reasons that could contribute to this success include transmission of forces from the restoration,[9-11] an increase in the elastic modulus of the supporting core,[12] structure modification of the internal surface of the restoration with resin filling the cracks[13-14] and increased crack radius caused by acid etching, thereby avoiding crack propagation.[14]

The resin-luting agent should provide sufficient bond strength and mechanical support to prevent fracture of ceramic restorations. However, adhesive ability and elastic modulus can be influenced by variation in the composition of cements.[12,15] Increasing the elastic modulus of the supporting core structure has been suggested as a means of increasing the fracture resistance of all-ceramic crowns.[16-17]

Several studies have investigated resin cements; however, there is a lack of information in the literature regarding the relationship among bonding capacity, flexural modulus and the resistance to fracture of restored tooth. This study evaluated the tensile bond strength to enamel and dentin and the flexural modulus of resin cements and determined the influence these properties have on the fracture resistance of teeth restored with ceramic inlays.

METHODS AND MATERIALS

The research protocol was approved by the Ethics Committee (Federal University of Pelotas). Tensile bond strength, flexural modulus and fracture resistance were tested.

Tensile Bond Test

Thirty lower human third molars were selected. The teeth were examined under magnification and only those free of cracks were used. Soft tissue was removed with dental curettes and the teeth were disinfected for 72 hours in 1% chloramine.[18] The teeth were washed in tap water (24 hours) and stored frozen until tested. The roots were sectioned and embedded in a PVC matrix using acrylic resin (Artigos Odontológicos Clássico Ltda, São Paulo, Brazil), leaving the buccal surface lightly uncovered. The exposed enamel surface was polished with 220-, 400- and 600-grit sandpaper under water-cooling.

Thirty ceramic discs of Vitadur Alpha (Vita Zahnfabrik, Bad Säckingen, Germany) were produced using a standard metallic matrix (5-mm in diameter and 4-mm in height), with three firings (600°C–960°C). The discs were embedded in metallic cylinders with acrylic resin, with the bottom surface of the ceramic disc exposed to allow for cementation. The discs were polished with 220-, 400- and 600-grit sandpaper under water-cooling to standardize the surface.

After prophylaxis with pumice, the bonding area was delimited using adhesive tape with a circular hole 3.5 mm in diameter, providing an adhesive interface of 9.6 mm^2. The ceramic discs were cemented to the buccal surface of the teeth with three different resin cements (n=10) (Table 1) that were applied according to the manufacturers' instructions.

The enamel surfaces were conditioned with 37% phosphoric acid for 15 seconds, washed and lightly dried. Two coats of the respective adhesive system of each resin cement were applied and photo-cured (10 seconds) with an XL 3000 light-curing unit (3M ESPE, St Paul, MN, USA), with energy higher than 450 mW/cm^2. The ceramic surfaces were conditioned with 10% hydrofluoric acid for four minutes, they were then washed/dried and two coats of silane were applied.

Equal portions of the base and catalyst pastes were mixed for each of the three resin cements tested. A thin layer was applied over the conditioned enamel surface, and the ceramic was positioned over the enamel surface with a 1-kgf load using an adapted Vicat needle to produce standard pressure. The excess cement was removed with a disposable brush and the cement was polymerized in three different positions (120°, 240° and 360°).

The specimens were stored in distilled water at 37° ± 1°C and protected from light for seven days. The speci-

Table 1: *Cement Systems Used in the Cementation: (s) Silane, (a) Adhesive and (c) Resin Cement*

	Material (color)	Composition	Batch #	Manufacturer
G1	s – Silane	Silane, Ethanol and Acetic Acid	178	Dentsply, Petrópolis, RJ, Brazil
	a – Prime & Bond 2.1	Elastomeric resins, PENTA, Acetone, Cetylamine Hydrofluoride	055	
	c – Enforce (A2)	BisGMA , BHT, EDAB, TEGDMA, Fumed Silica, Silanized Barium, Aluminum Borosilicate Glass (66% wt)	731	
G2	s – Ceramic Primer	Silane, Alcohol and Water	7KH	3M ESPE, St Paul, MN, USA
	a – Single Bond	Water, Alcohol, HEMA, BisGMA, Dimethacrylate, Photoinitiator, Copolymers of the Polyacrylic Acid and Polyitaconic Acid	3HL	
	c – RelyX ARC (A3)	BisGMA, TEGDMA, Silica and Zirconium Glass (67,5% wt)	BHBH	
G3	s – Silane	Silane, Ethanol and Acetic Acid	00502	Vigodent, Rio de Janeiro, RJ, Brazil
	a – Magic Bond DE	HEMA, Dimethacrylates, Phosphinic Acid Acrylate, Silicon Dioxide, Alcoholic Solution, initiators and stabilizers	03902	
	C – Fill Magic Dual Cement (A2)	Methacrylic monomer, Silica, Fluor	00802	

mens were then submitted to the tensile bond test using a universal testing machine MEM 2000 (EMIC, São José dos Pinhais, Brazil) with a crosshead speed of 0.5 mm/minute and a 50-kgf load cell. A device was developed (Figure 1) to allow for better alignment of the testing assembly. Force measurements were converted into strength (MPa) according to the bonded surface area.

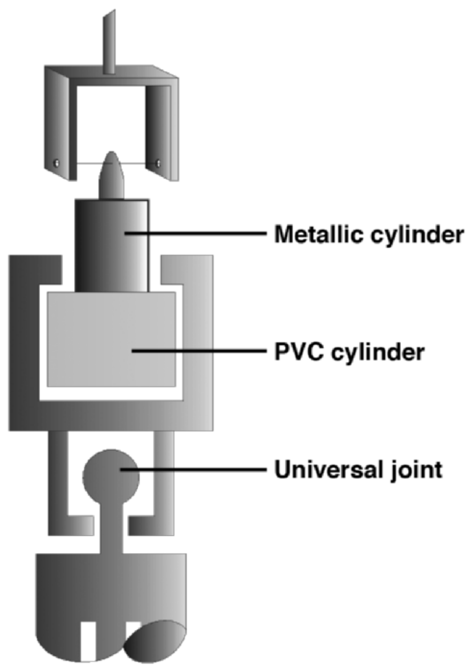

Figure 1: *Special device adapted to the testing machine for the tensile test.*

The teeth and ceramic discs were then repolished under water-cooling, with the objective of exposing the dentin and ceramic without conditioning. The same cementation procedures were repeated to determine the tensile bond strength to dentin.

Flexural Modulus Test

The flexural modulus of each resin cement was calculated using the Tesc 3.01 software of the Universal test machine (EMIC) based on the straight-line tension-deformation curve, which uses the three-point flexure method. Five specimens (rectangular beams 2 mm x 2 mm in cross section and 25-mm long) of each material were prepared according to ISO/FDIS 4049:2000, stored in distilled water and protected from light at 37°C ± 1°C for seven days. The specimens were then loaded at a crosshead speed of 0.05 cm/second.

Fracture Resistance Test

Forty sound human premolars extracted for orthodontic reasons were selected. To be included in the study, the premolars had to have the crown dimensions proposed by Galan Junior: 9.0–9.6 mm of the bucco-lingual distance; 7.0–7.4 mm of the mesio-distal distance and 7.7–8.8 mm of the cervico-occlusal distance.[19] Also, the teeth had to be free of cracks under 10x magnification. The selected teeth were cleaned, disinfected and stored as described.

The teeth had their roots included in a PVC matrix, using acrylic resin (Artigos Odontológicos Clássico Ltda, São Paulo, Brazil) up to 1 mm below the cement-enamel junction. Impressions of each tooth were taken with a polyvinylsiloxane material (Express, 3M ESPE, St Paul, MN, USA) that was used as an anatomic guide

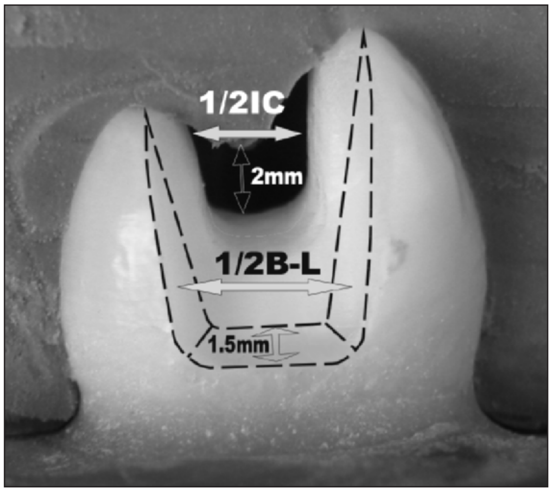

Figure 2: *MOD ceramic inlay preparation sizes (IC—intercuspid distance; B-L—bucco-lingual distance).*

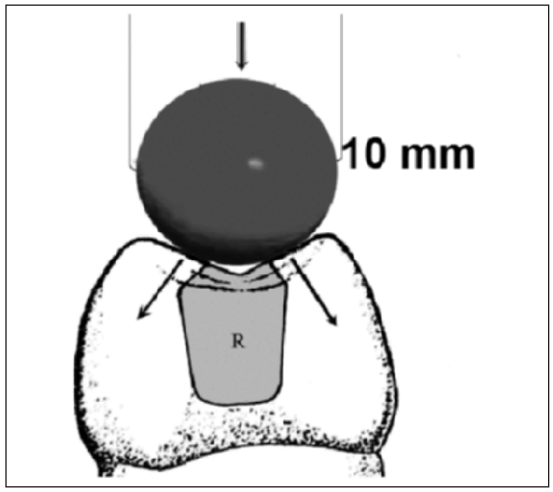

Figure 3: *The representation of the load application of the sphere used for the axial compression test.*

during tooth reduction. The teeth were randomly divided into four groups (n=10).

Standard inlay MOD preparations were performed in the three study groups, with one group remaining intact to serve as a control. Diamond burs #4137 (KG Sorensen, Barueri, Brazil) were used for cavity preparation at high speed under copious air-water cooling. The burs were replaced after every fourth tooth preparation to ensure high cutting efficiency. To finish the preparations and to standardize the convergence angle of the cavity walls, the same bur was used at a low speed adapted in a special device. The occlusal box was 2-mm deep and had half of the intercuspidal distance. The proximal boxes were prepared at a width half of the bucco-lingual distance, 1.5 mm at the cervical wall in a proximal-proximal direction, with the cervical wall 1 mm above the cementoenamel junction (Figure 2).[20] The total amount of dental structure reduction was controlled using the polyvinylsiloxane impression that was made prior to the cavity preparations also using a digital caliper. The restorations were made with Vitadur Alpha ceramic, which was submitted to four firings (600°C-930°C). The ceramic restorations were finished with silicone points and glazed following the manufacturers' instructions. The internal surfaces were cleaned with glass-particle blasting.

The cementation procedures were similar to those described for the previous tensile bond tests. Finishing and polishing procedures were completed with the Sof-Lex system (3M ESPE). The specimens were stored in distilled water and protected from light at 37°C for seven days.

All the specimens were subjected to the axial compression test in a universal testing machine (EMIC) using a steel sphere (10 mm in diameter) (Figure 3). The sphere made contact on the enamel cups, with a 500 Kgf load at a crosshead speed of 0.5 mm/minute until the specimen fractured. The fracture pattern was evaluated based on a standard ranking developed by Habekost and others:[21]

a) Pattern I–Fracture restricted to the restoration;
b) Pattern II—Fracture of the dental structure, but not through the long axis of the tooth;
c) Pattern III–Fracture of the tooth and restoration, but not through the long axis of the tooth and
d) Pattern IV–Fracture through the long axis of the tooth, being in the tooth/restoration or only at the tooth.[21]

During this study, only one experienced operator performed the steps.

Statistical Analysis

The data obtained with the tensile bond strength, flexural modulus and axial compression tests were submitted to parametric tests (ANOVA and Tukey's test). Evaluation of the fracture patterns was performed using the non-parametric Kruskal-Wallis test.

RESULTS

The means and standard deviations for tensile bond strengths are listed in Table 2. Data underwent log transformation to obtain normal and homogeneous distribution to allow for parametric analysis. The two-way analysis of variance test identified a significant difference among the tested groups ($p<0.05$). Regardless of the luting agent used, the tensile bond strengths in enamel were higher than those found in dentin ($p<0.05$). The Tukey interval test revealed that RX and FM had higher tensile bond strengths than E in both substrates ($p<0.05$).

Means and standard deviations for flexural moduli are listed in Table 3. Analysis of variance showed sig-

Table 2: *Means and Standard Deviations of Tensile Bond Strengths (MPa) of Resin Cements*		
	Means (SD)	
Cements	**Enamel**	**Dentin**
Enforce	10.4 (1.9)Aa	6.9 (1.5)Ab
RelyX ARC	14.8 (3.6)Ba	10.9 (2.6)Bb
Fill Magic Dual Cement	16.2 (4.8)Ba	11.9 (2.8)Bb

The same upper case letters indicate no significant differences (p>0.05) among means in columns.
The same lower case letters indicate no significant differences (p>0.05) between means in rows.

Table 3: *Means and Standard Deviations of Flexural Modulus (GPa) of Resin Cements*

Cements	**Enamel**
Enforce	7.29 (0.02)A
RelyX ARC	5.70 (0.07)B
Fill Magic Dual Cement	2.59 (0.11)C

All the means were significantly different (p<0.05).

Table 4: *Means and Standard Deviations of Fracture Resistance (Kgf)*

Cements	**Means (SD)**
Control	188 (57)A
Enforce	110 (13)B
RelyX ARC	111 (21)B
Fill Magic Dual Cement	91 (14)C

The same upper case letters indicate no significant differences (p>0.05) among means.

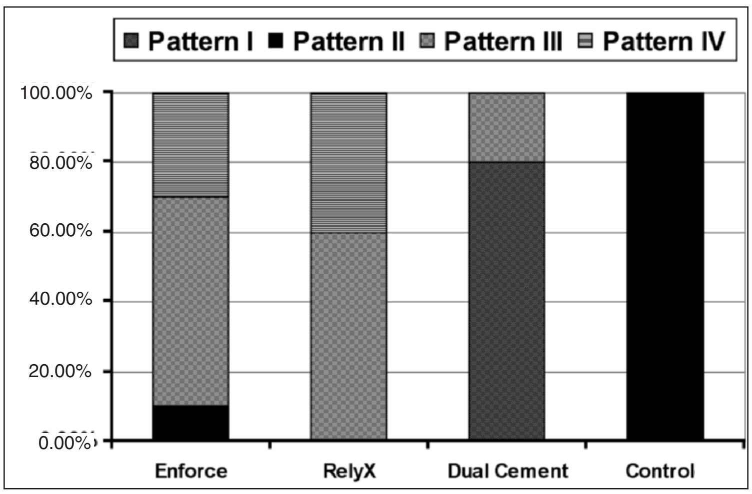

Figure 4: *Frequency rate of the failure pattern observed for each group.*

nificant differences between values found for different cements ($p<0.05$). The Tukey interval revealed that the flexural moduli of all cements were different ($p<0.05$). Cement E had the highest and FM had the lowest flexural modulus. Intermediate values were found for RX and were different from the other groups.

The means and standard deviations for fracture resistance are reported in Table 4. Analysis of variance showed a significant difference between values found for the different cements ($p<0.05$). The Tukey interval revealed that no experimental group had resistance to fracture that was similar to sound teeth. Inlays cemented with E and RX had similar results and both produced higher resistance to fracture than FM.

Analysis of the Failure Patterns

After the compression test, the specimens were analyzed to determine the failure pattern (Figure 4). The groups cemented with E and RX had a higher frequency of Pattern III failure, and both were statistically different from the FM group, which exhibited a higher frequency of Pattern I failure.

DISCUSSION

This study was designed to evaluate the bond strength and elastic modulus of resin cements and their influence on the fracture resistance of conjunct tooth/ceramic restorations. This research protocol was based on the fact that, in a clinical situation, resistance will be provided by the tooth-cement-restoration complex.[22]

The resin cements had different bond strengths. RX and FM had higher bond strengths than E. These results partially agree with those reported by Braga and others, who compared RX and E in dentin.[10] Nonetheless, Mota and others[15] demonstrated that E and RX had similar tensile bond strengths to the same substrate. Such disagreement could be related to the different laboratory methodologies used.

The differences in tensile bond strengths may be attributed to the different adhesive systems associated with the resin cements employed. While RX and FM were applied with an ethanol/water-based adhesive (Single Bond) or an ethanol-based adhesive (Magic Bond DE), respectively, cement E was applied after an acetone-based adhesive system (Prime & Bond 2.1) was used.

The monomer/solvent ratio can significantly influence adhesive viscosity, and an adequate amount of monomers should be present to guarantee surface infiltration.[23] Eliades and others, evaluating the separation of monomers in one-bottle systems applied to the dental surface, reported that Prime & Bond 2.1 was the adhesive system that presented the highest separation rate.[24] While solvent in Single Bond adhesive is responsible for 30% of the adhesive volume, in Prime & Bond 2.1, the amount of solvent increases to 80%.[25]

The lower monomer volume present in Prime & Bond 2.1 could generate deficient infiltration of the etched surface, causing areas of stress concentration that could cause disruption of the adhesive interface.[26-27] Perhaps the application of additional adhesive layers might compensate for this difference in monomer/solvent ratio; however, in this study, the number of layers applied was standardized.

Again, in this study, resin cements performed better in enamel than dentin, corroborating these findings with others studies.[15,28-29] Such a finding can be due to the mechanism of adhesion on enamel being quite different from the mechanism to dentin. Bonding to enamel depends on the micromechanical retention of resin tags to the etched enamel surface. The mechanism of bonding to dentin is based on a monomer mixture, with hydrophilic groups for the wetting and penetrating into exposed dentin collagen fibrils.[25] The results of this study are partially questioned by the findings of Reis and others,[25] where Single Bond and Prime & Bond 2.1 performed similarly on enamel and dentin.

One important concern of the tensile test is for it to maintain alignment of the specimen during decoding procedures.[30] When correct alignment is obtained, failure will predominantly be of an adhesive nature.[31] To produce this correct alignment, a special device adapted to the universal testing machine with a universal joint was used in this study.

Compared to the tensile test, the microtensile bond strength test induces a better stress distribution at the adhesive interface, with lower failure inclusion and increased bonding values. However, when both tests are used, there is no difference in materials ranking, thus allowing for a comparison of the results.[32] In this study, the tensile test was used, because it is difficult to control the incorporation of failures into the ceramic mass produced by the vibration and condensation technique, which could produce premature failure of the specimens during the cutting procedures.

The flexural modulus is a measure of the stiffness or resistance to deflection of a given material. A material with a higher flexural modulus can resist deformation from a given load better than one with a lower elastic modulus.[12] All the cements presented differences among themselves. Enforce presented the greatest rigidity and Dual Cement presented the most deflection.

In terms of the compressive test, the teeth restored with ceramic inlays luted with E and RX presented better results than those inlays cemented with FM. These findings demonstrated that higher bond strength will not generate higher fracture resistance to complex tooth/restorations. As demonstrated in this study, the influence of bond strength on the fracture strength of ceramic restorations appears to be minimal; however, this factor cannot be neglected due to its potential influence on the retention, marginal infiltration and adaptation of the restoration.

From the results of this study, the elastic modulus of resin cements could suggest having more of a pronounced influence on the fracture strength of ceramically restored teeth than the adhesiveness of resin cements. The forces applied to the enamel surface generate stress that is transferred to dentin, where they are absorbed. In ceramic inlay restored specimens, the stress could be transferred to the cement.[4,8] The cement elasticity modulus is directly linked to its capacity to transfer the tensions of the restoration to the dental structure and indicate its ability to resist elastic deformation; the elastic modulus of the supporting structure may be a significant factor in determining the fracture resistance of all-ceramic restorations.[17,33-34]

Wakabayashi and Anusavice observed higher ceramic resistance when forces were applied over higher elasticity module bases.[11] According to Li and White, cements with an intermediate elasticity modulus between that of dentin and the restorative material should be chosen to reduce stress concentration at the interface.[35] Banditmahakun and others reported the effect of the elastic modulus of supporting structure on the fracture load of ceramic inlays. These authors showed that fracture load increased when the elastic modulus of cement agents increased.[12]

Although RX presented a smaller flexural modulus than E, the fracture resistance of the ceramic restorations cemented with both was similar.

Despite differences among the experimental groups, the restored teeth did not have any fracture resistance similar to that obtained with sound teeth. Other studies, however, have demonstrated that feldspathic ceramic inlays did not have fracture resistance comparable to intact teeth.[21,36]

Analysis of the failure patterns showed that, involving tooth and restoration, specimens with higher fracture resistance produced the most catastrophic failures, which was similar to previous reports.[12,21,36-37] In all likelihood, the failure pattern observed in inlays luted with FM (restoration fracture) could be due to the lower flexural modulus of this cement, which allowed for material deflection, leading to premature failure. With rigid support, flexure of the restoration would be reduced. The use of base materials with a sufficiently high elastic modulus can provide sufficient support to reduce stress on the internal surface of ceramic inlays.[12]

In the mouth, failures can occur from fatigue, which generates cumulative microcracks until failure of the restoration.[38] Although the fracture resistance test did not adequately reproduce all conditions of the oral cavity, it could indicate the weaker components of the group tooth/restoration.[20] The size of extracted teeth, cavity dimensions and convergence angle were standardized to reduce operator errors.

The results of both tests could be influenced by cement thickness and polymerization time.[5-6,8] To avoid these influences, standard loading was applied during cementation, and the tests were performed after seven days, which is considered to be the reference time for chemical cements to achieve their highest resistance.[5]

Although laboratory tests are an important parameter of analysis, they do not replace the need for clinical evaluation. Long-term clinical trials should be considered to determine clinical performance of the materials.[7]

CONCLUSIONS

Within the limitations of this study, it was possible to conclude that:

a) RX and FM had higher tensile bond strengths to enamel and dentin than E;

b) Tensile bond strength to enamel was higher than to dentin;

c) Cement E had the highest elastic modulus, followed by RX and FM;

d) Teeth with adhesively-luted ceramic inlays could not provide fracture resistance similar to the values obtained with intact teeth, but when the restorations were cemented with E and RX, a higher fracture resistance was obtained compared to teeth where the inlays were cemented with FM;

e) Generally, cements with a higher flexural modulus had better resistance to fracture for the ceramic/tooth conjunct.

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