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Shear Strength between Lithium Disilicate and Enamel Following Cementation with Photopolymerizing Resin Cement or Pre-Heated Compound Resin, with and Without Ultrasound

Lissandro Hybiner Teixeira[1], Geraldo Alberto Pinheiro de Carvalho[2*], Aline Batista Gonçalves Franco[2], Elimário Venturin Ramos[2], Ivone de Oliveira Salgado[3], Sérgio Candido Dias[4]

[1]Master in Dental Prosthesis - Department of Restorative Dentistry, São Leopoldo Mandic Dental School, Campinas - São Paulo–Brazil
[2]Associate Professor, DSc, Department of Restorative Dentistry, São Leopoldo Mandic Dental Research Center, Campinas - São Paulo Brazil
[3]DSc, Juiz de Fora Federal University, Minas Gerais Brazil
[4]Coordinator, Adjunct Professor, DSc, Department of Restorative Dentistry, São Leopoldo Mandic Dental Research Center, Campinas - São Paulo Brazil

***Corresponding author:** Geraldo Alberto Pinheiro de Carvalho | **Received:** 07.04.2019 | **Accepted:** 15.04.2019 | **Published:** 30.04.2019
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Abstract **Original Research Article**

The objective of this study is (i) to compare the shear strength of lithium disilicate IPS e.max® Press color A1 (Ivoclar Vivadent, Baden-Württemberg, Ellwangen, Germany) laminate disks to bovine incisive enamel using four cementation protocols; and (ii) to assess the failure mode, classified as adhesive (enamel or ceramics), cohesive or mixed, and its possible association with the technique used for cementation. To this end, 40 bovine incisives and 80 laminate disks measuring 0.5mm of thickness and 3mm of diameter were used. The test specimens were divided in four groups (n=10): G1 and G2 cemented with photopolymering resin cement RelyX® Venner (3M ESPE, St. Paul-MN, USA), using a vertical shaft in G1 and ultrasound for 5s in G2; G3 and G4 cemented with compound resin Filtek® Z350XT (3M ESPE, St. Paul-MN, USA) pre-heated at 50ºC, using vertical shaft in G3 and ultrasound for 5s in G4. Two laminate disks were cemented on each tooth, one at the mesial region, another at the incisal region with constant pressure of 500g. Shearing was performed using a chisel knife in a universal testing machine (EMIC DL2000) with load of 500Kgf at 0.5mm/s. Results for failure mode were obtained using a stereo microscope. There is no significant difference between the groups regarding shear strength or region of cementation, both within or between groups. Failure is predominantly adhesive. There is no correlation between cementation protocol and failure mode, whether at the mesial or incisal region. It is possible to conclude that all cementation protocols assessed here are indicated for clinical use since they present similar shear strength. Longitudinal clinical studies are necessary.
Keywords: Shear Strength; Dental Veneers; Resin Cements.

Introduction

Laminate veneers have been widely used in the clinical practice since it is a minimally invasive procedure and offers good aesthetical results in terms of shape and color [1].

The adhesion of laminates is based on the principles of micromechanical adhesion to enamel, dentin and the inner surface of laminates. In this sense, resin cements affect significantly the life span of the restoration [2] overcoming aesthetical limitations and adherence issues posed by conventional cements [3].

However, cementation coating is still important for the cementation durability, mainly in ceramic laminates since the thinner laminate layers require smaller amounts of organic monomers and yield less degradation. The cementation coating thickness changes according to the adhesive cement characteristics and to the professional's ability and strength [4].

Ultrasound has been used as an aid in the cementation of prosthetic components [5]. The technique consists in the placement of the ultrasound tip on the laminate for a few seconds after its positioning on the substrate. The vibration traverses the prosthetic piece and reaches the underlying cement. The ultrasound alters the cement viscosity, resulting in a smoother slide between the piece and the substrate and, thus, in an easier fitting. Thus, ultrasound application not only reduces the cementation coating, but also allows a more uniform distribution of cement under the laminate. Therefore, it is possible to assume that the use of ultrasound during cementation leads to a better polymerization and, thus, increased adhesion of laminates to the tooth [6].

In addition to resin cement, pre-heated compound resin has been employed for the cementation of ceramic laminates. According to cementation protocols, compound resins can be pre-heated to 50ºC - 68ºC using pre-heating devices such as Calset™ (AdDent Inc., Danbury, USA) and EnaHeat (Micerum, Avegno- GE, Italy). The procedure results in reduced resin viscosity [7, 9] leading to better marginal adaptation [7,8] increased dispersion[10] and reduced coating thickness[11] improving its application[12]. However, some points are still in debate, such as the increase in polymerization degree, flexural strength, reticulation, and microhardness of the compound resin during pre-heating.

Literature presents several cementation protocols for ceramic laminates. In this sense, this study aims to compare the shear strength of four different cementation protocols of ceramic laminates: photopolymerizing resin cement using metal shaft; photopolymerizing resin cement using ultrasound; pre-heated compound resin using metal shaft; and pre-heated compound resin using ultrasound. The study also assesses the failure modes formed at cementation rupture. Failure modes for enamel or laminate disks are classified as adhesive, cohesive or mixed.

Materials and Methods

This study was approved by the Animal Research Ethics Committee (CONEA) of the São Leopoldo Mandic School under protocol #2016/033.

Forty healthy incisive bovine teeth were used for the substrate. The teeth were stored in a solution of thymol 0.1% and distilled water. They were cut at the cementoenamel junction using a precision cutting machine (Labcut1010, Extec, Enfield, USA), and were included in Epoxi resin (Redelease®, Sao Paulo, Brazil) in a PVC tube measuring 20mm of diameter and 25mm of height (Tigre, Sao Paulo, Brazil).

The enamel was polished with wet-and-dry paper (220 and 600 grit) (Norton®, Sao Paulo, Brazil) on the grinding and polishing machine (PL02, Metalprisma, Guarulhos-SP, Brazil). The specimens were stored in a heating chamber, with distilled water at 37ºC until the cementation procedure. Tablets IPS e. max® Press color A1 (Ivoclar Vivadent, Baden-Württemberg, Ellwangen, Germany) were used for the Lithium disilicate laminates.

The ceramic injection started with the inclusion of four cylindrical sprue wax standards (Babinete, Maringa-PR, Brazil) measuring 3.0mm of diameter and 25.0mm of lenght. IPS®PressVestSpeed (IvoclairVivadent, Schaan, Ellwangen, Germany) was used in coating. The injection molding machine Programat® EP500 (IvoclairVivadent, Schaan, Ellwangen, Germany) was set in its default heating cycle to obtain the ceramic sticks.

The ceramic sticks were glued together using impression wax Godiva Exata (DFL, Curiacica-RJ, Brazil) and taken to a universal cutting machine Labcut1010 (Extec, Enfield, USA) for the manufacturing of disks measuring 3.0mm of diameter and 0.5mm of thickness [27]. All measurements were checked using a digital pachymeter (Mitutoyo, Absolute, Suzano-SP, Brazil).

The selected laminates were then manually finished using wet-and-dry paper with 220 and 600 grit (Norton®, Guarulhos-SP, Brazil) [34] (Figure 1).

The test specimens were distributed in four groups (n=10) with two cemented disks per specimen, one applied to the medium portion and the other to the incisal portion of the tooth.

G1 – Resin cement RelyX®Veneer/ cementation using metal shaft

G2 – Resin cement RelyX®Veneer / ultrasound cementation;

G3 – Pre-heated resin Filtek® Z350XT/ cementation using metal shaft;

G4 – Pre-heated Filtek® Z350XT/ ultrasound cement.

The cementation area - defined as the area to receive treatment for the cementation of the ceramic disk - was standardized as 4mm of diameter. The ceramic disks were treated with hydrofluoric acid 10% for 20 seconds, rinsed with deionized water, left to dry, and treated with a layer of silane (RelyX® Ceramic Primer 3M ESPE, St. Paul-MN, USA).

To standardize the cementation, an apparatus with a movable vertical shaft and constant weight of 500g was used to apply pressure for 1 minute. Ultrasound was, and then applied vertically on the ceramic disk for 5s at 30% of power.

Photopolymerization was obtained using an Optilight Max (Gnatus, Ribeirao Preto-SP, Brazil), checked before the beginning of each new group using a radiometer Demetron® 100 (Kerr, Orange-CA, USA), ensuring minimum intensity of 600 mW/cm^2. The substrates were treated prior to the cementation according to the manufacturers' instructions.

Groups were treated as following. Groups G1 and G3 (cemented without ultrasound): application of phosphoric acid 37% for 30s, rinsing for 30s and drying of surface; application of two layers of the adhesive Adper™Scothbond™Muti-porpuse (3M ESPE, St. Paul-MN, USA) and photopolymerization for 10s at a distance of 10mm; Group G1: insertion of cement RelyX® Venner (3M ESPE, St. Paul-MN, USA);

Group G3: application of the resin Filtek® Z350XT (3M ESPE, St. Paul-MN, USA) pre-heated using EnaHeat (Micerum, Avegno-GE, Italy); the temperature was gauged at every cementation using an infrared thermometer Pyromed® PY-380 (Pyromed, Contagem-MG, Brazil). Cementation was done at constant pressure by the vertical shaft weighting 500g for 60s; photopolymerization was carried on for 20s juxtaposed to the laminate disk. Following cementation, the specimens were submersed in distilled water and stored in a heating chamber at 37oC.

Groups G2 and G4 (cemented with ultrasound) were submitted to the same cementation procedure except that, before the application of pressure by the shaft, the specimens were subjected to 5s of ultrasound application with a Jet Sonic (Gnatus, Ribeirao Preto - SP, Brazil) and tip number G20.

After 7 days of storage, the test specimens were subjected to a shear mechanical test using an universal testing machine EMIC DL2000 (EMIC, Sao Jose dos Pinhais-PR, Brazil) with a load cell of 500Kgf and actuator speed of 0.5mm/min (Figure 2).

Data were organized in a spreadsheet and subjected to Kolmogorov-Smirnov and Shapiro-Wilk normality tests followed by ANOVA and Levene's. The statistical tests were conducted on Bio Estat, version 3.0. For the failure mode assessment, the samples surfaces were assessed with a stereo microscope (Discovery V8, Carl Zeiss, Birkerod, Denmark), using magnification of 16X.

RESULTS

This study considered two dependent variables - resistance (MPa) in two levels (mesial and incisal regions), and one independent variable (group) in four levels: G1, G2, G3 and G4.

The individual shear strengths of each group (G1, G2, G3, G4) and region (mesial and incisal) are represented in figure 3. A descriptive analysis of each group, containing average and standard deviation for mesial and incisal regions is given in Table 1.

The Kolmogorov-Smirnov and Shapiro-Wilk normality tests were applied to the two dependent variables in order to verify the data distribution. Levene's test was applied to verify variance homogeneity between groups and to perform intergroup and interfactor comparisons. Using two-way repeated measures ANOVA, we compared the groups (independent variable) considering mesial or incisal regions (independent variable). The analysis shows no significant difference between groups or regions. It also shows no significant interaction between factors.

For the failure mode assessment, three association tests (chi-square) were performed to check association between:

- Group and failure at the incisal region: no significant association observed;
- Group and failure at the mesial region: no significant association observed (Table 2);

Fig-1: Selection of the laminate disks

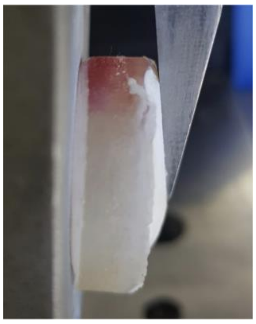

Fig-2: Shear strength mechanical test

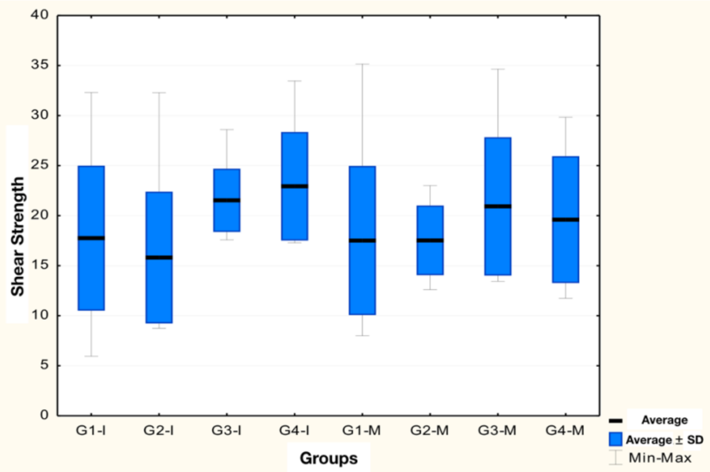

Fig-3: Box-plot representing shear strengths the four groups (G1, G2, G3, G4).

Table-1: Descriptive analysis of the groups

Group	I_Mpa Average	I_Mpa Stand. Dev.	M_Mpa Average	M_Mpa Stand. Dev.
G1	17.76	7.24	17.52	7.45
G2	15.82	6.59	17.53	3.48
G3	21.54	3.16	20.93	6.92
G4	22.94	5.42	19.60	6.34

Table-2: Group x mesial region

Groups	G1	G2	G3	G4	
Adhesive-mesial region	10	9	10	10	Result
	9.75	9.75	9.75	9.75	Expected Result
	25.641	23.077	25.641	25.641	Row%
	100	90	100	100	Column%
	25	22.5	25	25	Total %
Mixed-mesial region	0	1	0	0	Result
	0.25	0.25	0.25	0.25	Expected Result
	0	100	0	0	Row %
	0	10	0	0	Column %
	0	2.5	0	0	Total %
Chi-square= 3.077 with 3 degrees of freedom. (P = 0.380)					

DISCUSSION

Ceramic laminates used in Dentistry have shown safe and satisfactory results for the clinical practice [13]. Treatment success, given by its long lifespan [14, 15] depends on the choice of materials, the clinical case, the technique applied, and the tooth's wearing [16]. Laminates have shown fracture strength similar to that of intact teeth when manufactured with enamel preparation [17].

Among all the aforementioned aspects, the proper adhesive technique is the main factor of success [18, 19]. Literature presents different cementation protocols that might include photopolymerizing resin cement [20, 21] dual resin cement [6, 13] flow compound resin [22] pre-heated compound resin [8, 10-12] and with and without ultrasound [3, 6].

The pre-heating of compound resin is consistently associated with lower viscosity, better marginal adaptation and easier management [8,9,12] however, the specialized literature disagrees in terms of the degree of conversion of resinous monomers, with some reports claiming an increase [12, 20] while others claim no increase in conversion[8] in association with pre-heating. Regardless of that, this study shows that resin pre-heating does not affect resistance to adhesion in comparison with the photopolymerizing resin cement.

The materials used here are considered high-grade: cementing agents Rely X Venner® and compound resin Filtek® Z350 [8, 11, 22]. A constant pressure of 500g for 1 minute was used to standardize the cementation procedure. Also, ultrasound and pre-heating of compound resin to 50ºC were used to improve viscosity and runoff [9, 12].

Bovine enamel was used as substrate because it has already been ruled as an excellent a proxy for human enamel [23] and is adequate for the bond strength tests [24]. Given the conservative nature of this treatment, preparation with minimum wearing of enamel is of paramount importance given that the maximal adhesive resistance is obtained on enamel, not dentin [16, 19].

The use of lithium disilicate (IPS e.max® Press) for the ceramic disks followed the parameters defined in Zandinejad *et al*. [25] Morimoto *et al*. [26]. The thickness choice (0.5mm) considered the degree of conversion of photopolymerizing resin cement monomers and compound resin, which is not affected up to a maximum of 1.5mm [27] and the resistance to masticatory forces [28]. The diameter of 3mm is in agreement with shear strength studies [29].

The shear strength between groups and cementation region (mesial and incisal) lacked significant difference, corroborating previous studies on adhesion to enamel [6, 30, 31] while opposing studies on cementation on dentin [3, 32]. This creates a concern about the differentiation of cementation protocols according to the substrate to which the ceramic laminate will be adhered. This is reiterated by the use of ultrasound for the application of adhesive systems to different substrates [33].

Regarding failure mode, whether adhesive (enamel or ceramic), cohesive or mixed, the analysis shows a predominance of the adhesive type on enamel, corroborating the results of Hattar *et al*. [30] Oztrk *et al*. [31] Hu, Weger and Fisher [34] also found by Bulut and Atsü [35]. Hence, it is possible to infer that, in the event of a failure, it might be favorable to a new cementation. Notwithstanding, no significant association between type of failure, group or cementation region was found.

Finally, regarding the clinical implications of this *in vitro* study, it is concluded that the four protocols investigated here are safe for the cementation of ceramic laminates on enamel.

Conclusion

- There was no significant difference of shear strength between the four groups;
- There was no significant difference of shear strength between mesial and incisal regions, both within and between groups;
- The predominant failure mode was adhesive on enamel;
- There is no correlation between cementation protocol and failure mode, whether on the mesial or the incisal regions.

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