PONTIFÍCIA UNIVERSIDADE CATÓLICA DO RIO GRANDE DO SUL
FACULDADE DE ODONTOLOGIA
PROGRAMA DE PÓS-GRADUAÇÃO EM ODONTOLOGIA
MESTRADO COM ÁREA DE CONCENTRAÇÃO EM MATERIAIS DENTÁRIOS

INTERAÇÃO DE CIMENTOS RESINOSOS E MATERIAIS CAD/CAM: RESISTÊNCIA DE UNIÃO EM DENTINA

MARIÁ CORTINA BELLAN

Porto Alegre

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Dissertação apresentada como requisito parcial à obtenção do título de Mestre em Odontologia, curso de Pós-graduação em Odontologia, área de concentração em Materiais Dentários, pela Pontifícia Universidade Católica do Rio Grande do Sul.

Orientador: Prof. Eduardo Gonçalves Mota

Porto Alegre

2016

“All our dreams can come true, if we

have the courage to pursue them.”

Walt Disney

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RESUMO

O objetivo deste estudo foi avaliar a resistência de união (µTBS) dos novos materiais CAD/CAM a diferentes cimentos resinosos. Trinta e duas coroas foram usinadas utilizando materiais CAD/CAM (Vita Mark II, Vita Suprinity, Vita Enamic and Lava Ultimate) e cimentadas à dentina hígida, utilizando diferentes cimentos (Relyx ARC, Relyx Unicem 2 and Relyx Ultimate). As espécimes foram mantidas em umidade relativa de 100% a 37ºC por 24 horas e após seccionadas em palitos com disco diamantado em água corrente. As amostras (n=16) com área de aproximadamente 1 mm² foram submetidas ao teste de microtração através de uma máquina universal com velocidade de carregamento de 0.5 mm/min. As amostras foram analisadas por MEV para determinar o modo de falha. Os dados foram analisados por meio do ANOVA de dois fatores e do teste Tukey (α=0,05). A resistência de união foi significantemente afetada pelo material e pela interação deste com os cimentos resinosos (p=0.001). Os valores de µTBS variaram de 12.17 para Mark II com Relyx ARC e 32.93 para Lava Ultimate com Relyx Unicem 2. Considerando o comportamento geral dos materiais testados, Vita Enamic (29.39 Mpa) e Lava Ultimate (28.42 Mpa) obtiveram os maiores valores de µTBS em comparação ao Vita Mark II (13.13 MPa) e o Vita Suprinity (14.55 MPa). Os valores de µTBS para Vita Enamic não diferiram estatisticamente daqueles obtidos com Lava Ultimate (p=0.845) e o mesmo ocorreu com Vita Mark II e Vita Suprinity (p=0.986). Não houve diferença estatística entre os cimentos resinosos (p=0.176). Uma união promissora pode ser alcançada com os materiais CAD/CAM híbridos à estrutura dentária e a diferentes cimentos resinosos após 24 horas de armazenamento em água.

Palavras-chave: união dentária, resistência de união, dentina, cimentos resinosos, cad-cam.

ABSTRACT

The aim of this study was to evaluate the bond strength (µTBS) of novel CAD/CAM restorative materials to different luting cements. Thirty two crowns were milled using CAD/CAM materials (Vita Mark II, Vita Suprinity, Vita Enamic and Lava Ultimate) and luted to sound and fresh cutted dentin using different cements (Relyx ARC, Relyx Unicem 2 and Relyx Ultimate). The specimens were stored in relative humidity of 100% at 37ºC for 24 h and, then sectioned into sticks with water-cooled diamond blade with low-speed cutting saw. The samples (n=16) with cross-sectional areas of approximately 1 mm^2 were submitted to tensile bond strength test in a universal testing machine with crosshead speed of 0.5 mm/min. The samples were analyzed with SEM to determine the failure mode. Data were analyzed using 2-way ANOVA and Tukey's test (α=0,05). Bond strength was significantly affected by the material and interaction between them and luting cements (p=0.001). The µTBS (MPa) values ranged from 12.17 for Vita Mark II with Relyx ARC and 32.93 for Lava Ultimate with Relyx Unicem 2. Considering overall behavior of tested materials Vita Enamic (29.39 MPa) and Lava Ultimate (28.42 MPa) obtained higher µTBS values in comparison to Vita Mark II (14.14 MPa) and Vita Suprinity (14.55 MPa). The µTBS values for Vita Enamic did not differ from those obtained with Lava Ultimate (p=0.845) and the same occurred with Vita Mark II and Vita Suprinity (p=0.986). There was no statistical difference between cements (p=0.176). A promising bonding performance can be achieved with the hybrid CAD/CAM restorative materials to tooth structure and to different types of luting cements after 24h of water storage.

Key Words: dental bonding, tensile strength, dentin, resin cements, cad-cam.

LISTA DE FIGURAS, TABELAS E GRÁFICOS

LISTA DE SIGLAS, SÍMBOLOS E ABREVEATURAS

µTBS – resistência de união à microtração

CAD/CAM – desenho assistido por computador / manufatura assistido por computador

% - porcentagem

°C – grau Celsius

3D – tridimensional

TEGDMA – dimetacrilato de trietilenoglicol

UDMA – uretano de metacrilato

bis-EMA – bisfenol etoxilato dimetacrilato

bis-GMA – bisfenol glicidil metacrilato

HEMA - 2-hidroxietil metacrilato

nm – nanômetro

Kg – quilograma

s – segundo

mW/cm^2 – mili Watt por centímetro quadrado

h – hora

min – minuto

µm – micrômetro

mm - nanômetro

N - Newton

MPa – megapascal

n – número de amostras confeccionadas

p – valor de significância

α – significância estatística

GPa – Giga Pascal

MDP - meta-criloiloxidecil diidro-genofosfato

SUMÁRIO

INTERACTION OF LUTING CEMENTS AND CAD/CAM MATERIALS: MICROTENSILE BOND STRENGTH IN DENTIN

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1 INTRODUCTION

The use of CAD/CAM (computer-aided design/computer-aided manufacturing) became popular during the past decades in dentistry. This technology allows the dentist to mill restorations in a single visit, merging features such as speed and easy handling with longevity[1]. The restorations can be milled using ceramics (feldspar, leucite, lithium based, zirconia and alumina) [2] and hybrid materials (resin nanoceramic and polymer-infiltrated ceramic network)[3,4].

In order to associate the ceramic characteristics (high aesthetic, wear resistance, biocompatibility and color stability) with those of composites (viscoelastic behavior, less wear of the opposite arch), improving its properties, new materials with different compositions to be used in CAD/CAM system have been recently introduced in the market, named hybrid materials[3-7].

Within this materials, Lava Ultimate[3-4,8], (3M ESPE, St. Paul, MN, USA) is a resin nanoceramic, composed of nanoceramic particles embedded in a highly cross-linked resin matrix and Enamic[4,9-10] (VITA Zahnfabrik, Bäd Sackingen, Germany) is a material polymer-infiltrated ceramic network that consists of a feldspar ceramic network infiltrated by dimetacrylate polymer network.

An additional material that became available for CAD/CAM technology is Suprinity (VITA Zahnfabrik, Bäd Sackingen, Germany) that have excellent optical properties. This material is a zirconia-reinforced lithium silicate ceramic that supports a wide range of applications and after crystallization, exhibits higher mechanical properties[11-12].

To bond CAD/CAM materials to tooth structure and support the oral environment adhesive luting is recommended. However, total etch adhesive strategy is a complex, multistep technique and may compromise the effectiveness of bonding. Due this, self-adhesive resin cements were developed to simplify the bonding procedures, reducing clinical steps, and shortening the “window of contamination”. These luting materials do not require any pretreatment of the tooth surface such etchant, primer, or bonding agent, thus, the cementation can be done in a single step[13-16].

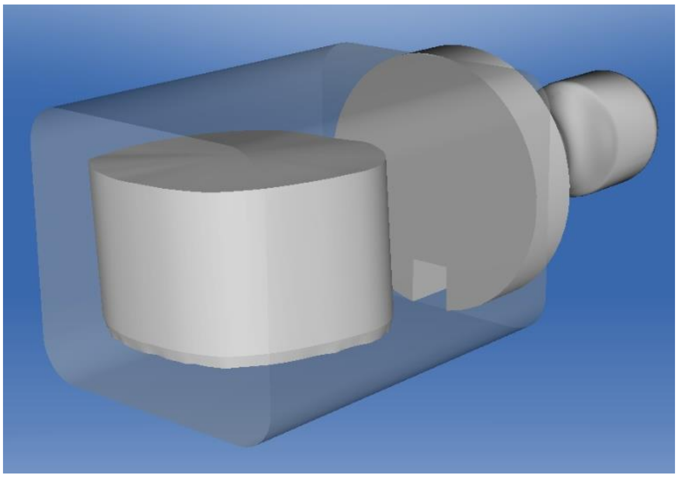
Due to innumerous variables widely described in the literature, doubts about interactions of luting cement, restorative material, conditioning protocol treatment remains in clinical practice. The present study aimed to evaluate and compare the microtensile bond strength of novel CAD/CAM restorative materials to three resin cements in order to test the following null hypothesis: (1) there is no difference in the µTBS between restorative materials, (2) there is no difference in the µTBS between the luting cements and (3) there are no interactions in µTBS between restorative materials and luting cements.

2 MATERIALS AND METHODS

Thirty two intact caries-free extracted human third molars were selected for this study. All teeth were stored in an aqueous solution of 0,5% chloramine-T at 4ºC[15,16,17] during 7 days after extraction and thereafter stored in distilled water at 4ºC for a maximum of 6 months. The teeth were collected under a protocol reviewed and approved at local ethics committee (PUCRS; CAAE 48466815.7.0000.5336). Flat coronal dentin surfaces were exposed by removing occlusal enamel and superficial dentin with a slow-speed, water-cooled diamond saw (Labcut 1010, Exterc Corp., London, England). Dentin surfaces were abraded with #600 grit silicon carbide (SiC) paper under running water to create standardized smear layers and then ultrasonically cleaned in distilled water for 5 minutes.

The flat surface were coated (CerecOptispray, Sirona, Bensheim, Germany) and scanned with CEREC®Omnicam (Sirona, Bensheim, Germany). The result was an exact virtual 3D model of the flat surface and in this model was design a telescopic crown as described in Figure 1. The crowns were milled with the manufacture's instructions by Cerec MC XL (Sirona, Bensheim, Germany).

Figure 1. CAD model of the telescopic crown positioned into the material block.



The materials used in this study are presented in Table 1. The specimens were divided into 12 groups according to material (Vita Mark II, Vita Suprinity, Vita Enamic and Lava Ultimate) and luting cement (Relyx ARC, Relyx Unicem 2 and Relyx Ultimate). Detailed cementation process is described in Table 2.

Table 1 – Materials, composition, batch number and manufacters.

Material (batch #)	Product and composition*	Manufacter
Vita Mark II (45500)	Silicon dioxide 56–64%, aluminum oxide 20–23%, sodium oxide 9–11%, potassium oxide 6–8%, calcium oxide 0.3–0.6%, titanium dioxide 0.0–0.1%	Vita Zahnfabrik
Vita Suprinity (48940)	Zirconia reinforced lithium silicate ceramic, zirconium oxide 8–12%, silicon dioxide 56–64%, lithium oxide 15–21%, various >10%	Vita Zahnfabrik
Vita Enamic (45810)	Hybrid ceramic (resin infiltrated ceramic network) Ceramic: silicon dioxide 58–63%, aluminum oxide 20–23%, sodium oxide 9–11%, potassium oxide 4–6%, boron trioxide 0.5–2%, zirconia and calcium oxide	Vita Zahnfabrik

	Polymer part (25%): UDMA and TEGDMA	
Lava Ultimate (N708503)	Cured dental restorative, consisting of silica nanomers (20 nm), zirconia nanomers (4–11 nm), nanocluster particles derived from the nanomers (0.6–10 nm), silane coupling agent, resin matrix (BisGMA, Bis-EMA, UDMA and TEGDMA).	3M ESPE
Relyx ARC (1526500149)	PASTE A: silane treated ceramic, TEGDMA, BisGMA, silane treated silica, functionalized dimethacrylate polymer, triphenylantimony PASTE B: silane treated ceramic, TEGDMA, BisGMA, silane treated silica, functionalized dimethacrylate polymer, 2-benzotriazolyl-4-methylphenol, benzoyl peroxide	3M ESPE
Adapter Single Bond 2 (N688653)	BisGMA, HEMA, UDMA, dimethacrylates, ethanol, water, camphorquinone, photoinitiators, polyalkenoic acid copolymer, 5-nm silica particles	3M ESPE
Relyx Unicem 2 (588286)	Base paste: silane‑treated glass powder, 2‑propenoic acid, 2‑methyl‑, reaction products with 2‑hydroxy‑1,3‑propanediyl dimethacrylate and phosphorus oxide, TEGDMA, silane, treated silica, sodium persulfate, glass powder, tertbutyl peroxy‑3,5,5‑ trimethylhexanoate, cooper acetate monohydrate Catalyst paste: Silane‑treated glass powder, substituted dimethacrylate, 1‑benzyl‑5‑phenyl‑barbic‑acid, calcium salt, silane‑treated silica, sodium p‑toluenesulfinate, 1,12‑dodecane dimethacrilate, calcium hydroxide, methacrylated aliphatic amine, titanium dioxide	3M ESPE
Relyx Ultimate (1516800384)	Base paste: Silane‑treated glass powder, 2‑propenoic acid, 2‑methyl‑, reaction products with 2‑hydroxy‑1,3‑propanedyl dimethacrylate and phosphorus oxide, TEGDMA, silane‑treated silica, oxide glass chemicals, sodium persulfate, tertbutyl peroxy‑3,5,5‑ trimethylhexanoate, copper acetate monohydrate Catalyst paste: Silane‑treated glass powder, substituted dimethacrylate, 1,12‑dodecane dimethacrylate, silane‑treated silica, 1‑benzyl‑5‑phentyl‑barbic‑acid, calcium salt, sodium p‑toluenesulfinate, 2‑propenic acid, 2‑methyl‑, di‑2,1‑ethanediyl ester, calcium hydroxide, titanium dioxide	3M ESPE
Scotchbond Universal (1516800384)	BisGMA, HEMA, Decamethylene dimethacrylate, ethanol, water, silane treated silica, 2-propenoic acid, methacrylated phosphoric acid, copolymer of acrylic and itaconic acid, ethyl-4-dimethylaminobenzoat, camphorquinone, (dimethylamino)ethyl methacrylate, methyl ethyl ketone	3M ESPE

*The chemical composition information was obtained from the manufacturer's material safety data sheet. BisGMA: Bisphenol A‑diglycidyl dimethacrylate; Bis-EMA ethoxylated bisphenol A dimethacrylate; TEGDMA: Triethylene glycol dimethacrylate; UDMA: urethane dimethacrylate; HEMA: hydroxyethyl methacrylate.

The crowns were etched according to manufacture's instructions described in Table 2. A standardized constant pressure of 2 Kg was applied to lute the crowns to the preparation, using a customized metallic device. The specimens were light cured with a high-intensity LED curing unit calibrated at 1900 mW/cm^2 (LED, 3M ESPE, St. Paul, MN, USA) for 40 s in each side in Relyx ARC groups and 20 s for the other luting cements groups. The bonded specimens were stored in distilled water for 24 hours at 37ºC waiting for the monomers conversion.

Table 2 – Description of bonding protocols.

<table>
<tr><th>Luting Cement</th><th>Relyx ARC</th><th>Relyx Unicem 2</th><th>Relyx Ultimate</th></tr>
<tr><td>Dentin surface</td><td>Cleaned with pumice at low speed.
Etched with 37% phosphoric acid (FGM, Joinville, SC, Brazil, batch #300115) for 15 s, washed and gently air-dried.
Application of Adper Single Bond 2 agitating for 15 s. Gently air-dried for solvent evaporation and photopolymerized for 20 s.</td><td>Cleaned with pumice at low speed.</td><td>Cleaned with pumice at low speed.
Application of Scotchbond Universal agitating for 20 s and gently air-dried for solvent evaporation (5 s).</td></tr>
<tr><td>Material Preparation</td><td colspan="3">1. Sandblasted abraded with 50-μm aluminum-oxide (Al_2O_3) particles with a dental airborne-particle abrasion unit (Bioart, São Carlos, SP, Brazil).
2. Ultrasonically cleaned for 5 min in distilled water[6,16-20] and air dried.
3. Etched with hydrofluoric acid 10%[21-23] (FGM, Joinville, SC, Brazil, batch #270715) - Mark II (60 s), Suprinity (20 s), Enamic (60 s), except for Lava Ultimate that was only sandblasted.
4. Ultrasonically cleaned for 5 min in distilled water and air dried.
5. Application of silane coupling agent (Dentisply, Petrópolis, RJ, Brazil, batch #101338H) for 60 s and air dried in all restorative materials for Relyx ARC and Relyx Unicem 2 luting cement. For the cement Relyx Ultimate was applied a Scothbond Universal coat and gently air-dried.</td></tr>
</table>

After 24 h the specimens were vertically sectioned into serial slabs and further into sticks with water-cooled diamond blade with low-speed cutting saw. Sixteen samples (n=16) with cross-sectional areas of approximately 1mm² were obtained for each group. The specimens were attached to the universal testing machine (DL-2000 EMIC, São José dos Pinhais, Paraná, Brazil) device using cyanoacrylate (Super Bonder Gel, Loctite Brazil Ltda, SP, Brazil) and stressed to failure with a low cell of 50N and a crosshead speed of 0.5 mm/min. The load at failure (N) and the surface area (mm²) for each sample was used to calculate the µTBS in MPa through Mtest software (T-Systems, São Paulo, Brazil).

Fractured specimens from each group (n=16) were air dried, mounted on metallic stubs, sputtered with gold layer, and then examined under a scanning electron microscope (SEM) (Inspect F50, FEI, Oregon, USA) at different magnifications (250x, 1000x and 2000x) to determinate the failure modes. Failure mode at the fractured interface was classified into five types: cohesive failure into cement, adhesive failure between dentin or adhesive and cement, adhesive failure between cement and restorative material, cohesive failure into restorative material and mixed failure when more than one type has occurred[19-20].

Statistical analysis was conducted using SPSS, version 17 (SPSS Inc., Chicago, IL, USA). Two-way analysis of variance was perfomed (α=0.05) with the bond strength as the dependent variable and type of restorative material (Vita Mark II, Vita Suprinity, Vita Enamic and Lava Ultimate), and cement type (Relyx ARC, Relyx Unicem 2 and Relyx Ultimate) were treated as between subjects factors followed by post-hoc Tukey's test (α=0.05).

3 RESULTS

The mean µTBS values (MPa) and standard deviations are presented in Table 3. Two-way ANOVA of the µTBS data revealed that: the bond strength was significantly affected by the material (*p*=0.001) and interaction between restorative materials and luting cements (*p*=0.001), however there was no statistical difference between the tested luting cements (*p*=0.176). Considering overall behavior of tested material, Vita Enamic and Lava Ultimate obtained higher µTBS values, 29.39 MPa and 28.42 MPa respectively, in comparison to Vita Mark II and Vita Suprinity that obtained the lowest µTBS values, 14.14 MPa and 14.55 MPa respectively. The µTBS values for Vita Enamic did not differ from those obtained with Lava Ultimate (*p*=0.845) and the same occurred with Vita Mark II and Vita Suprinity (*p*=0.986). For luting cements, the µTBS ranged from 20.68 MPa for Relyx Ultimate followed by 22.00 MPa for Relyx Unicem 2, and 22.20 MPa for Relyx ARC.

Table 3. Overall µTBS mean values (MPa).

Material	N	Mean
Vita Mark II	48	14.14^{c}
Vita Suprinity	48	14.55^{c}
Vita Enamic	48	29.39^{a}
Lava Ultimate	48	28.42^{a}
Relyx ARC	64	22.20^{b}
Relyx Unicem 2	64	22.00^{b}
Relyx Ultimate	64	20.68^{b}

Different superscripted letters indicate statistical difference according to Tukey's test (p>0.05).

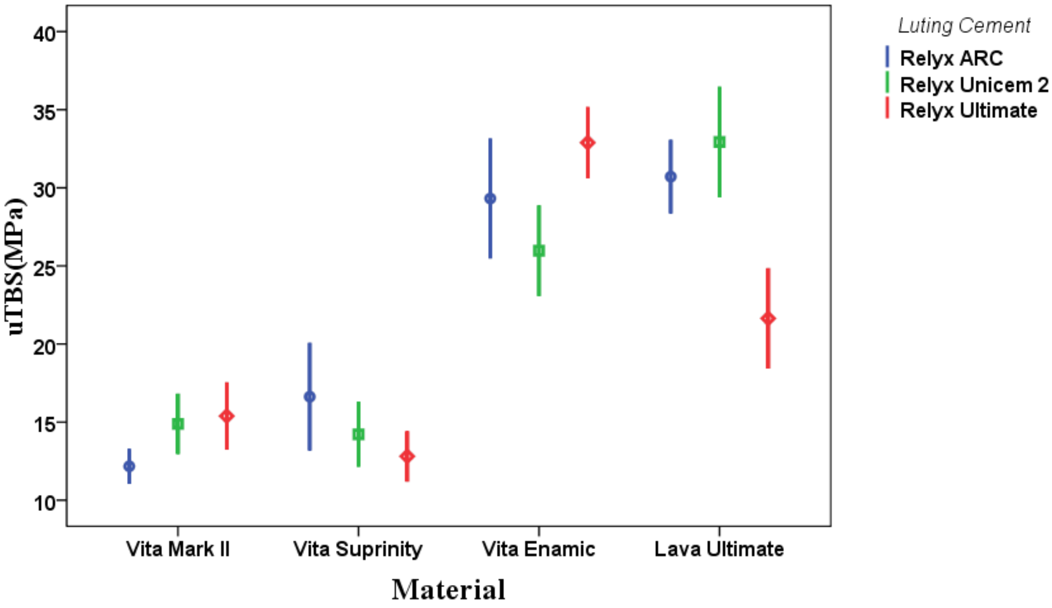
The interaction between restorative materials and luting cements is describe in Table 4 and Graph 1. The µTBS (MPa) ranged from 12.17 when Mark II was luted with Relyx ARC to 32.93 when Lava Ultimate was luted with Relyx Unicem 2. For Vita Mark II, higher values were recorded when Relyx Unicem 2 and Relyx Ultimate were used. However, for Vita Suprinity, all luting cement were equal. For Vita Enamic, higher values were recorded with Relyx Ultimate and Relyx ARC, though Relyx ARC and Relyx Unicem 2 did not differ statistically. The higher values obtained for Lava Ultimate was using Relyx ARC and Relyx Unicem 2.

Table 4. The µTBS mean (MPa) and standard deviations.

	Relyx ARC	**Relyx Unicem 2**	**Relyx Ultimate**
Vita Mark II	12.17 ± 2.08^{Bb}	14.88 ± 3.61^{Cab}	15.38 ± 4.05^{Ca}
Vita Suprinity	16.62 ± 6.46^{Ba}	14.21 ± 3.88^{Ca}	12.81 ± 3.03^{Ca}
Vita Enamic	29.31 ± 7.20^{Aab}	25.97 ± 5.45^{Bb}	32.88 ± 4.29^{Aa}
Lava Ultimate	30.71 ± 4.42^{Aa}	32.93 ± 6.63^{Aa}	21.64 ± 6.02^{Bb}

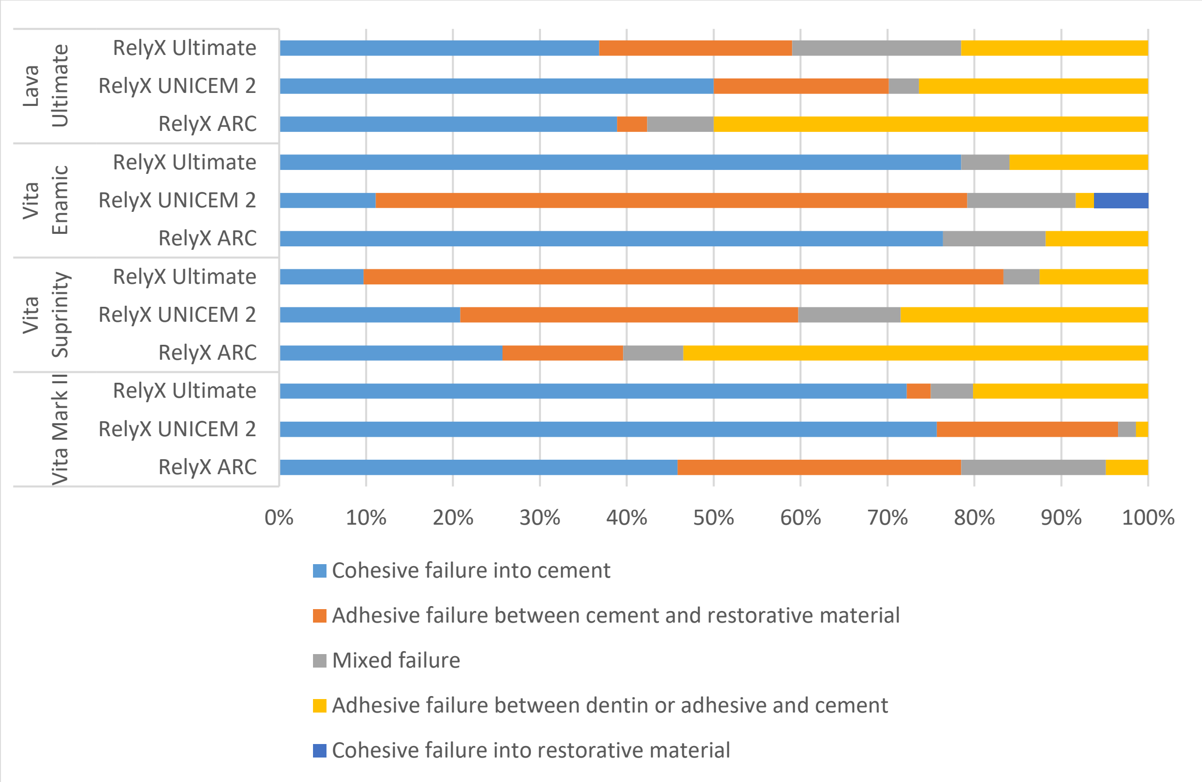
Mean values represented with same superscript uppercase letters (collum) indicate no differences between the factor “Restorative material” and same lowercase letters (line) indicate no differences between the factor “Luting Cement” according to Tukey’s test (p>0.05).

Graph 1. The mean µTBS values (MPa) and standard deviations.



The failure modes are represented in Graph 2. All groups obtained at least three types of failure. For the Vita Mark II groups, the predominant mode failure was the cohesive into cement. However, for the Vita Suprinity groups, when used Relyx ARC the failure mode was adhesive between dentin or adhesive and cement, while for Relyx Unciem 2 and Relyx Ultimate the failure mode was adhesive between cement and restorative material. The main failure mode observed with Enamic and Relyx Unicem 2 was adhesive between cement and restorative material, but for Relyx ARC and Relyx Ultimate was cohesive into cement. The predominant failure mode noted for Lava Ultimate with Relyx Unicem 2 and Relyx Ultimate was cohesive into cement, on the other hand with Relyx ARC was adhesive between dentin or adhesive and cement.

Graph 2. Failure mode analysis.



4 DISCUSSION

Recent improvements in CAD/CAM technology and adhesive dentistry in association to patients demand for esthetic treatment allowed the development of novel materials. The aim of the present study was evaluate the interaction of three different luting cements to bond to novel CAD/CAM restorative and dentin. The results of the present study state that there was difference in µTBS between restorative materials, rejecting the first null hypothesis (p=0.001). The second null hypothesis was accepted (p=0.176), i.e. there was no difference in µTBS between luting cements. There was interaction in µTBS between restorative materials and luting cements, rejecting the third null hyphotesis (p=0.001).

In this study two vitreous ceramics (Vita Mark II and Vita Suprinity), one hybrid material (Vita Enamic), and one nanoceramic (Lava Ultimate) were evaluated. Overall, the hybrid material and the nanoceramic obtained the higher µTBS values, that might be explained by the differences in elastic modulus[24]. In previous studies the elastic modulus of Vita Enamic and Lava Ultimate was respectively 30.1 GPa[25] and 12.8 GPa[4], similar from dentin[5] (16-20.3 GPa)[7] and lower than Vita Mark II (57.2 GPa)[26] and Vita Suprinity (70.44 GPa)[11]. Van Noort[27] et al. proved when the higher elastic modulus of the material, higher is the stress generated in the bond interface. Besides that, these materials also have similar elastic modulus to luting cements[28]. Such characteristics guarantee these materials a good ability to distribute a uniform stress through dental structure and ensure a higher bond strength value than ceramics[24].

In an attempt to improve bonding between luting cements and restorative material, various surface treatments that facilitate chemical and micromechanical retention have been suggested. Increasing the surface energy of the composite by sandblasting, followed by silanization has been recommended as a predictable means to ensure retention between the luting cement and restorative composite[29-31]. In this study, in addition to sandblasting and application of silane coupling agent, all restorations were milled simulating the clinical practice and this factor may be reason for the higher µTBS values for hybrid material and resin nanoceramic. Furthermore, silane coupling agent also contribute for microtensile bond strength, it is an adhesion promoter between organic (methacrylate monomers of the resin cement matrix) and inorganic (fillers of the indirect composite) surface and create a chemical bond between CAD/CAM resin blocks and luting cements[30,32-33]. The failure modes between Vita Enamic with Relyx Unicem 2 demonstrated a higher percentage of adhesive failures between luting cement and restorative material, this implies the need to find alternative surface treatment protocol to stabilize this bond.

The adhesion of ceramic materials may be enhanced by increasing the surface energy and it can improve the wettability on the surface of the luting cement to bond. For this, internal surface etching with hydrofluoric acid will dissolve glassy phase (matrix and crystals) and will create a reactive area promoting resin infiltration. Besides that, when the ceramic is etched, hydroxyl groups are exposed and this groups allow chemical interaction with silane coupling agent[34-36]. The current study used the etching time recommended by the manufacture, Ramakrishnaiah[18] et al. evaluated an extended etching time of Vita Mark II and Vita Suprinity, and showed that the surface roughness and also wettability of silica based ceramics increases. It suggested that extended etching time could increase the µTBS values of these ceramics. When Vita Suprinity was luted with Relyx Unicem 2 or Relyx Ultimate a higher percentage of adhesive failures between luting cement and restorative material was observed. It indicated the etching protocol to Vita Suprinity must be developed. Al-Thagafi[12] et al. evaluated different etching protocols on zirconia-reinforced lithium silicate ceramic and obtained the higher values for tribochemical silica coating with a silane coat.

The present study evaluated one separate bottle of silane coupling agent and one universal adhesive, that contains silane, HEMA, MDP and BisGMA in its composition, into one-bottle solution[37-38]. The presence of silane and MDP monomer in the universal adhesive showed similar µTBS values from those obtained with the separate bottle of silane. It suggests that the multimode adhesive Scotchbond Universal can be used with different resin cements and substrates, reducing the steps of luting strategy. In disagreement with this study, previously studies reported that separate silane step perform better than silane-containing universal adhesive[39], especially in long-term storage[37] and thermocycling[38,40] due the hydrolysis of silane coupling agents by the acidic monomers presents in the multimode adhesive[41].

The success of CAD/CAM restorations depends largely on the bonding system to ensure an effective and stable bond between the restorative material and dental substrates[6,24], however, in present study, there was no significate difference between tested cements. Different bond strategies were applied total etch, self-etch, and self-adhesive. This study demonstrated that simplified cements did not differ from those sensitive protocols. Therefore, these cements should be used in clinical practice reducing clinical steps and simplifying luting strategies.

In present study, one system was evaluated varying three different resin cement systems to novel CAD/CAM restorative materials in dentin, which makes it a most reliable study, and there were no statistically differences between luting cements (p=0.176). Suzuki[42] et al. tested Relyx Unicem and Relyx ARC at µTBS and also recorded no significant differences. Hikita[17] et al. evaluated the same strategy and concluded an equal bond strength to dentin. In both studies the restorative material used was a direct resin composite with lower elastic modulus in comparison to all tested materials.

Previous studies[19-20,30,43] recorded different data from the present findings, the µTBS values found were higher for total-each than self-adhesive resin cement. These studies used similar material, once again with direct composite resin. The possible explanation for the observed difference between studies is the experimental design, while Frankenberger[44] et al. only evaluated µTBS between luting cement and restorative material, in this study was evaluated the system CAD/CAM material-luting cement-dentin, as well as the difference of etching protocols and the fact that the samples were not milled.

This study showed that using hybrid materials is promising. In addition, these materials do not need the crystallization in a dental furnace after milling, shortening the chair-side procedures. The µTBS was evaluated 24 hours after bonding. The main limitation of this study is not have evaluated the effectiveness the µTBS in a long-term water storage and thermal mechanical conditions.

Therefore, further studies should be conducted to evaluate the mechanical and adhesive properties of these materials in long-term water storage and thermal aging conditions even as clinical follow-ups are recommended.

5 CONCLUSION

Within the limitations of this *in vitro* study, it can be concluded that under the recommended protocol used, a promising bonding performance can be achieved with the novel hybrid CAD/CAM restorative materials to tooth structure and to different types of luting cements.

6 CLINICAL RELEVANCE

Due to innumerous variables widely described in the literature doubts about interactions of luting cement, restorative material, conditioning protocol treatment remains in clinical practice. Clinicians should consider choosing CAD/CAM materials prior to luting strategies. Luting agent did not differ, therefore, simplified step ones are more indicates. Hybrid materials showed a higher immediate bond strength in comparison to vitreous ceramic.

ANEXO A

S I P E S Q

Sistema de Pesquisas da PUCRS

Código SIPESQ: 6660 Porto Alegre, 1 de julho de 2015.

Prezado(a) Pesquisador(a),

A Comissão Científica da FACULDADE DE ODONTOLOGIA da PUCRS apreciou e aprovou o Projeto de Pesquisa "COMPARAÇÃO DA RESISTÊNCIA DE UNIÃO DE DOIS CIMENTOS RESINOSOS A DIFERENTES MATERIAIS UTILIZADOS PARA CAD/CAM" coordenado por EDUARDO GONCALVES MOTA. Caso este projeto necessite apreciação do Comitê de Ética em Pesquisa (CEP) e/ou da Comissão de Ética no Uso de Animais (CEUA), toda a documentação anexa deve ser idêntica à documentação enviada ao CEP/CEUA, juntamente com o Documento Unificado gerado pelo SIPESQ.

Atenciosamente,

Comissão Científica da FACULDADE DE ODONTOLOGIA

ANEXO B

16-Sep-2016

Dear Miss Bellan:

Your manuscript entitled "Interaction of luting cements and CAD/CAM materials: μTBS in dentin" has been successfully submitted online and is presently being given full consideration for publication in the The Journal of the American Dental Association.

We will consider your article for publication with the understanding that
- it has not been published previously;
- it has been submitted solely to JADA;
- each author has fully disclosed any financial, economic or other conflicting interests in products or services described in the article.

Before we can put the manuscript into review, we need copies of the Copyright Transfer and Conflict of Interest forms electronically signed by all authors. Each author will receive a separate email with instructions on how to submit both forms with their electronic signature. As the corresponding author, we ask that you please ensure that each author is aware of this and submits their forms electronically.

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Thank you for submitting your manuscript to The Journal of the American Dental Association.

Respectfully,
The Journal of the American Dental Association Editorial Office

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