MESTRADO INTEGRADO EM MEDICINA DENTÁRIA
Faculdade de Medicina da Universidade de Coimbra

Efeito de diferentes sistemas de cimentação nas forças de adesão a blocos CAD/CAM de resina: estudo piloto

Effect of different luting systems on the microtensile bond strength of CAD/CAM resin blocks: pilot study

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Summary

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Resumo

Objetivo: Avaliar o efeito de cinco sistemas diferentes de cimentação nas forças de adesão a blocos CAD/CAM de resina.

Materiais e métodos: Os cinco blocos Brilliant Crios CAD/CAM [Coltene/Whaledent] foram sequencialmente seccionados com um disco diamantado, numa máquina de corte, em duas metades, sendo posteriormente jateados por um jato de óxido de alumínio e as metades cimentadas uma à outra com os seguintes materiais de cimentação: Brilliant EverGlow® [Coltene/Whaledent], Brilliant EverGlow® [Coltene/Whaledent] com aplicação de ultrassons, Brilliant EverGlow® [Coltene/Whaledent] aquecida, Brilliant EverGlow® Flow [Coltene/Whaledent] e Duo Cem® Sample Trans. Posteriormente, os blocos foram novamente seccionados com uma serra de precisão diamantada e refrigerada de forma a obter bastonetes uniformes (1,38 mm^2), que foram submetidos ao ensaio de microtração (μTBS) à velocidade de 0,5 mm/min (n=20/grupo). As superfícies resultantes da divisão foram examinadas em microscopia óptica para determinar os padrões de fratura. Foi realizada a comparação entre os vários grupos dos dados obtidos nos ensaios de microtração através de One-Way ANOVA, considerando a correção de *Bonferroni* para as análises *post-hoc* ($\alpha=0{,}05$). Foi efetuada a avaliação qualitativa da interface adesiva em todos os grupos através da observação em Microscopia Eletrónica de Varrimento (MEV).

Resultados: Os resultados da microtração mostraram diferentes forças adesivas nos diferentes protocolos de cimentação usados: Grupo 1 (45,48 ± 18,14 MPa); Grupo 2 (42,15 ± 14,90 MPa); Grupo 3 (41,23 ± 15,15 MPa); Grupo 4 (58,38 ± 15,65 MPa); Grupo 5 (81,07 ± 8,75 MPa). Como resultado dos testes post-hoc, verificaram-se diferenças estatisticamente significativas em relação às forças de adesão no grupo 5, Duo Cem®, quando comparado com os restantes grupos. Verificou-se, em todos os grupos, que a fratura adesiva foi predominante. A avaliação qualitativa das amostras por MEV revelou existir uma interface bem agregada e homogénea de cimento-bloco em todos os materiais de cimentação usados.

Conclusões: Dentro das limitações deste estudo, no ensaio de microtração foram encontradas diferenças na força de adesão nos protocolos estudados.

Palavras-chave: “Forças de adesão”, “restaurações indiretas”, “CAD/CAM”, “resina composta”.

Effect of different luting systems on the microtensile bond strength of CAD/CAM resin blocks: pilot study

Abstract

Aim: To evaluate the effect of different luting materials on the microtensile bond strength (µTBS) of CAD/CAM resin blocks.

Materials and methods: Five Brilliant Crios CAD/CAM [Coltene/Whaledent] blocks were sequentially sectioned in a diamond disk cutting machine into two halves, sandblasted with aluminum oxide jet and each was luted to another according to the cementation protocol: Brilliant EverGlow® [Coltene/Whaledent], Brilliant EverGlow® [Coltene/Whaledent] with ultrasound application, Brilliant EverGlow® [Coltene/Whaledent] heated, Brilliant EverGlow® Flow [Coltene/Whaledent] and Duo Cem® Sample Trans. Afterwards the blocks were sectioned using an automatic precision water-cooled diamond saw to obtain uniform sticks (1,38 mm^2) that were then submitted to microtensile test (µTBS) at 5 mm/min speed (n=20 per group). The surfaces were examined with optical microscopy to determine the fracture patterns. The resulting data of the microtensile tests was analyzed using One-Way ANOVA considering *Bonferroni* test for post- hoc tests (α=0,05). The qualitative bonding interface Scanning Electron Microscope (SEM) was also evaluated for each group.

Results: The microtensile test results showed different adhesive forces according to the cementation protocol: Group 1 (45,48 ± 18,14 MPa); Group 2 (42,15 ± 14,90 MPa); Group 3 (41,23 ± 15,15 MPa); Group 4 (58,38 ± 15,65 MPa); Group 5 (81,07± 8,75 MPa). According to the *post-hoc* tests, statistically significant differences in bond strength were found in group 5 (Duo Cem®) comparing to other groups. It was found in all groups that the adhesive fracture was predominant type. The qualitative evaluation of the samples by SEM revealed a tight and homogeneous cement-block interface for all the luting materials.

Conclusions: Within the limitations of this study, differences between the microtensile bond strength were found in relation to the protocol studied.

Keywords: “Microtensile bond strengths”, “indirect restorations”, “CAD/CAM”, “resin composite”.

Abbreviations

TEGDMA - Triethylenglycol dimethacrylate
Bis-EMA - Ethoxylated bisphenol-adiglycidyl methacrylate
UDMA - Urethane dimethacrylate
10-MDP - 10-methacryloyloxydecyl dihydrogen phosphate
HEMA - 2-hydroxyethyl methacrylate
Bis-GMA - Bisphenol A-diglycidyl methacryl
SB - Sandblasting with 50 µm Al_2O_3
UNI - Universal adhesive (One Coat 7 Universal®)
BEG - Brilliant EverGlow®
BEG+US - Brilliant EverGlow® with ultrasound
BEG+H - Brilliant EverGlow® heated
BEGF - Brilliant EverGlow® Flow
DUO CEM - Duo Cem® Trans

Effect of different luting systems on the microtensile bond strength of CAD/CAM resin blocks: pilot study

Introduction

Computer-aided design and computer-aided manufacturing (CAD/CAM) composites are one of the fastest growing stranding in the field of restorative materials, competing with glass-ceramics for single-unit restorations. (1) Digital systems allow the whole process to be made in just one appointment, meaning that it is possible to make an optical impression, the restoration design on the computer and mill out of CAD/CAM block for later bonding. (2)

Indirect techniques are advised for large cavities, usually involving one or more cuspids and proximal surfaces. This kind technique requires materials with better mechanical properties, such as resistance to fracture, which is one of the first causes of failure of direct composites, particularly with larger restorations. (2)

A recently published work classified the CAD/CAM composite blocks based on their microstructure: dispersed filler (DF) and polymer-infiltrated ceramic network (PICN). (1,3) The difference between them is the way dimethacrylates such as urethane dimethacrylate (UDMA) and triethylene glycol dimethacrylate (TEGMA) are incorporated in the matrix of the CAD/CAM block. Their incorporation in DF is achieved by mixing them in the matrix and polymerizing under high temperature, while in the PICN they are secondarily infiltrated and polymerized under high temperature and high pressure. (4,5)

In comparison to ceramic, these composite blocks are notable for their better machinability, higher resilience, lower elastic modulus, hardness, and brittleness. (1,3,5) They are also cheaper, exhibit a higher damage tolerance, a lower tendency to marginal chipping and smoother milled margins. Therefore, they are able to be milled to a reduced thickness in comparison with ceramics. CAD-CAM composite blocks have a high degree of polymerization and increased degree of conversion (up to 96%). This increased degree of conversion overcomes some disadvantages related with direct restorations, such as the decrease in the presence of flaws and pores, increasing their homogeneity. However, the high conversion rate leads to a decrease in the potential of chemical bonding as the amount of free double bonds of carbon decreases. (1,3,5,7).

Because of these characteristics it is necessary to carry out a pre-treatment on the restoration surface. In order to achieve this, researchers advocate that composite CAD/CAM blocks should be sandblasted in order to increase the roughness of their surface, promoting an interface with higher micromechanical adhesion, both to the adhesive and to the cement. However, sandblasting should be performed with reduced pressure to avoid the possibility of subsurface cracks formation. (8) The International Academy for Adhesive Dentistry (IAAD) recommends pretreatment for CAD/CAM composite resins with either air abrasion with 50 μm aluminum oxide or 30 μm silicon oxide at a pressure of 2 bar (0.2MPa), which is lower than the pressure commonly recommended for ceramic and metal restorations. (4) Yoshihara *et al.* evaluated the effects of sandblasting with 50μm aluminum oxide at an air pressure of 0.2 MPa on the various CAD/CAM resin composites (Cerasmart, GC; Katana Avencia, Kuraray Noritake; KZR – CAD HR, Yamakin; Lava Ultimate, 3M ESPE; Shofu Block HC, Shofu) and noticed that sandblasting caused microfractures of 1 to 10 μm in the surface of the composite block. The author concluded that despite of the fact that sandblasting induced an altered surface, the procedure was necessary to improve bond strength. (9) Surface treatment via air-particle abrasion seems to be the best choice for CAD/CAM composite adhesion because the procedure causes surface enlargement, enhancing micro-mechanical retention as well as removing a possible smear layer from grinding or milling procedures. (4)

The bonding interface (the tooth structure and the fitting surface of the restoration) remains a challenge. The loss of adhesion between the restoration and tooth induces microleakage, ultimately resulting in secondary caries and inflammatory pulp irritation, so it is crucial to establish a strong, durable bond and an appropriate treatment of the respective surface. (2,3,6,7)

The specific selection of the material contents of the dental restoration as well as the bonding and cement system affects the adhesion properties. There are three ways to achieve adhesion to the resin matrix. Physical adhesion is one of them and depends on the Van der *Walls* forces or hydrogen bonds so resin primers need to contain hydroxyl or amino groups that link to the corresponding groups within the matrix. Another way to create adhesion is to ensure that monomers of the resin primer penetrate the matrix and co-polymerize there, which is known as mechanical adhesion. Finally, chemical adhesion can be obtained by forming new covalent bonds between monomers of the adherent and pending double bonds still available in the substrate. (10,12)

Cementation is a crucial step in the process of ensuring the retention, marginal sealing, and durability of indirect restorations. A desirable dental cement for a successful cementation should fulfill specific biological, physical and mechanical characteristics: stable bonding between the remaining dental structure and restoration, mechanical strength to the forces of mastication, reduced wear, low solubility in oral fluids, low film thickness, biocompatibility with oral tissues, radiopacity, color stability and easy handling. (13) Generally, cements used over the past years are resin-based composites, which can be used either as resin cements or composite resin (which can be applied with ultrasonic application or as thermo-modified resin). Currently, resin luting agents are divided either according to their polymerization reactions into light-curing, chemical-curing and dual-activated cements, or by how their adhesive systems operates: etch-and-rinse, self-etch, and self-adhesive. (3)

Thermo-modified composite resin has shown good physical and mechanical properties, in spite of having a high technical sensitivity, due to the rapid cooling of the resin from the moment it is withdrawn from the heating device. Thus, it becomes useful to thermo-modify the composite when it is intended to use it as a cementing agent in order to obtain reduced viscosity with increased temperature. (14) The ultrasonic application of composite also showed great clinical applicability because the restoration adaptation to the preparation is faster and more precise. (15)

Adhesion of indirect restorations is one of the main factors that contributes to their clinical behavior and longevity (10), making the restauration more susceptible to bonding failure if the bonding protocol is not strictly followed. (16)

The aim of this study is to evaluate the microtensile bond strength (μTBS) and the qualitative bonding interface by scanning electron microscopy after using five different types of adhesive luting materials to a CAD/CAM composite resin, Brilliant Crios® Coltene.

There are two null hypotheses in this study:

- (H0) There are no differences in the microtensile bond strength among the different luting materials.
- (H1) There are no differences in the micromorphology of the bonding interface produced by the different adhesive luting materials.

Keywords: “Microtensile bond strengths”, “indirect restorations”, “CAD/CAM”, “resin composite”

Material and Methods

2.1 Mechanical and Chemical surface treatment

This study evaluated the microtensile bond strength of five different luting materials and protocols to a composite block, Brilliant Crios® Coltene CAD/CAM (LOT H00414, 2019/01) (table I). Five blocks were used after being transversely sectioned in two halves with a diamond disk-cutting machine (Accutom 5, Struers, Ballerup, Denmark) at a speed of 1000 rpm at 0,100 mm/s. Both disposable surfaces were sequentially polished with a 320 and 600 grit silicon-carbide (SiC) (WSFlex 16®, Hermes Schleifmittel GmbH, Hamburg, Germany) abrasive paper for 60 seconds under running water. Afterwards, the surfaces were sandblasted with 50 µm aluminum oxide particles (Airsonic® mini sandblaster, Hager Werken) at a 10 mm distance by attaching a gutta-percha cone to the jet tip. Six linear applications were made on each surface of the sample, without repetition. Subsequently, samples were washed with distilled water followed by ultrasonic vibration (BioSonic® UC 125, Coltene) in 96% alcohol for 2 minutes (L.996P067 2022/07, 96%) and dried with absorbent paper.

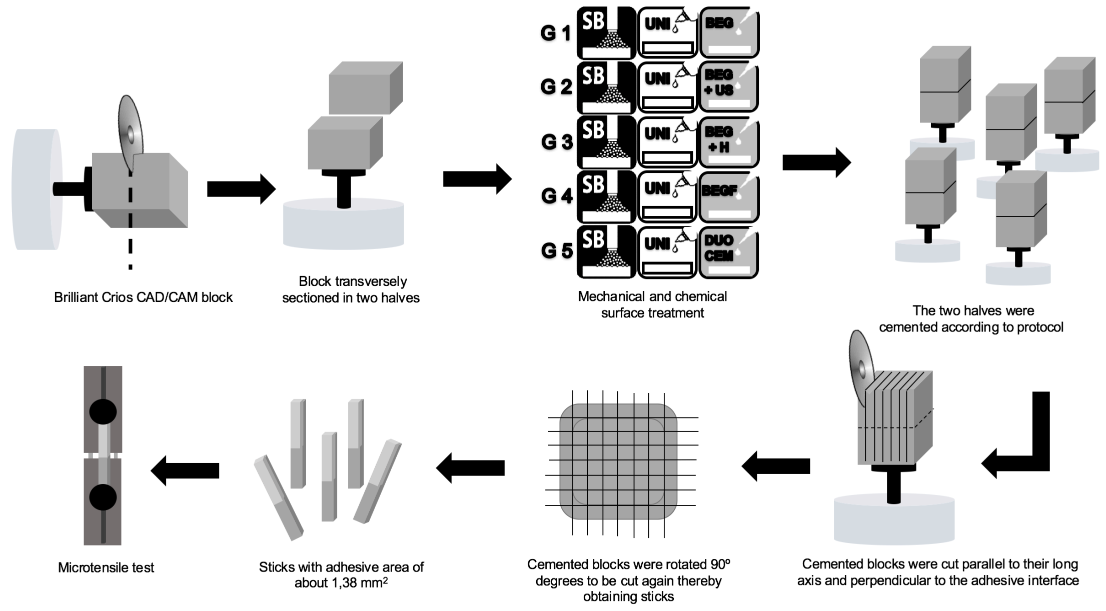

Fig. 1. Schematic presentation explaning the study set. SB: Sandblasting with 50 µm Al_2O_3; UNI: Universal adhesive (One Coat 7 Universal®); BEG: Brilliant EverGlow®; BEG+US: Brilliant EverGlow® with ultrasound; BEG+H: Brilliant EverGlow® heated; BEGF: Brilliant EverGlow® Flow; DUO CEM: Duo Cem® Trans.

Table I. Application procedure of the different mechanical and chemical surface treatment.

	Product Name (manufacturer)	Application procedure	Procedure after treatment
Mechanical surface treatment	SiC abrasive paper (WSFlex 16®,Hermes Schleifmittel GmbH)	Polished with a 320 and 600 grit for 60 seconds under running water	Specimens were cleaned in distilled water, ultrasonically vibrated in 96% alcohol for 2 minutes and dried with absorbent paper
	Alumine oxide (Airsonic® mini sandblaster, Hager Werken)	Sandblasting to the surface at a distance of 10 mm	
Chemical surface treatment	One Coat 7 Universal® (Coltene)	Actively applied with a microbrush for 20 seconds	Air dried for 5 seconds for evaporation of the solvent
	Brilliant EverGlow® (Coltene) with ultrasonic vibration (Dentsurg Pro®, CVDentus, São José dos Campos, Brazil)	Luting material were homogeneous distributed and constant load was applied for 20 seconds. Application of ultrasound during the first phase of polymerization, for 20 seconds, on each face	Speciments were light-cured for 20 seconds with load and another 20 seconds without the effect of the load on each face. The blocks were stored in distilled water at 37° C for 24 hours prior to microspecimens preparation
	Brilliant EverGlow® heated (Coltene)	Placed inside the oven at 50 °C and homogeneous distributed with a constant load application for 20 seconds	
	Brilliant EverGlow® (Coltene)	Luting material was homogeneous distributed and constant load was applied for 20 seconds	
	Brilliant EverGlow® Flow (Coltene)		
	Duo Cem® (Coltene)		

The blocks were divided into five groups for determination of bond strength for all combinations of bonding agents and resin cements on the basis of the microtensile bond strength method (μTBS). This procedure was performed on one another, according to one of five protocols (table I):

I. One Coat 7 Universal® + Brilliant EverGlow® A2/B2 (BEG);
II. One Coat 7 Universal® + Brilliant EverGlow® A2/B2 with ultrasound (BEG+US);
III. One Coat 7 Universal® + Brilliant EverGlow® A2/B2 heated (BEG + H);
IV. One Coat 7 Universal® + Brilliant EverGlow® Flow A3/D3 (BEGF);
V. One Coat 7 Universal® + One coat 7.0 activator® + Duo Cem® Trans (DUO CEM).

The adhesive system (One Coat 7 Universal® LOT H14305, 2018/08) was actively applied with a microbrush for 20 seconds, air dried for 5 seconds and according to the protocol established for each group the luting material were homogeneous distributed. A constant load was applied (3 Newtons (N)) for 20 seconds and all groups were light-cured (Bluephase®, "Low" mode, IvoclarVivadent, Schaan, Liechtenstein) for 20 seconds with load and another 20 seconds without the effect of the load on each face. In group 2, Brilliant EverGlow® (BEG) A2 / B2 was applied with ultrasonic vibration (Dentsurg Pro®, CVDentus, São José dos Campos, Brazil) during the first phase of polymerization, for 20 seconds, on each face and in group 3, Brilliant EverGlow® (BEG) A2 / B2 was heated inside an oven (Ease-it ™, Ronvig) at 50 °C for one hour before application of the adhesive system. Finally, the adhesive (One Coat 7 Universal® LOT H14305, 2018/08) in group 5 was mixed with the activator Blend (One Coat 7.0 activator® LOT H13425, 2018/07) 30 seconds prior to is application. The blocks were stored in distilled water at 37° C for 24 hours prior to microspecimen preparation.

2.2 Microtensile Bond Strength Test (μTBS)

Each block was sectioned with a precision cutting machine with a diamond disk with a 0,3 mm thickness (Accutom 5, Struers, Ballerup, Denmark) at a slow-speed of 1000 rpm at 0,100 mm/s under permanent water cooling. The blocks were first cut parallel to their long axis and perpendicular to the adhesive interface, then rotated 90º degrees to be cut again, thereby obtaining sticks with an adhesive area of approximately about 1.38 mm^2. The outer sticks of each block were excluded. Only the internal samples were used, remaining a total of 20 sticks for each study group. Then, all the sticks were measured with a digital caliper (Mitutoyo digital caliper; Japan) for later calculation of the adhesive area.

Microtensile bond strength (μTBS) was conducted with an universal test machine (Autograph®, Model AG-I, Shimadz Corporation, Kyoto, Japan). For that purpose, the ends of each sticks were fixed to the jig (Od04-Plus, Luzerna, SC, Brazil) with cyanoacrylate rubber enhanced superglue gel (CE10Flex®, Ce Chem Limited, Derbyshire, UK). The jig was fixed into the universal machine and stressed under tensile force until failure at a rate of 5 mm/minute providing a moment-free axial force application. The load at failure was recorded in Newtons and microtensile bond strength was calculated according to the following equation: $\mu TBS = F/A = N/mm^2 = MPa$, where F is the load at fracture (N) and A is the bond area (mm^2).

2.3 Failure types analysis

The mode of failure was analysed under an optical microscope (Leica CLS 150 MR, Switzerland) with a x35 magnification. The fracture pattern was classified as follow: (A) adhesive at the bonding interface; (CC) cohesive in the CAD/CAM block; (CL) cohesive in the luting composite; (M) mixed, both cohesive in the luting composite and CAD/CAM block.

2.4 Scanning electron microscopy (SEM)

Adhesive interface evaluation of each group was conducted with scanning electron microscope (SEM) evaluation. Two samples of each group were polished, rinsed with an ascending series of ethanol (50, 75, 90, 100%) for 15 minutes per solution and further sonicated in absolute ethanol for the same time to complete dehydration. All samples were positioned in aluminum supports and sputter-coated with gold-palladium (Polaron E-5000 Sputter-Coater, Polaron Equipment Lta, Watford, U.K.) for further observation on a scanning electron microscope (Hitachi S-4100 microscope; Hitachi, Tokyo, Japan) with an accelerating voltage of 25kV, at x250 and x2500 magnifications.

2.5 Statistical analysis

Statistical analysis was performed with the IBM SPSS Statistics 23.0® program (SPSS Inc., Chicago, IL, USA). One-way analysis of variance (ANOVA) was used to compare means of microtensile bond strength data between groups. *Post-hoc* pairwise comparisons were performed using the *Bonferroni* correction. The significance level was set at α=0.05.

Table II. Materials evaluated in the study.

Product Name	Brand	Validity	LOT	Composition
Brilliant Crios® CAD/CAM	Coltene/Whaledent, Langenau, Germany	2019/01	H00414	Cross-linked methacrylates (Bis-GMA, BIS-EMA, TEGMA), 71 wt% barium glass and silica particles
Brilliant EverGlow® (BEG) A2/B2	Coltene/Whaledent, Langenau, Germany	2018/08	H15193	Bis-GMA,TEGDMA, Bis-EMA, prepolymerized particles containing glass and nano-silica, agregated and non-aggregated coloidal silica and barium glass
Brilliant EverGlow® Flow (BEGF) A3/D3	Coltene/Whaledent, Langenau, Germany	2019/08/31	H33890	Methacrylates, barium glass, silinized amorphous hydrophobic silica
Duo Cem® Sample Trans	Coltene/Whaledent, Langenau, Germany	2018/05	H01432	Bis-EMA, Bis-GMA, TEGMA, barium glass salinized, amorphous silicic
One Coat 7.0 activator®	Coltene/Whaledent, Langenau, Germany	2018/07	H13425	Bis-GMA, TEGMA, UDMA, fluoride, barium glass, amorphous silicic (68 wt%, 0,1-5mm), etanol, water, activator
One Coat 7 Universal®	Coltene/Whaledent, Langenau, Germany	2018/08	H14305	HEMA, MMA-modified polyacrylyic acid, UDMA, amorphous silicic, 10-MDP, etanol, water, ph=2,8

Abbreviations: TEGDMA: triethylenglycol dimethacrylate; Bis-EMA: ethoxylated bisphenol-Adiglycidyl methacrylate; UDMA: urethane dimethacrylate; 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate; Bis-GMA: bisphenol-A-diglycidyl methacryl.

Results

3.1 Microtensile Bond Strength Test Results (μTBS)

A total of 100 specimens were available for microtensile testing. Descriptive statistics, the number of tested specimens and μTBS results are depicted in figure 2 and table III.

Table III. Descriptive statistics of the five groups tested. Min: lower strength value of adhesion; Max: higher value of adhesion strength; SW (Shapiro-Wilk), p>0.05.

Group	n	Mean (Mpa)	Std. Deviation	Minimum (Mpa)	Maximum (Mpa)	SW (p>0.05)
BEG	20	45,48	18,14	15,00	76,68	0,38
BEG + US	20	42,15	14,90	12,88	72,39	0,43
BEG + H	20	41,23	15,15	20,97	72,65	0,30
BEGF	20	58,38	15,65	34,11	91,06	0,67
DUO CEM	20	81,07	8,75	63,37	97,31	0,80

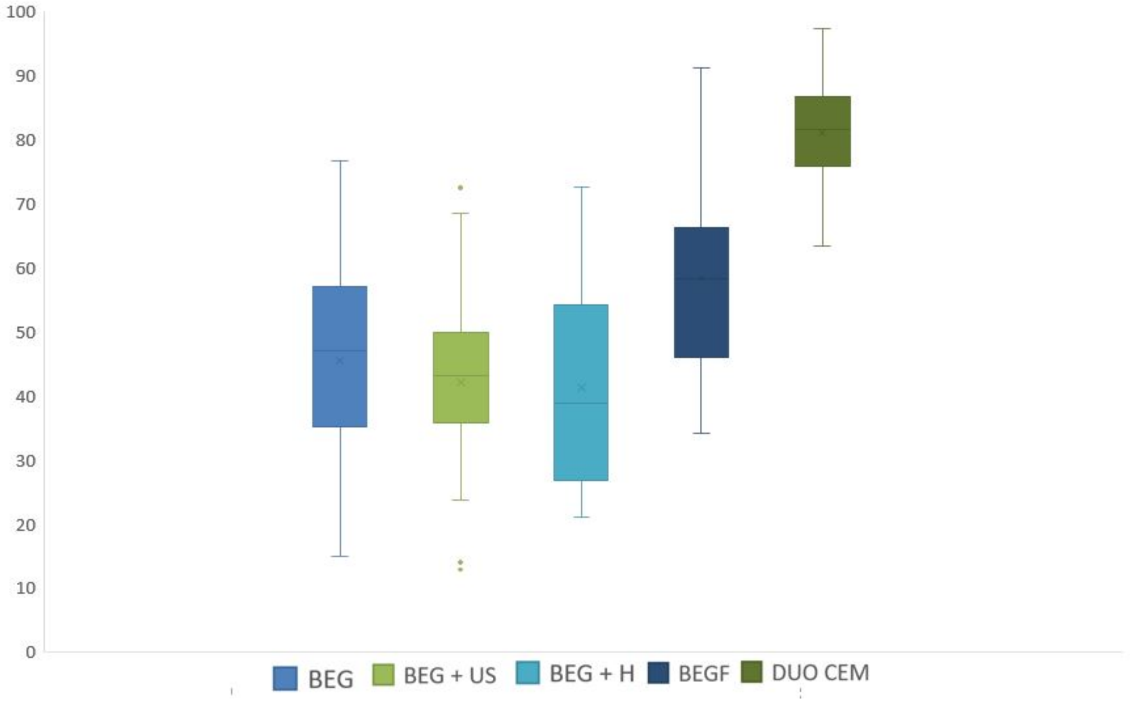

Fig. 2. Boxplots of the TBS results. The box represents the spreading of the data between the first and third quartile. The central horizontal line and the “x” represent the median and mean, respectively. The whiskers extend to the minimum and maximum values measured, with exception of the outliers that are represented with dots (•).

The Shapiro-Wilk test revealed that all groups respected normality because they presented p values higher than 0,05. It was also verified the homogeneity of variances (Levène test= 2,02, p>0.05). One-way ANOVA revealed statistically significant differences amongst groups (F(4,99)= 25,6, p<0.01). Pairwise comparisons between groups indicated significant differences among the µTBS mean values of group 5 (DUO CEM) and in all other groups, which recorded the highest bond strength values. Also, group 4 (BEGF) was statistically different from all except from BEG (G1). By ordering the tested groups according to microtensile values it was found that G5> G4> G1> G2> G3. Multiple comparisons are summarized in table IV.

Table IV. Table of multiple comparisons between groups.

					95% Confidence Interval	
		Mean Difference	Std. Error	p	Lower Bound	Upper Bound
G1	G2	3,34	4,69	1,000	-10,15	16,83
	G3	4,26	4,69	1,000	-9,24	17,75
	G4	-12,90	4,69	0,072	-26,39	0,59
	G5	-35.59	4,69	<0.05*	-49,08	-22,10
G2	G1	-3,34	4,69	1,000	-16,83	10,15
	G3	0,92	4,69	1,000	-12,57	14,41
	G4	-16.24	4,69	<0.05*	-29,73	-2,74
	G5	-38.93	4,69	<0.05*	-52,42	-25,43
G3	G1	-4,26	4,69	1,000	-17,75	9,24
	G2	-0,92	4,69	1,000	-14,41	12,57
	G4	-17.15	4,69	<0.05*	-30,65	-3,66
	G5	-39.84	4,69	<0.05*	-53,34	-26,35
G4	G1	12,90	4,69	0,072	-0,59	26,39
	G2	16.24	4,69	<0.05*	2,74	29,73
	G3	17.15	4,69	<0.05*	3,66	30,65
	G5	-22.69	4,69	<0.05*	-36,18	-9,20
G5	G1	35.59	4,69	<0.05*	22,10	49,08
	G2	38.93	4,69	<0.05*	25,43	52,42
	G3	39.84	4,69	<0.05*	26,35	53,34
	G4	22.69	4,69	<0.05*	9,20	36,18

*Results with statistically significant differences between the two groups compared.

According to the results, statistically significant differences between the different adhesive strategies occurred and the first null hypothesis should be rejected.

3.2 Failure types analysis results

The failure pattern frequency and distribution can be analysed in table V and figure 3. For all groups, failure was predominantly adhesive, although for group 2, group 3 and group 4 a lower percentage of cohesive failure in the CAD/CAM block or in the luting composite occurred.

Table V. Distribution of fracture patterns by groups. Absolute number of samples (percentage).

Group	BEG	BEG+US	BEG+H	BEGF	Duo Cem
Adhesive	16 (80)	15 (75)	14 (70)	11 (55)	17 (85)
Mixed Cohesive	0(0)	1 (5)	2 (10)	2 (10)	1 (5)
Cohesive in the CAD/CAM	0(0)	3 (15)	2 (10)	2 (10)	0(0)
Cohesive in the luting composite	4 (20)	2 (10)	2 (10)	5 (25)	2 (10)

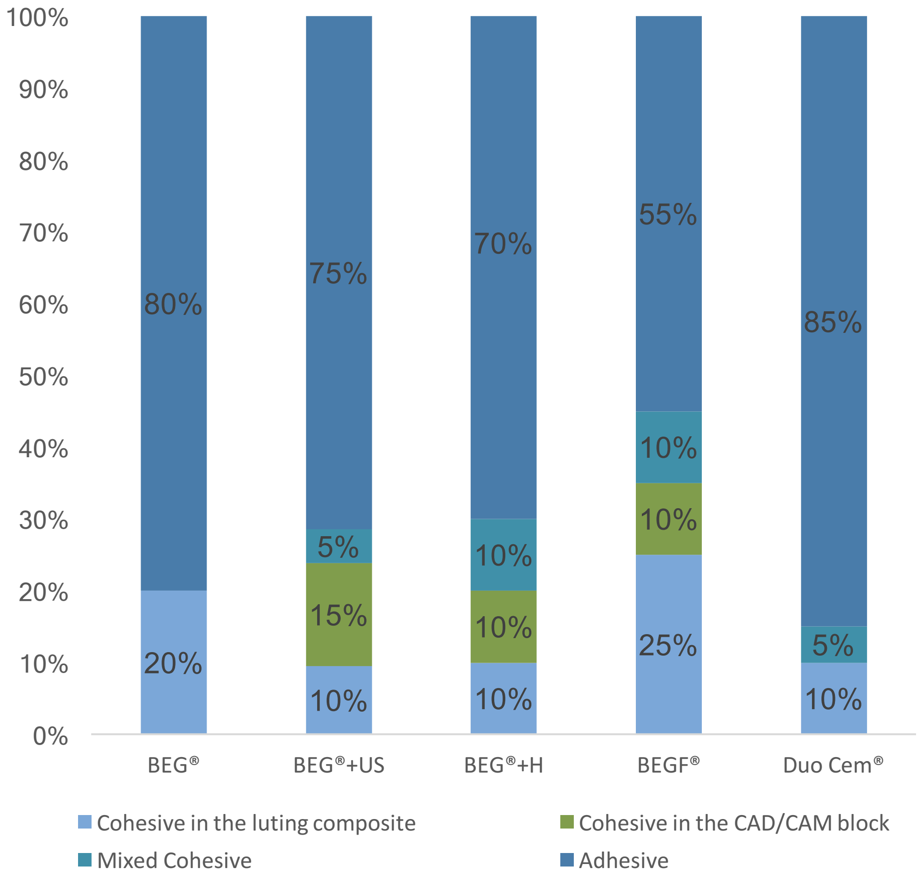

Fig. 3. Failure type results after tensile bond strength test.

3.2 Scanning electron microscopy results

The photomicrographs obtained by SEM with a beam acceleration of 25.0 kV, at 250x and 2500x magnifications, were qualitatively analyzed. A representative photomicrograph of each group can be seen in Figs. 4 to 8, with magnification of 250x and in Figs. 9 to 13 with magnification of 2500x.

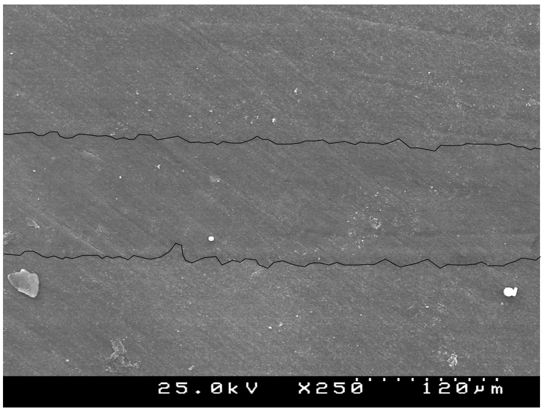

Fig. 4. One Coat 7 Universal® + Brilliant EverGlow® A2/B2 (BEG).

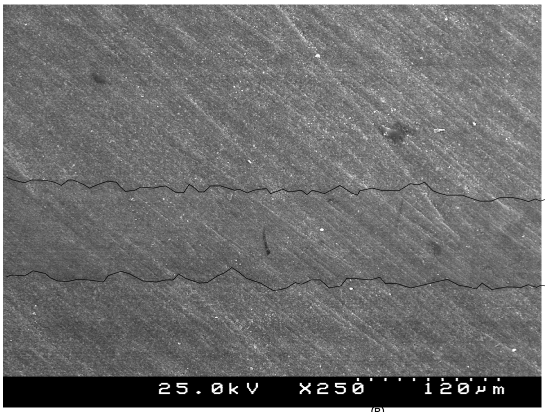

Fig. 5. One Coat 7 Universal® + Brilliant EverGlow® A2/B2 with ultrasound (BEG+US).

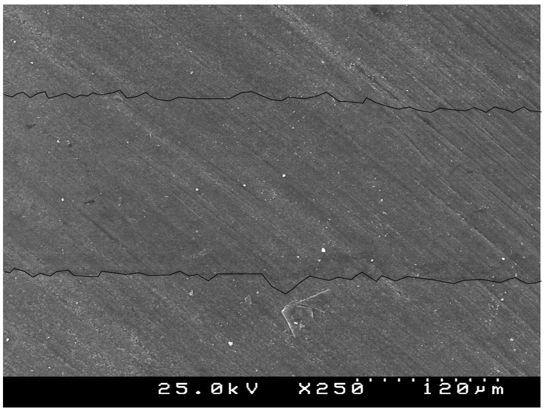

Fig. 6. One Coat 7 Universal® + Brilliant EverGlow® A2/B2 heated (BEG + H).

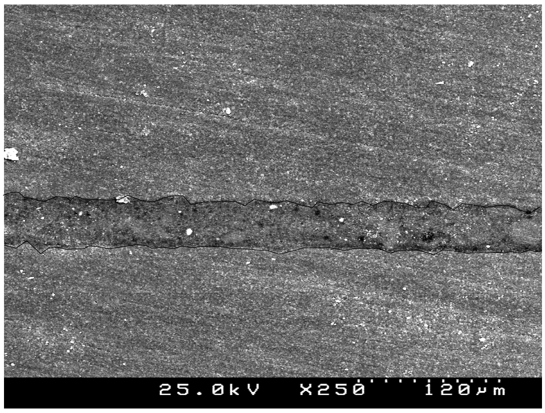

Fig. 7. One Coat 7 Universal® + Brilliant EverGlow® Flow A3/D3 (BEGF).

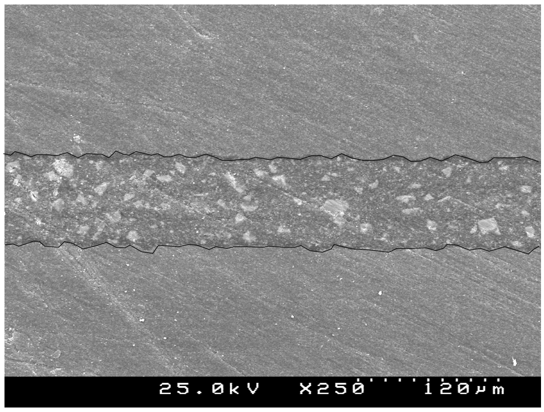

Fig. 8. One Coat 7 Universal® + One coat 7.0 activator® + Duo Cem® Trans (DUO CEM).

The bonding interface SEM images revealed a tight cement-block interface for all the luting materials. Group 4 (BEGF) and 5 (DUO CEM) have the thinner cementation line and the adhesive layer is more visible than the other groups. When ultrasounds were applied, a more densely packed and less porous cement layer was observed.

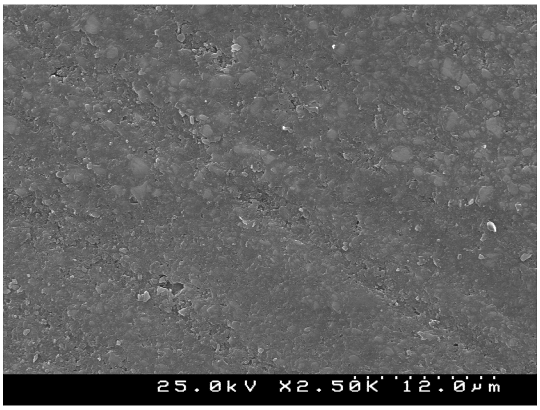

Fig. 9. One Coat 7 Universal® + Brilliant EverGlow® A2/B2 (BEG).

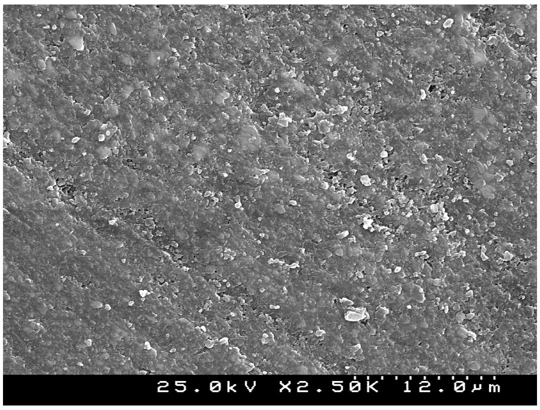

Fig. 10. One Coat 7 Universal® + Brilliant EverGlow® A2/B2 with ultrasound (BEG+US).

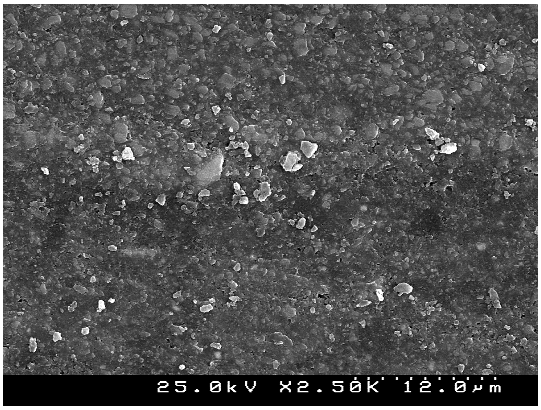

Fig. 12. One Coat 7 Universal® + Brilliant EverGlow® Flow A3/D3 (BEGF).

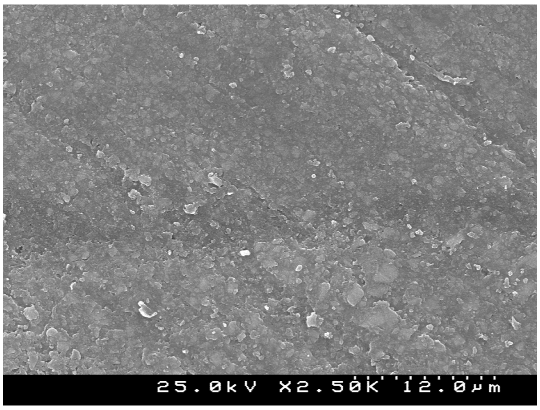

Fig. 11. One Coat 7 Universal® + Brilliant EverGlow® A2/B2 heated (BEG + H).

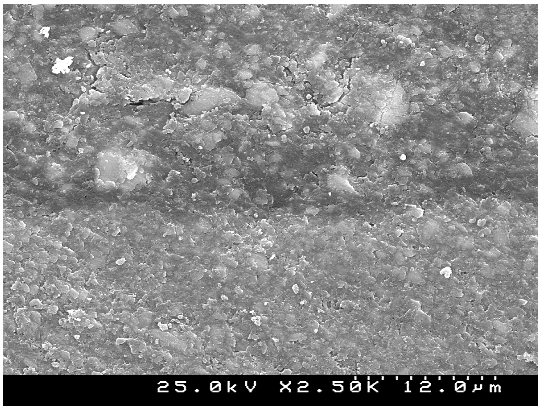

Fig. 13. One Coat 7 Universal® + One coat 7.0 activator® + Duo Cem® Trans (DUO CEM).

Discussion

The adhesion of indirect restorations to luting materials has progressed in the past years. Successful adhesion can be achieved by creating a reliable bond between the internal surface of the restoration and the luting agent. This study focused in understanding the best method of bonding different luting agents to the Brilliant Crios CAD/CAM resin block.

Resin CAD / CAM blocks sandblasting with aluminum oxide at 50 μm was been used as a substrate cleaning technique in several studies, allowing to increase roughness and surface area and, consequently, to improve adhesion. Tekçe *et al.* evaluated the effect of sandblasting powder particles on microtensile bond strength (μTBS) of dual-cure adhesive cement to CAD/CAM blocks. The author concluded that μTBS values of specimens that were sandblasted with 50 μm Al_2O_3 powder were higher than 30 μm SiO_2 and 27 μm Al_2O_3 for all resin blocks study. (17)

Researchers in various studies have proved that performing sandblasting followed by silanization enhances micromechanical and chemical retention and bond strength between CAD/CAM resin composites and the luting material. (9, 18) In a recent study, Reymus *et al.* compared the tensile bond strength of various pretreatments (air abrasion (Al_2O_3, 50 μm, pressure 0.1 MPa) vs. no air abrasion and silane primer (Clearfil Ceramic Primer, Kuraray) vs. a resin primer (One Coat 7 Universal) on different CAD/CAM resin blocks (Brilliant Crios, Cerasmart, Shofu Block HC and Lava Ultimate) luted with DuoCem and found that using One Coat Universal as a resin primer containing MMA (TBS(Brilliant Crios)= 29±12MPa) showed the best results in tensile bond strength and is preferable to the single use of silane primer (TBS(Brilliant Crios)= 12 ±10 MPa) for all groups, but in particularly in the CAD/CAM resin block Brilliant Crios. (7)

Bond strength tests have been used to predict the clinical performance of adhesive interfaces. Although shear bond tests are well established, often produces cohesive bulk fracture of the substrate away from the bonding interface. In this study, microtensile test was been used as it allows a more uniform and homogeneous stress distribution during loading and failure predominantly occurs at the adhesive interface due to the small bonded interfaces. Gilbert *et al.* assessed the bonding properties between a CAD/CAM composite block (Xplus3, Echzell, Germany) and two conventional dual-cured resin cements (RelyX ARC, Variolink II) and a self-adhesive dual-cured resin cement (Clearfil SA Cement) combined with different bonding agents (VP connect, visio.link, Clearfil Ceramic Primer) using three test methods (shear bond strength (SBS), tensile bond strength (TBS) and work of adhesion (WA)). This study showed that higher bonding values can be achieved with conventional dual-cured resin cements for the three *in vitro* methods and the author agreed with the citation of Kelly *et al.,* who mentioned that during shear bond test methods, the tensile stresses are even higher than the shear loads, and therefore, the test design includes failures caused by tensile stresses. (6)

Previous studies focusing on the bonding properties of composite cement to CAD/CAM composite blocks evaluated either in shear bond strength, microshear or microtensile bond strength (μTBS) according to the type of pretreatment, composite cement, or block material (19,20), yet there are no studies that evaluate the different bonding strategies on one resin block with the same block treatment surface. Our study showed, in average, greater bond strength values that in the ones exhibited in the previously mentioned studies for all groups. These higher results can be due to the use of materials from the same manufacturer, which leads to a better chemical compatibility. Reymus *et al.* also assumed that the higher μTBS could be explained by the higher concentration of carbon-carbon double bonds on the surface of the Brilliant Crios CAD/CAM. (7)

In addition to the surface pretreatment, other factors may interfere with the adhesive cementation of indirect restorations, such as the type of resin cement. In the literature, resin cements are described as having a high modulus of elasticity and resistance to bending and compression. Therefore, nowadays resin cements present higher bond strength. (21) Gilbert *et al*. showed that higher bonding values were achieved with conventional resin cements due to the presence of multifunctional dimethacrylates that allow a substantial chemical bonding to PMMA-based CAD/CAM resin. (6) This is in accordance with our study that found statistically higher bond strength when the dual cure resin cement was applied.

Lise *et al*. analyzed the bond strength and surface treatment (sandblasting 27μm Al_2O_3 with a pressure of 0,27 MPa vs 5% hydrofluoric acid etching vs 37% phosphoric acid etching and no treatment vs silane) of a dual-cure, self-adhesive composite cement (G-CEM LinkAce, GC) and a light-cure flowable composite (G-ænial Universal Flo) on two types of CAD/CAM blocks (Cerasmart, GC; Enamic, Vita Zahnfabrik) and concluded that the microtensile bond strength was higher in all the groups luted with the composite cement but not statistically different from the flowable composite (22), similarly to our results.

Groups 1, 2 and 3 that used the Brilliant EverGlow® composite resin and obtained a higher dispersion of the data. This finding can be probably explained by the higher technical sensitivity of these protocols.

Silva *et al*. evaluated the bond strength of self-adhesives resin cements (Rely X Unicem; Maxcem Elite) to dentin with or without ultrasonic application. In this study, a higher µTBS was found in ultrasonic application, but the mean difference was 3 MPa. This results are opposite to our study, although the author stated that it seemed doubtful whether it would be any clinical significance. (23) In other hand, Cantoro *et al.* assessed the influence of the cement manipulation and ultrasound application on the bonding potential of self-adhesive resin cements to dentin by microtensile bond strength testing and found that µTBS increased following ultrasonic vibration, but wasn't statically different in all groups. (15) The authors also concluded that this technique can have clinical significance because the results in the restoration adaptation to the preparation are more precise and faster, requiring a lower cementation load to seat the restoration in comparison with a static load along with a thinner cementation line.

The studies on preheated resins are not consensual. Pappachini *et al*. stated that the bond strength improved by increasing the temperature from 4°C to 23°C. In other hand, Foes-Salgado *et al.* revealed that raising the temperature from 25°C to 68°C had a significant effect on marginal adaptation but did not affect other mechanical properties. (24, 25) In this study, the pre-heated resin did not have any significant effect on the microtensile bond strength. As Davari *et al*. mentioned, this result can be explained by rapid change in composite temperature during application. (26) Preheating composite resin for luting procedures may not improve µTBS, although it could be used to reduce material viscosity and improve restoration setting.

The failure mode evaluated in this study after µTBS testing the luting materials bonded to the resin block showed that the majority of the fractures were through the adhesive interface, which indicates that the stress was concentrated in this area during the tensile test. Flexural strength (FS) is, according to Lise *et al*., closely related to tensile strength, and this might explain why failures propagated more often through the substrate in group 4 (Brilliant EverGlow® Flow). (22) More mixed failures, with large parts of cohesive fractures in the luting composite and in CAD/CAM block, were seen for Brilliant EverGlow® Flow (FS (manufacturer): 96 MPa); this might be a result of lower flexural strength of this material in comparison, for example, with Brilliant EverGlow® (FS (manufacturer): 117 MPa).

Once luted, the bonding interface SEM images revealed a tight cement-block interface for all the luting materials, having infiltrated into the roughened sandblasted surface of the composite blocks. Group 4 (Brilliant EverGlow® Flow) had the thinner cementation line followed by group 5 (Duo Cem®), as it was found in previous studies. (27) This result may come from the composition of Brilliant EverGlow® Flow. Baroundi *et al.* stated that these materials are formed by suspending solid ceramic particles in resin matrixes, resulting in viscoplastic fluids, promoting a viscosity that allows the material to flow easily. (28) As for Duo Cem®, due to its lower inorganic filler composition, the resin cement has a considerably lower viscosity compared to a conventional composite at room temperature, which provides a greater flow and film thickness more appropriate to the cementation. (29)

It should be emphasized that *in vitro* study regimes are unable to simulate all the individual conditions a restoration is exposed to in the oral cavity. To get a more comprehensive picture, it is therefore necessary to collect a large amount of data generated from various studies testing different aspects of the characteristics certain materials possess. Finally, in the present study immediate bond strengths were measured and only a few studies evaluated bond strength after aging and showed that for all materials bond strength decreased with time. (19,22) Nevertheless, long-term validation of *in vitro* tests do not necessarily correspond to the clinical results.

It seems that the selection of the luting agent assumes to be a significant factor when bonding to Brilliant Crios CAD/CAM block. However, this findings must be interpreted with caution and cannot not be generalized to all composite CAD/CAM block materials.

The null hypothesis, (H0) There are no differences in the microtensile bond strength among the different luting materials and (H1) There are no differences in the micromorphology of the bonding interface produced by the different adhesive luting materials are rejected by the findings of this study, since different outcomes for each luting material were clearly observed.

Conclusions

Within the limitations of this *in vitro* study, the following conclusions can be drawn: 1) The bond strength to Brilliant Crios CAD / CAM block is influenced by luting material; 2) Duo Cem® has presented the highest μTBS value (81,07 MPa); 3) The majority of the fractures were through the adhesive interface; 4) SEM revealed a tight cement-block interface for all the luting materials and 5) Brilliant EverGlow® Flow promoted the thinner cementation line.

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