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Comparative Analysis for Selection of Resin Luting Cements Based on Filler Content: An *in vitro* Study

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ABSTRACT

Context: Resin luting of all-ceramic restorations results in increased performance; however, the strengthening mechanism and the role of the mechanical properties of the resin are not fully understood.

Objective: The aim of this study was to investigate the effect of filler content on the flexural properties of resin luting agents and thereby selecting an appropriate resin luting cement.

Materials and methods: Three esthetic resin luting agents studied were Calibra (Dentsply); RelyX ARC and Panavia F. Ten beam-shaped specimen (L × W × H: 30 × 8 × 2 mm) were made for each of the material tests carried out. The specimens were stored in distilled water for 24 hours at 37ºC. The specimens were then tested for flexural strength (MPa) and flexural modulus (GPa) using the three-point bending method on a universal material testing machine at a cross head speed of 0.5 mm/min. Data obtained were statistically analyzed using ANOVA followed by post-hoc - Tukey's test with $p < 0.05$ for statistical significance.

Results: Increase in mean strength related to an associated increase in the elastic modulus which in turn was related to the filler loading of the resin luting cements.

Conclusion: Strength and performance of resin-cemented all-ceramic restoration can be enhanced by the use of a resin luting cement having increased filler content.

Clinical implications: Resin-composite cements may be most suitable for adhesively bonded restorations, where margins are placed on supragingival enamel, and where ultimate strength and energy absorption are paramount. The selection criteria for a resin cement depends on its flexural strength. To a great extent, the flexural strength is dependent on the filler loading of the resin luting cement.

Keywords: Elastic modulus, Biaxial flexure strength, Filler content.

Key message: Fracture of brittle all-ceramic restoration can be prevented by using resin luting cement with higher filler loading. In other words, any resin luting cement with higher filler content has an improved performance in terms of clinical function and durability.

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INTRODUCTION

Luting cements must withstand masticatory and parafunctional stresses for many years in a warm and wet oral environment. They must maintain their integrity while transferring stresses from crown/fixed partial dentures (FPDs) to tooth structure.[1]

The mechanical properties of resin cements have been dictated by favorable working characteristics and adhesion between the restoration and the tooth substrate. The role of the resin in transferring stress from the loaded restoration to the underlying tooth structure has not been studied in detail, although it has been proposed that luting cements require an intermediary elastic modulus (γ) between dentin and the ceramic. The elastic moduli of commercially available resin cements range from 5 to 12 GPa (Li and White, 1999), while dentin is relatively elastic (γ = 18 GPa) and ceramic is inelastic (γ = 55-236 GPa; Li and White, 1999).[2]

The present study is carried out to determine the role of filler content on the flexural strength and elastic moduli of the resin luting cements. The hypothesis was made that flexural properties/strength enhancement is dependent upon the filler content of the resin cement chosen. The null hypothesis tested was that the filler content would not influence the flexural properties/strength enhancement of the selected resin luting agents.

MATERIALS AND METHODS

Three esthetic resin luting agents studied were Calibra (Dentsply); RelyX ARC and Panavia F. Ten beam-shaped specimens (L × W × H: 30 × 8 × 2 mm) were made for each of the material tested. The specimens were stored in distilled water for 24 hours at 37ºC. The specimens were then tested for flexural strength (MPa) and flexural modulus (GPa) using the three-point bending method (Fig. 1) on a universal material testing machine at a cross head speed of 0.5 mm/min. Data obtained were statistically analyzed using Analysis of variance (ANOVA) followed by posthoc - Tukey's test with $p < 0.05$ for statistical significance.

Mechanical properties of dental materials are usually evaluated by flexural tests.[3-5] Flexural testing is an ideal method of mechanically testing brittle materials, such as composite resin luting agents, because gripping of the specimen is not required as it is for tensile testing. Also, flexural testing yields larger displacement at a given load, thus making it easier to measure modulus accurately as compared to tensile or compressive tests. These may be the reasons why the ISO specification for polymer-based restorative and luting dental materials (ISO 4049) only tests flexural strength.[6] ISO 4049 classifies dental polymer-based restorative materials into two types: Type I materials are purported by the manufacturer to be suitable for restorations involving occlusal surfaces, and all other polymer-based restorative materials are considered Type II materials.[6] The minimum flexural strength requirement for Type I is 80 MPa and for Type II is 50 MPa, for all three polymerization modes.[6] Flexural strength describes the amount of force required to bend and break the material when a test piece of specific thickness is loaded.[5] This is also called the 'transverse strength'; a higher number indicates greater stiffness.[7] A low flexural modulus indicates a material that bends easily and requires more support.[8] One of the commonly used flexural strength test methods is the three-point bending test.

The flexural modulus is measured in the elastic region, where the force-deflection curve is linear. During the three-point bending test, there is a neutral axis in the specimen; below the loading point and above the neutral axis, the specimen is in compression, whereas in the lower portion of the specimen it is in tension.[1] The test evaluates the mechanical properties of a limited area directly below an applied load.

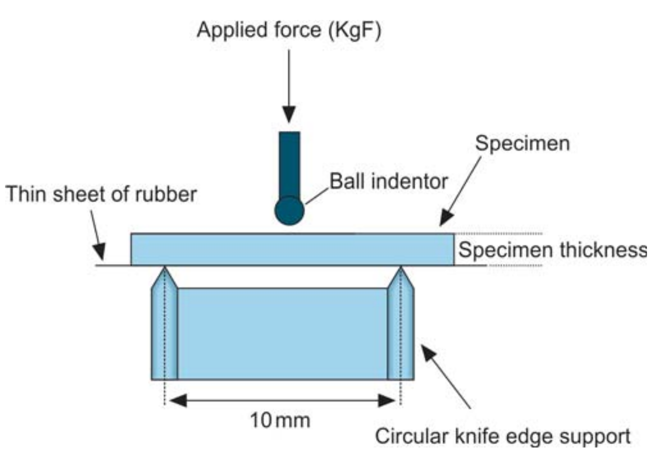

Fig. 1: Schematic representation of the flexural strength determination setup

The purpose of this study was to investigate the effect of filler loading on flexural properties and elastic moduli of three esthetic resin luting agents tested by three-point bending. The null hypothesis tested was that filler loading would not influence the flexural properties of esthetic resin luting agents.

The following test materials were investigated: Resin composite cement (Panavia F, Kuraray, Osaka, Japan) (1:1 paste:paste by volume, hand mixed with a spatula in 20 seconds);[8] Calibra (Dentsply, 1:1 paste:paste by volume, hand mixed with a spatula in 30 seconds); RelyX ARC (3M, 1:1 paste:paste by volume, hand mixed with a spatula in 30 seconds).[9] All materials were handled and proportioned according to manufacturers' instructions. Polytetrafluoroethylene molds, used to form the specimens, were filled and closed between glass microscope slides.[10,11] Test specimens used in this study were made in bulk because layered specimens are weaker than bulk specimens and luting cements are not layered clinically.[10] To make large test specimens, cement mixes were many times larger than usually used for clinical purposes, but the proportions were not changed. Specimens were individually measured with a micrometer (± 50 mm) for all calculations.

A series of 10 specimens were allocated into three groups as follows:

- *Group A:* RelyX ARC cement (3M ESPE), n = 10
- *Group B:* Calibra resin luting cement (Dentsply), n = 10
- *Group C:* Panavia F resin luting cement (Kuraray), n = 10.

Elastic modulus is important because it is a measure of the ability of a material to resist elastic deformation on when loaded. Elastic modulus is the most structure insensitive of the basic material properties, because it is determined by binding forces between atoms. Hence, it is a useful parameter to follow overtime to discern changes within a material. Beam-shaped specimens, length 30 mm, width 8 mm and height 2 mm, were evaluated with a nondestructive dynamic method that used impulse excitation of vibration. The resonance frequencies of the specimens were measured by a sonic method (Grindosonic, JW Lemmens Inc) and the elastic moduli were calculated in Giga pascals (GPa) according to the methods of Spinner and Teft.[12,13]

Flexural elastic modulus (E) was calculated as follows:

$$E = 0.9465\,(mf_f^2/b)(L3/t3)T1 \;.......... (1)$$

where m = mass of the bar,
b = width of the bar,

L = length of the bar,
t = thickness of the bar,
ff = fundamental frequency of the bar in flexure,
T1 = correction factor for fundamental flexural mode to account for finite thickness of bar, Poisson's ratio and other experimental constants. T1 was calculated as follows:

T1 = {1 + 6.585 (1 + 0.0752 m + 0.8109 m^2)(t/L)2 – 0.868 (t/L)4} – {[8.34 (1 + 0.2023 m + 2.173 μ^2) (t/L)4]/[1 + 6.338 (1 + 0.1408 m + 1.536) (t/L)4]} .... (2)

where m = Poisson's ratio.

Poisson's ratios were estimated by using an iterative process gathered from published data.[13,14] This test was repeated 5 times for each specimen, after 1 hour storage at 37°C in an atmosphere of 100% humidity, after an additional 23 hours and 1 week, during storage in water at 37°C.[15] Storage water was changed at each measurement interval and then on weekly basis.

For flexural testing, beam-shaped specimens, length 30 mm, width 8 mm and height 2 mm, were made and stored at 37°C in 100% humidity for 1 hour before being transferred to water at 37°C for an additional 23 hours. Then specimens were mounted in three-point flexure with a span of 20 mm and loaded at a crosshead rate of 5 or 0.5 mm/min with the same universal testing machine and the load plotted against time on a chart recorder at appropriate sweep rates. First measurable deflection from linearity was used to describe the proportional limit. Flexural proportional limits and strengths were calculated in MPa as follows:

FS = 3 F l/(2 b d2) .... (3)

where F = force,
l = distance between supports,
b = width and
d = thickness.

Flexural resiliences and toughnesses were calculated in MJm–3 by measurement of the areas under the appropriate curves. This test was repeated 10 times for each material/loading rate group.

Two-way ANOVA was calculated for each of the previously mentioned experimental test parameters by material ($p < 0.05$). In the event that significant differences were discerned among materials, Tukey's multiple comparisons tests were performed to determine which materials were different from each other ($p < 0.05$).

RESULTS

The mean three-point flexural strengths of the Rely X ARC, Calibra and Panavia F were 118.2, 114 and 134.5 MPa respectively, and the paired Tukey test comparisons identified significant difference ($p < 0.001$) among groups (Tables 1 to 3). The mean elastic moduli were 8.2, 7.3 and 11.5 GPa respectively (Table 2). One-way ANOVA and paired Tukey test comparisons identified significant differences ($p < 0.001$) among the mean elastic moduli for the three materials tested (Table 3).

The one-way ANOVA and paired Tukey test comparisons identified a significant difference ($p < 0.01$) between the mean flexure strength of the (Group A) 1.41(0.26) MPa, (Group B) 1.27 (0.19) MPa and (Group C) 1.43 (0.28) MPa (Table 4).

DISCUSSION

The survivability of the multimaterial clinical structures will be influenced by material thickness ratios, geometric design factors, processing variables and thermal history in addition to the mechanical and elastic properties of cementing materials.[1] Conventional cements used with many crowns in the past have not provided the necessary transfer of stress from restoration to tooth.[2]

Plastic deformation has been suspected to contribute to the dental cement failure. Excessive elastic deformation and substantial plastic deformation during loading may be related to fatigue, failure and ultimately to undesirable clinical performance.[16] Elastic moduli in the intermediate range between those of restorations and tooth structure are desirable because this can reduce interfacial stress concentrations without causing excessive strains.[16]

Elastic modulus is important because it's a measure of the ability of a material to resist elastic deformation when loaded. Elastic modulus is the most structure sensitive of the basic material properties, because it is determined by binding forces between atoms. Hence, it is a useful parameter to follow overtime to discern changes within a material.[16] Methods used for determination of elastic modulus have some theoretical or practical drawbacks. When static stress-strain measurements are used to evaluate elastic modulus, it is important not only to consider deformation of the test specimen, but also to consider deformation of the load cell, test frame, and test jig, so that the resultant total deformation can be correctly attributed to all its sources. Furthermore, the load cell and both dimensions of the chart recorder (sweep rate and load) must be accurately calibrated.

Another factor associated with the favorable performance of resin luting cements is the synergistic nature of bond between the dentin and cementing material and the cementing material to porcelain. This reduces the risk of crack propagation, thereby producing a better performance of the luted ceramic prosthesis.[17]

Table 1: Composition of the selected resin luting cement with its respective filler loading and average size of filler particle

Resin luting cement	*Manufacturer*	*Resin matrix composition*	*Filler loading*	*Average size (range) of filler particle (µm)*
RelyX ARC Two-paste clicker system	3M ESPE	• Bisphenol-A-diglycidyl ether dimethacrylate (BisGMA) • Triethylene glycol dimethacrylate (TEGDMA) • Zirconia/silica filler	67.5 wt%	1.5
Calibra Two-paste system	Dentsply/Caulk, Milford, Del	Base: • Dimethacrylate resins • Camphorquinone (CQ) • Photoinitiator • Stabilizers • Glass fillers • Fumed silica • Titanium dioxide, pigments Catalyst: • Dimethacrylate resins • Stabilizers • Glass fillers, fumed silica	67.0 wt%	1.3-1.5
Panavia F Two-paste system	Kuraray Medical, Tokyo, Japan	• MDP, comonomers filler, NaF, BPO • Comonomers filler, NaF, amine, initiator • HEMA: 2-hydroxyethyl methacrylate • MDP: 10-methacryloyloxydecyl dihydrogen phosphate • 5-NMSA: N-methacryloxyl-5-aminosalicyclic acid	70.8 wt%	2.0

Table 2: Mean flexural strengths and flexural moduli of resin cements RelyX ARC (3M ESPE), Calibra (Dentsply) and Panavia F (Kuraray)

	RelyX ARC	*Calibra*	*Panavia F*
Mean flexural strength (MPa)	118.2	114	134.55
Mean elastic modulus (GPa)*	8.2	7.3	11.5
Filler loading (%)*	67.5%	67.0%	78.0%
Biaxial strength range (Mpa)	1.03-2.05	1.04-1.85	1.07-2.02
Biaxial flexure stress (Mpa), SD	1.41 (0.26)	1.27 (0.19)	1.43 (0.28)
Mean porcelain strength of coated specimens	16.39%	4.10%	17.2%

Table 3: Descriptive statistics of all the groups in terms of minimum, maximum and mean biaxial flexure strength of the specimens and their standard deviations

Study groups	*Minimum*	*Maximum*	*Mean*	*SD*
Group A	1.03	2.05	1.42	0.27
Group B	1.04	1.85	1.27	0.19
Group C	1.07	2.02	1.43	0.28

Table 4: Comparison values of the biaxial flexure strength of all the groups using one-way ANOVA and Tukey's multiple range test

Study groups	*Mean*	*p* value*	*Significant pairs***
Group A	1.42	p < 0.001	A and B
Group B	1.27	Highly significant	B and C
Group C	1.43		C and A

*One-way ANOVA test; **Tukey's multiple range test

The present study with a sample size of n = 10 in each group, the elastic moduli of the respective cements were 8.2 (0.4), 7.3 (0.3), and 11.5 (0.5) GPa and associated mean respectively (within parenthesis). A linear relationship was observed between the mean elastic moduli and the filler loading of the individual resin luting agents, highlighting a dependence of the strengthening process on the filler loading of the resin. The elastic moduli of the resins were related to the filler loading of 67.5, 67.0 and 78.0 wt% for RelyX ARC (Group A), Calibra (Group B), and Panavia F

(Group C) respectively. The observations were consistent with the findings of Fleming et al (2006), who reported that the strengthening of ceramics following resin coating was independent of a controlled defect population.

Resin composite cements are universally used for luting porcelain veneers.[18] The ideal material for inserting porcelain laminate veneers would be composite resin with a high filler concentration for strength, low polymerization shrinkage, high modulus of elasticity and a high flowability that allows the ceramic restoration to be easily placed. The material would have low solubility, as well as high compressive and tensile strengths. The proportional limit would be high and have excellent adhesion to porcelain as well as tooth structure.[23] The dental porcelains and ceramics have low critical strain values, thus a small amount of deformation results in catastrophic failure. In the case of porcelain veneers, restorations are bonded to equally stiff enamel. Thus, the stiff underlying enamel protects the brittle veneer from excessive deformation and resultant fracture. Resin-composite cements are widely used for luting all-ceramic crowns and FPDs, presumably for their esthetic qualities and other reasons.[19] All ceramic crowns and FPDs have much higher fracture rates than porcelain veneers.[20] This may be partly attributed to exposure to higher masticatory forces. However, these restorations are largely supported by flexible dentin, not by stiffer enamel.[14] Dentin undergoes viscoelastic behavior;[15] thus, all-ceramic restorations supported by dentin may more readily reach their critical strains and fracture.[15] Proportional limits, strengths, resilience, and toughness of the resin-composite cements are at least equal to those of other cements. However, their relatively low elastic moduli may remain a disadvantage in preventing deformation and fracture of brittle all-ceramic restorations (Fig. 1).[15]

The proposed hypothesis that strengthening is a function of resin elastic modulus, which again depended on the filler loading, was thereby accepted. Consequently, the strength and performance of resin-cemented all-ceramic restorations can be enhanced by the use of higher elastic modulus cements.[21]

CONCLUSION

Relatively low filler loading may remain a disadvantage in preventing deformation and fracture of brittle all-ceramic restorations. The results also showed that the flexural strength can be increased by the filler loading, but is also influenced by various factors that modulate the elastic moduli and flexural properties.[24] It is suggested that manufacturers should strive to develop adhesive resin cement with higher filler loading thereby enhancing the elastic modulus.[22]

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