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Study of bond strength, failure probability and reliability of cementing interfaces

Estudo da resistência de união, probabilidade de falha e confiabilidade de interfaces de cimentação

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Bond strength, failure probability and reliability of cementing ceramics and dental interfaces

Resistência de união, probabilidade de falha e confiabilidade de interfaces cerâmicas e dentárias de cimentação

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“Superação é ter a humildade de aprender com o passado, não se conformar com o presente e desafiar o futuro”

(Hugo Bethlem)

RESUMO

Associar a correta seleção da cerâmica odontológica e do protocolo de cimentação adesiva, bem como obter uma união confiável nas interfaces cerâmica/cimento, cimento/substrato dentário, é essencial para longevidade das restaurações indiretas. Com este intuito, este trabalho analisou a resistência de união ao microcisalhamento de substratos cerâmicos e dentários ao cimento resinoso autoadesivo e autopolimerizável Multilink® Speed (Ivoclar Vivadent, Schaan, Liechtenstein), como também a probabilidade de falha, módulo de Weibull e resistência característica destas interfaces por meio da análise estatística de Weibull. Para tanto, 20 (vinte) fatias de cada material cerâmico e dentário, sendo estes: 1) LD – Dissilicato de lítio (IPS e.max CAD®); 2) ZLS - Silicato de lítio reforçado por zircônia - (Celtra Duo®); 3) Y-TZP C - Zircônia tetragonal policristalina estabilizada com ítria na cor Convencional (IPS ZirCAD MO®); 4) Y-TZP T - Zircônia tetragonal policristalina estabilizada com ítria na cor Translúcida (IPS ZirCAD MT®); 5) E + E - esmalte com condicionamento; 6) E – esmalte sem condicionamento e 7) D - Dentina média. As cerâmicas continham dimensões de 2 mm de espessura e os dentes foram obtidos a partir de terceiros molares humanos, todos os espécimes foram preparados e incluídos em tubos de PVC com resina acrílica, seguidas de polimento. O cimento resinoso autoadesivo foi inserido no interior da matriz plástica sobre cada unidade de superfície destes 7 tipos de substratos, resultando em um cilindro com diâmetro de 1,40 mm e altura de 1 mm, seguindo as recomendações do fabricante. Os espécimes foram armazenados em água deionizada a 37° C por 24 h, cada espécime foi submetido ao teste de resistência de união ao microcisalhamento. Os dados do ensaio mecânico foram submetidos à análise estatística por meio dos testes Kruskal-Wallis e Dunn. Análise qualitativa de falha foi realizada por meio do estereomicroscópio (e AxioCam MRc, ZEISS®) e classificadas em adesiva, coesiva e mista, sendo um espécime representativo de cada falha submetido ao microscópio eletrônico de varredura (MEV). A análise estatística de Weibull foi realizada para a obtenção das seguintes métricas: probabilidade de falha, módulo de Weibull (m) (confiabilidade) e resistência característica ($\sigma 0$). No geral, o grupo E + E, obteve maior resistência de união, menor probabilidade de falha, maior confiabilidade e maior resistência característica dentre todos os grupos avaliados, contendo semelhança significativa aos grupos ZLS e LD e diferença estatisticamente significativa aos outros substratos dentários (E e D), contendo a dentina menor mediana dentre todos os substratos examinados. Dentre as cerâmicas, os maiores valores de resistência de união e resistência característica foram obtidos, depois do E + E, em ZLS (Celtra Duo) o qual apresentou alta variabilidade dos dados, o grupo LD (e-max) apresentou maior confiabilidade dentre as cerâmicas analisadas. O modo de falha adesiva foi predominante dentre os tipos de falhas. Com isso, conclui-se que a interface adesiva das vitrocerâmicas apresentaram maior resistência característica, menor probabilidade de falha e maior confiabilidade, sendo similares ao esmalte condicionado seletivamente.

Palavras-Chave: Cerâmica; Materiais Dentários; Adesão; Prótese Dentária; Cimentação.

ABSTRACT

Study of bond strength, failure probability and reliability of cementing interfaces

Associating the correct selection of dental ceramics and the adhesive cementation protocol, as well as obtaining a reliable union at the ceramic/cement, cement/dental substrate interfaces, is essential for the longevity of indirect restorations. This work analyzed the microshear bond strength of ceramic and dental substrates to the Multilink® Speed self-adhesive and self-curing resin cement (Ivoclar Vivadent, Schaan, Liechtenstein), as well as the failure probability, Weibull modulus and characteristic strength of these interfaces through Weibull statistical analysis. For that, 20 (twenty) slices of each ceramic and dental material, as follows: 1) LD – Lithium disilicate (IPS e.max CAD®); 2) ZLS - Zirconia reinforced lithium silicate - (Celtra Duo®); 3) Y-TZP C - Yttria-stabilized polycrystalline tetragonal zirconia in Conventional color (IPS ZirCAD MO®); 4) Y-TZP T - Translucent color yttria-stabilized polycrystalline tetragonal zirconia (IPS ZirCAD MT®); 5) E + E - Enamel Etched; 6) E – unetched Enamel and 7) D – Dentin. The ceramics had dimensions of 2 mm thickness and the teeth were obtained from human third molars, all specimens were prepared and included in PVC tubes with acrylic resin, followed by polishing. The self-adhesive resin cement was inserted inside the plastic matrix on each surface unit of these 7 types of substrates, resulting in a cylinder with a diameter of 1.40 mm and a height of 1 mm, following the manufacturer's recommendations. The specimens were stored in deionized water at 37°C for 24 h, each specimen was subjected to the microshear bond strength test. The mechanical assay data were submitted to statistical analysis using the Kruskal-Wallis and Dunn tests. Qualitative failure analysis was performed using a stereomicroscope (and AxioCam MRc, ZEISS®) and classified as adhesive, cohesive and mixed, with a representative specimen of each failure submitted to a scanning electron microscope (SEM). Weibull statistical analysis was performed to obtain the following metrics: probability of failure, Weibull modulus (m) (reliability) and characteristic strength (σ0). In general, the E + E group had higher bond strength, lower probability of failure, higher reliability, and higher characteristic resistance among all the evaluated groups, containing significant similarity to the ZLS and LD groups and a statistically significant difference to the other dental substrates (E and D), containing the median minor dentin among all substrates examined. Among the ceramics, the highest values of bond strength and characteristic strength were obtained, after E + E, in ZLS (Celtra Duo) which presented high data variability, the LD group (e-max) presented greater reliability among the ceramics. analyzed ceramics. The adhesive failure mode was predominant among the failure types. With this, it is concluded that the adhesive interface of glass ceramics presented higher characteristic strength, lower probability of failure and greater reliability, being similar to selectively etched enamel.

Keywords: Ceramics; Dental Materials; Adhesion; Dental Prosthesis; Cementation.

SUMÁRIO

1. INTRODUÇÃO

As restaurações indiretas por meio das cerâmicas odontológicas estão presentes no cotidiano da prática clínica por suprirem necessidades estéticas, proporcionando o mimetismo das características ópticas dos dentes naturais,[1] aliando a resistência à química e mecânica satisfatórias, levando à longevidade clínica.[2] Quando corretamente indicados, os aspectos relacionados aos sistemas cerâmicos e estudos referentes ao seu comportamento podem fornecer parâmetros para seleção do material de trabalho com base em evidências científicas.[3]

As propriedades das cerâmicas odontológicas dependem da sua composição e microestrutura, as características da fase cristalina de reforço, tais como sua natureza e quantidade, possuem influência direta nas características ópticas e disseminação de trincas[4] as quais podem se originar em defeitos estruturais e/ou de processamento, ditando a resistência mecânica do material.[5] Podem ser classificadas segundo sua composição em: cerâmicas de matriz vítrea (feldspática e cerâmicas reforçadas), cerâmicas policristalinas e, mais recentemente, após a abrangência da definição do termo "porcelain/ceramic", versão de 2013 da American Dental Association,[6] o qual trouxe a definição da cerâmica como compostos refratários predominantemente inorgânicos, abriu precedente para inclusão de novas microestruturas as quais foram incorporadas a esta classificação, a exemplo das cerâmicas de matriz resinosa,[4,7] que são materiais compósitos com propriedades fisico-quimico-mecânicas predominantes da natureza de sua matriz polimérica.[5,8,9]

O método de processamento empregado também influencia na resistência do material, tais métodos consistem em: estratificação e sinterização de pós cerâmicos (convencional), prensagem do lingote cerâmico no interior de molde de revestimento sob altas temperaturas (prensagem ou injeção) ou fresagem dos blocos cerâmicos por sistema CAD/CAM (Computer Aided Design/Computer Aided Manufacturing) (usinagem).[5] Os blocos, dependendo de sua composição, apresentam-se pré-sinterizados ou totalmente sinterizados, contendo sua disseminação impulsionada devido ao progresso da odontologia digital. Além disso, com o advento do desenvolvimento do sistema de Reconstrução Cerâmica ou Chairside Restoration CEREC® (Dentsply Sirona), o qual foi possível a partir do trabalho de Mörmann em 1985[10], alguns sistemas permitem que o desenho, a fresagem do bloco e a cimentação da restauração indireta seja realizada em consulta odontológica única.[10-12] A etapa de cimentação com a formação de duas interfaces (1) a interface substrato dentário/cimento e (2) a interface cimento/substrato restaurador, influenciam no sucesso e longevidade das restaurações indiretas.[13,14]

Os sistemas adesivos e cimentos resinosos estão em constante evolução para o aprimoramento de características, tais como resistência, estética e simplificação da técnica.[15] Faz-se necessário que o profissional siga, com cautela, as recomendações desde o tratamento de superfície tanto da estrutura dentária quanto da superfície interna da restauração cerâmica, até a escolha do sistema de cimentação, visto que cada substrato vai demandar especificidades da técnica.[14,16]

Sendo assim, para as cerâmicas vítreas o padrão ouro para o tratamento de superfície é realizado mediante o seu condicionamento com o ácido fluorídrico, no tempo recomendado pelo fabricante, seguido de aplicação do silano como agente de união do conteúdo vítreo da cerâmica à matriz do cimento resinoso.[13,14] As cerâmicas policristalinas demandam outras etapas para adesão, a literatura sugere diferentes abordagens de tratamento desta superfície na busca da melhoria adesiva da interface das cerâmicas policristalinas e cimento resinoso, tais como o uso de jateamento com óxido de alumínio, aplicação de primer cerâmico ou jateamento com deposição de sílica e aplicação de silano, resultando em uma superfície com radicais livres para reagir com o sistema de cimentação.[17-19]

Para que a adesão ocorra de forma satisfatória e efetiva, idealmente, os cimentos devem apresentar adequada resistência à dissolução no meio oral, união adequada com os diversos tipos de materiais, alta resistência às forças de tensão, boas características de manipulação,[20] mínima espessura de película a fim de não comprometer o assentamento da coroa como também seu desempenho clínico,[21] propriedades antibacterianas, estética compatível com os materiais empregados, e biocompatibilidade com o substrato.[22] No entanto, não há cimentos disponíveis no mercado que consigam englobar todas essas propriedades, sejam eles cimentos convencionais, como o de fosfato de zinco (pioneiro) e cimento de ionômero de vidro, ou cimentos resinosos, acarretando a falta de consenso quanto à predileção clínica de um protocolo de cimentação em detrimento de outro.[17,23]

No tocante aos cimentos resinosos convencionais, são comumente utilizados na prática odontológica para cimentação de restaurações indiretas. Aderem-se ao substrato e à peça cerâmica após tratamento de superfície em associação a um agente de união intermediário, seja ele sistema adesivo ou primer ácido, apresentam como propriedades a resistência à tensão de tração; baixa solubilidade e maior resistência de união, tendo como principal desvantagem a sensibilidade técnica, são indicados para cimentação de PPF com e sem metal, inlays, onlays, facetas e pinos intrarradiculares.[16,17]

Com o objetivo de simplificar a técnica adesiva, os cimentos resinosos autoadesivos, a exemplo do Multilink® Speed, vêm tornando-se uma boa alternativa por dispensar o condicionamento da superfície dentária e pelo não uso de sistemas adesivos na sua interface com a dentina ou material restaurador,[16,24] possuem boas propriedades físicas e adesão ao tecido dental[25] e são indicadas para cimentação de pinos intrarradiculares, inlay, onlays, coroas metalocerâmicas e totalmente cerâmicas sobre dentes e sobre implantes.[26] O modo de união dos cimentos autoadesivos dá-se pela presença de monômeros metacrilatos modificados por ácidos, os quais propiciam a desmineralização superficial e infiltração simultânea do substrato dentário, resultando não só em retenção micromecânica, por meio da desmineralização parcial e incorporação da smear layer na hibridização, formando uma zona de interdifusão entre tecido duro e agente cimentante,[27] como também boa interação química/iônica com o conteúdo mineral (cálcio da hidroxiapatita) por meio da interação dos ácidos, presentes no cimento com o cálcio da hidroxiapatita.[20,24]

Contudo, a ação de condicionamento do esmalte destes monômeros ácidos funcionais não é tão efetiva quanto o condicionamento convencional realizado com ácido fosfórico a 37%, com isso, têm-se proposto na literatura o condicionamento seletivo do esmalte com ácido fosfórico, previamente à aplicação do cimento autoadesivo como forma de fomentar a resistência de união,[28-30] além disso, esses monômeros funcionais ácidos podem retardar o início da polimerização do cimento autoadesivo devido à inativação da geração de radicais livres, o que leva ao comprometimento do processo de conversão de monômeros em polímeros.[17] Outro fator de importância é a neutralização dos monômeros ácidos, a partir da sua interação com o substrato dentário, elevando o pH do meio, o qual torna o cimento mais hidrófobo (a medida em ocorre a reação de polimerização), reduz da sorção de água e a solubilidade. No entanto, na adesão ao substrato não dentário, como resina ou metal, o comprometimento dessa neutralização, por depender apenas da reação de autoneutralização intrínseca do cimento, acarreta monômeros ácidos residuais resultando em elevado pH, polimerização deficiente e redução da resistência de união em longo prazo. [24,16] No entanto, a neutralização do pH vai variar de acordo com o cimento resinoso autoadesivo empregado, pois há alteração na proporção dos componentes nos cimentos autoadesivos disponíveis no mercado o que pode influenciar em seu desempenho de resistência de união.[31,32]

Nesse contexto, este estudo propõe-se analisar a resistência de união das interfaces (1) materiais cerâmicos; (2) esmalte (com e sem condicionamento seletivo prévio) ao cimento resinoso e (3) dentina ao cimento resinoso a fim de possibilitar respostas laboratoriais para embasar a conduta clínica frente às cerâmicas de diferentes composições e necessidades de tratamento de superfície ao cimento resinoso autoadesivo autopolimerizável Multilink® Speed (Ivoclar Vivadent, Schaan, Liechtenstein).

2 ARTICLE

The article presented in this Dissertation was written according to the **Operative Dentistry** instructions and guidelines for article submission.

Glass ceramics behavior like etched enamel on self-etching/curing resin cement interface

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ABSTRACT

This work aimed to analyze the microshear bond strength of ceramic and dental substrates to Multilink® Speed self-adhesive and self-curing resin cement (Ivoclar Vivadent, Schaan, Liechtenstein), as well as the failure probability, Weibull modulus and characteristic strength of these interfaces through Weibull statistical analysis. For that, 20 (twenty) slices of each ceramic and dental material, as follows: 1) LD – Lithium Disilicate (IPS e.max CAD®); 2) ZLS - Zirconia reinforced Lithium Silicate - (Celtra Duo®); 3) Y-TZP C - Yttria-stabilized Polycrystalline Tetragonal Zirconia in Conventional color (IPS ZirCAD MO®); 4) Y-TZP T - Translucent color Yttria-stabilized Polycrystalline Tetragonal Zirconia (IPS ZirCAD MT®); 5) E + E - Enamel with Etching; 6) E – unetched Enamel and 7) D – Dentin. The ceramics had dimensions of 2 mm thickness and the teeth were obtained from human third molars, all specimens were prepared and included in PVC tubes with acrylic resin, followed by polishing. The self-adhesive resin cement was inserted inside the plastic matrix on each surface unit of these 7 types of substrates, resulting in a cylinder with a diameter of 1.40 mm and a height of 1 mm, following the manufacturer's recommendations. The specimens were stored in deionized water at 37°C for 24 h, each specimen was subjected to the microshear bond strength test. The mechanical assay data were submitted to statistical analysis using the Kruskal-Wallis and Dunn tests. Qualitative failure analysis was performed using a stereomicroscope (and AxioCam MRc, ZEISS®) and classified as adhesive, cohesive and mixed, with a representative specimen of each failure submitted to a scanning electron microscope (SEM). Weibull statistical analysis was performed to obtain the following metrics: probability of failure, Weibull modulus (m) (reliability) and characteristic strength ($\sigma 0$). In general, the E + E group had the highest median value, lower probability of failure, greater reliability, and greater characteristic strength among all the evaluated groups, with significant similarity to the ZLS and LD groups and a statistically significant difference to the other dental substrates E and D, the latter had the lowest median among all substrates examined. Among the ceramics, the highest values of bond strength and characteristic strength were obtained in ZLS (Celtra Duo) which presented high data variability, the LD group (e-max) presented greater reliability among the analyzed ceramics. The adhesive failure mode was predominant among the failure types. With this, it is concluded that the adhesive interface of glass ceramics presented higher characteristic strength, lower probability of failure and greater reliability, being similar to selectively etched enamel.

Keywords: Ceramics. Resin Cements. Dental Prosthesis. Cementation.

INTRODUCTION

The clinical success of indirect ceramic restorations is largely linked to adhesive cementation. This step aims to promote the link between the internal surface of the restoration and micromechanical link with the tooth structure, by a resin cement.[1-3] The self-adhesive resin cement was developed to simplify the technique used in the conventional adhesive protocol. In this material, the steps of prior treatment of the tooth surface are eliminated: (1) acid etching; (2) primer, and; (3) bond. This provides less postoperative sensitivity in addition to avoiding problems arising from chemical incompatibility between simplified adhesive systems and chemical or dual polymerization resin cements.[1,4-6]

The adhesive process from ceramic to self-adhesive resin cement normally occurs through the link between the interfaces (1) ceramic/cement and (2) cement/tooth surface (enamel or dentin). Its binding mechanism is dependent on the substrate to which it is being used.[1,7,8] On the ceramic/cement interface, the bond depends on the composition and microstructure of the chosen ceramic.[7] In glass ceramics, etching with hydrofluoric acid is applied for partial surface dissolution of its vitreous content, followed by the application of the silane bonding agent. These steps promote the binding of the cement organic content (methacrylate groups) to the ceramic inorganic portion (silica) by chemical interaction.[9,10] As for polycrystalline ceramics, several surface treatments have been proposed in the literature. In these cases, ceramic primers, aluminum oxide blasting, and silica coating are usually used.[11-13] Self-adhesive self-curing cements do not require adhesive agents and additional primer. These materials promote stable chemical union between the radial OH- of the phosphoric acid group present in cement[7,14-16] and the grain boundary oxides of zirconia such as aluminum, yttrium, and hafnium oxides.[17] In the case of thick ceramics and/or with high opacity, the self-curing cements are more suitable than those of dual-curing. The light activation can stabilize the polymer chains, making it difficult to complete the subsequent reaction by the chemical setting, resulting in a lower degree of conversion.[18,19] Insufficient irradiance can result in a lower monomer conversion than chemical polymerization itself, interfering on the cement final hardness.[20,21]

On the cement substrate/tooth structure interface, micromechanical retention occurs through partial demineralization of hydroxyapatite and simultaneous infiltration of resin monomer, building an interdiffusion zone as a hybridization process.[8] In addition, the chemical/ionic interaction of acidic monomers with mineral content (calcium from hydroxyapatite) occurs. This process contributes to the neutralization of pH by an acid-base reaction which, associated with the chemical interaction between the acidic monomers and the inorganic ions of the cement, potentiates the neutralization of the remaining acid groups.[1,6] The bond strength of self-adhesive self-curing resin cements to dentin is reported to be compatible with those of conventional resin cements.[22,23] However, when applied to enamel, these strength presents lower results.[7,24]

In an attempt to reduce this deficiency, selective acid etching to enamel is applied prior to cementation when using self-adhesive cements, regardless of the type of polymer activation.[3,8,25,26] In order to combine technical simplicity with improved bond strength, the industry has constantly improved self-adhesive self-curing resin cements. However, there is little information available on the detailed composition of these materials, which dictates their behavior at both interfaces in terms of failure probability and reliability. Therefore, this study aimed to evaluate the bond strength of different commercial ceramics, enamel, and dentin to a self-adhesive resin cement as well as the failure probability, Weibull modulus and characteristic strength of these interfaces through Weibull statistical analysis. The hypotheses were that: (1) There is difference in the bond strength between the self-adhesive self-curing cement interface and different types of ceramics and (2) there is a difference in the bond strength of the cement to dental substrate interface and in relation to selective etching in enamel.

MATERIALS AND METHODS

Ethical aspects

This study was submitted and approved by the Ethics and Research Committee of the Bauru School of Dentistry, University of São Paulo (5,040,595). In accordance with ethical rules, 60 human third molars after extraction for therapeutic reasons were selected.

Material Selection

Four dental ceramics with different compositions were evaluated (Table 1). For the surface treatment of ceramics and teeth, hydrofluoric and phosphoric acids were used, respectively, in addition to universal primer (silane) and resin cement, applied according to the manufacturers' instructions and described in Table 2.

Table 1. Ceramic materials' composition according to the manufacturer.

Material	Manufacturer	Groups	Classification	Composition
IPS e.max CAD®	Ivoclar Vivadent, Schaan, Liechtenstein	LD	Lithium Disilicate	57–80% SiO_2 11–19% Li_2O 0–13% K_2O 0–11% P_2O_5 0–8% ZrO_2 0–8% ZnO 0–5% Al_2O_3 0–5% MgO
Celtra Duo®	Dentsply Sirona	ZLS	Zirconia-reinforced Lithium Silicate	SiO_2, P_2O_5, Al_2O_3, Li_2O, K_2O, ZrO_2, CeO_2, Na_2O, Tb_4O_7, V_2O_5, Pr_6O_{11}, Cr, Cu, Fe, Mg, Mn, Si, Zn, Ti, Zr, Al.
IPS ZirCAD MO®	Ivoclar Vivadent, Schaan, Liechtenstein	Y-TZP C	Conventional color; Yttria-stabilized Tetragonal Zirconia Polycrystal	88,0 – 95,5 % ZrO_2 > 4,5 % – ≤ 6,0 % Y_2O_3 ≤ 5,0 % HfO_2 ≤ 1,0 % Al_2O_3 ≤ 1,0 % other oxides

IPS ZirCAD MT®	Ivoclar Vivadent, Schaan, Liechtenstein	Y-TZP T	Translucent color; Yttria-stabilized Tetragonal Zirconia Polycrystal	86,0 – 93,5% ZrO_2 > 6,5% – 8,0% Y_2O_3 ≤ 5,0% HfO_2 1,0% Al_2O_3 ≤ 1,0% other oxides

Table 2. Surface treatment materials' composition according to the manufacturer.

Materials	Manufacturer	Composition
Universal primer	Monobond® N, Ivoclar Vivadent, Schaan, Liechtenstein	Silane methacrylate, phosphoric acid methacrylate and sulfide methacrylate (Alcoholic solution)
Hydrofluoric acid	Condac 5%, FGM, Joinville, SC, Brazil	5% hydrofluoric acid, water, surfactant and dye (gel).
Blasting	Mega-Ox®, OdontoMega, Ribeirão Preto, SP, Brazil.	Aluminum oxide (Al_2O_3),110 µm, 1.5 bar
Phosphoric acid	Condac 37%, FGM, Joinville, SC, Brazil	37% phosphoric acid, dye, deionized water, thickener (gel)
Self-adhesive, self-curing resin cement with light-curing option	Multilink® Speed Ivoclar Vivadent, Schaan, Liechtenstein	Dimethacrylate and acidic monomers matrix; Inorganic particles of barium glass, ytterbium trifluoride, copolymer and silicon dioxide; <1% Initiators, Stabilizers, and Color Pigments.

Specimens Preparation

Ceramic specimen

Ceramic blocks were sectioned into 2 mm thick slices in a cutting machine (Isomet 1000 Low Speed, Buehler, Lake Bluff, IL, USA) with a diamond disc (350 rpm; 15LC diamond nº 11-4254, Buehler) under refrigeration, to obtain the ceramic groups (n=20; LD, ZLS, Y-TZP C, Y-TZP T). All specimens were polished with sequential sanding discs (grain sizes, #800, #1000 and #1200; polishing paper K2000, Exact, Nordestedt, Schleswing-Holstein, Germany) using a polishing machine (Arotec Indústria e Comércio, Cotia, SP, Brazil) to standardize ceramic surfaces.

Subsequently, the polycrystalline ceramics were sintered (P1 Standard program for the conventional sintering of IPS e.max ZirCAD MT Multi/MT/LT/MO, Programat® S1 1600, Ivoclar Vivadent), as well as lithium disilicate and silicate glass ceramics. of lithium with crystallized zirconia (840ºC, 820ºC, respectively, Programat® EP 3000, Ivoclar Vivadent) according to the manufacturer's recommendations. Then, they were fixed on a polyvinyl chloride (PVC) matrix filled with self-curing acrylic resin (JET, Clássico, Artigos Odontológicas, São Paulo, Brazil). The specimens were randomly divided into 4 groups and embedded in acrylic resin. The surfaces were polished with sequential sanding discs (#800 to #1200; K2000 Polishing Paper, Arotec) to remove any acrylic resin that may have coated the samples, washed with deionized water, and dried with oil-free air at a pressure of 40 psi, at a standardized distance of 15 cm at 45°.

Tooth specimen

Sixty human third molars were inspected for integrity with a 10x magnification lens (Leica Zoom 2000 – Leica Microsystems®). The teeth were divided into 3 groups (n=20) for exposure of the middle dentin (D), for enamel with prior selective acid etching (E+E) and for enamel without etching (E), stored in a 0.1% thymol solution.

Root removal and occlusal surface were seccioned on a cutting machine (Isomet 1000, Buehler, LakeBluff, IL, USA), with a diamond disc (Extec XL12205 High Concentration, Extec Corp, Enfield, Connecticut, USA) at 275 rpm under cooling to expose the mid-denin surface (3 to 4 mm from the cusp tip). Then, they were flattened and polished with silicon carbide sandpaper (Extec Corp, CT, USA) #600 and #400 grit in a polisher (Arotec Indústria and Comércio, Cotia, SP, Brazil) for 60 seconds to remove the remaining occlusal enamel. The enamel samples were planed and polished using the same procedure used on the dentin ones. In addition, 37% phosphoric acid (Condac®, FGM, Joinville, SC, Brazil) was used for the enamel group with selective etching.

The teeth were embedded in a polyvinyl chloride (PVC) matrix containing self-curing acrylic resin (JET, Clássico, Artigos Odontológicas, São Paulo, Brazil) and polished for 30 seconds with #600 silicon carbide sandpaper aiming the standardization of smear-layer.

Bonding Procedure

The adhesion protocol of each ceramic piece and the dental substrate followed the manufacturer's recommendations (Table 3).

Table 3 - Surface treatment of each group

GROUPS	Surface Treatment
LD	Application of 5% hydrofluoric acid for 20s. Water spray (60 s), air jet drying for 20 s. Active application of silane for 60s (Monobond® N).
ZLS	Application of 5% hydrofluoric acid for 30s. Water spray (60s), oil-free air jet drying for 20s. Active application of silane for 60s (Monobond® N).
Y-TZP C	Blasting: Al_2O_3, 110 µm, 1.5 bar. Water spray (60s), oil-free air jet drying for 20s.
Y-TZP T	Blasting: Al_2O_3, 110 µm, 1.5 bar. Water spray (60s), oil-free air jet drying for 20s.
E + E	Selective enamel etching with 37% phosphoric acid for 30 s. Water spray, Oil-free air jet drying
E	Dental substrate cleaning: brush, pumice stone and water. Water spray, Oil-free air jet drying.
Dentin	Dental substrate cleaning: brush, pumice stone and water. Water spray, Oil-free air jet drying, avoiding dryness.
Self-Adhesive cement resin Multilink® Speed	Chemical polymerization Positioning and stabilization of the cylinder containing the cement, with uniform pressure. Removal of material excess immediately using microbrush or curette.

Surface treatment of each group according to the manufacturer.

In order to conform the cement dispensed from the automix syringe, a plastic matrix cylinder with an internal diameter of 1.40 mm and a height of 1 mm was used. The device was stabilized under each ceramic and dental surface on the PVC base using a double-sided self-adhesive tape (3M, Sumaré, SP, Brazil) containing a 1.4 mm diameter hole corresponding to the bonding area, then the plastic matrix with resin cement followed by the application of glycerin gel to assist in self-polymerization, resulting in a cylinder of cement on each surface (ceramic, dentin or enamel). Ten minutes after chemical polymerization, the plastic matrix was removed with a #11 blade (Embramed, Jurubatuba, SP, Brazil) to expose the resin cement cylinders. Samples lost during this removal were discarded and did not enter the statistical analysis. The specimens were stored in deionized water at 37 °C for 24 h.

Structural analyses

Microshear Bond Strength Test (μSBS)

The tests were performed using 0.2 mm metallic wire positioned as close as possible to the adhesive interface (cylinder of resin cement bonded to a ceramic or dental substrate) in a universal testing machine (Instron 3342, Illinois Tool Works, Norwood, MA, USA), with a 325 N load cell at a speed of 0.5 mm/min.

Failure Mode Analysis

After fracture, the specimens were analyzed under a stereomicroscope (AxioCam MRc, ZEISS®) at 32x magnification. Fracture patterns were classified into: (1) adhesive (failure at the dental substrate/cement or ceramic/cement interface), (2) cohesive in dental substrate (failure in dentin or enamel), cement (failure in cement) or ceramic (ceramic failure) and, (3) mixed (combination between adhesive and cohesive failure).[27,28]

Scanning Electron Microscopy (SEM)

In order to qualitatively analyze the fracture pattern, a representative specimen of the different types of failures, previously stored in an oven at 25°C, were fixed in stubs, vacuum-dried and pulverized with gold in a pulverizing machine (Denton Vacuum Desk IV, Moorestown, NJ, USA) for scanning electron microscope (SEM) analysis (JEOL JSM-T220A, Tokyo, Japan). The analysis was performed at 35x magnification.

Statistical Analysis

The microshear strength data were statistically analyzed using SigmaPlot software. Shapiro-Wilk test was performed for normality analysis. Not obtaining a normal distribution ($p>0.05$), the non-parametric Kruskal-Wallis test was performed, as well as the multiple comparison with Dunn's post-test. An overall significance level of 5% ($p<0.05$) was adopted.

Weibull Analysis

The bond strength data obtained by microshear test were subjected to Weibull statistical analysis (Weibull++, Reliasoft, Tucson, AZ, USA). The description of the Weibull distribution is given by:

$$P_f = 1 - \exp\left[\left(\frac{\sigma}{\sigma 0}\right)^m\right]$$

Where Pf is the probability of failure, given by:

$$P_f = \frac{\kappa}{N + 1}$$

κ (kappa) is the ranking in strength from smallest to largest. *N* denotes the total number of specimens in the group, *m* is the shape parameter (Weibull modulus), *σ (sigma)* is the fracture strength at a given P_f and σ_0 is the characteristic resistance. A Weibull cumulative failure distribution curve was established to assess failure probability and Weibull modulus (m) *versus* characteristic stress (σ0) for each group (90% confidence).

RESULTS

Microshear Bond Strength Test (μSBS)

The median and interquartile ranges (25%/median/75%) of the microshear bond strength values of the self-adhesive resin cement to different types of substrates are shown in Table 4 ($p<0.05$). The D group presented the lowest result, with statistical difference for all groups, except for group E with which it was similar. The E + E group presented the highest result and statistical similarity with LD and ZLS. There was no statistically significant difference between the ceramic groups (Figure 1).

Table 4 - Microshear bond strength data

Groups	N	Median	1 Quartil 25%	3 quartil 75%	Dunn
E + E	20	24.88	20.6	27.4	a
E	20	8.69	8.24	11.77	cd
D	20	4.51	3.62	6.19	d
ZLS	20	20.07	15.26	25.89	ab
LD	20	15.35	14.0	18.01	ab
Y-TZP T	20	14.99	11.96	18.73	bc
Y-TZP C	20	12.78	11.59	20.80	bc

According to Dunn's post-test, similar letters indicate statistically significant similarity and different letters indicate a statistically significant difference of 5% ($p<0.05$).

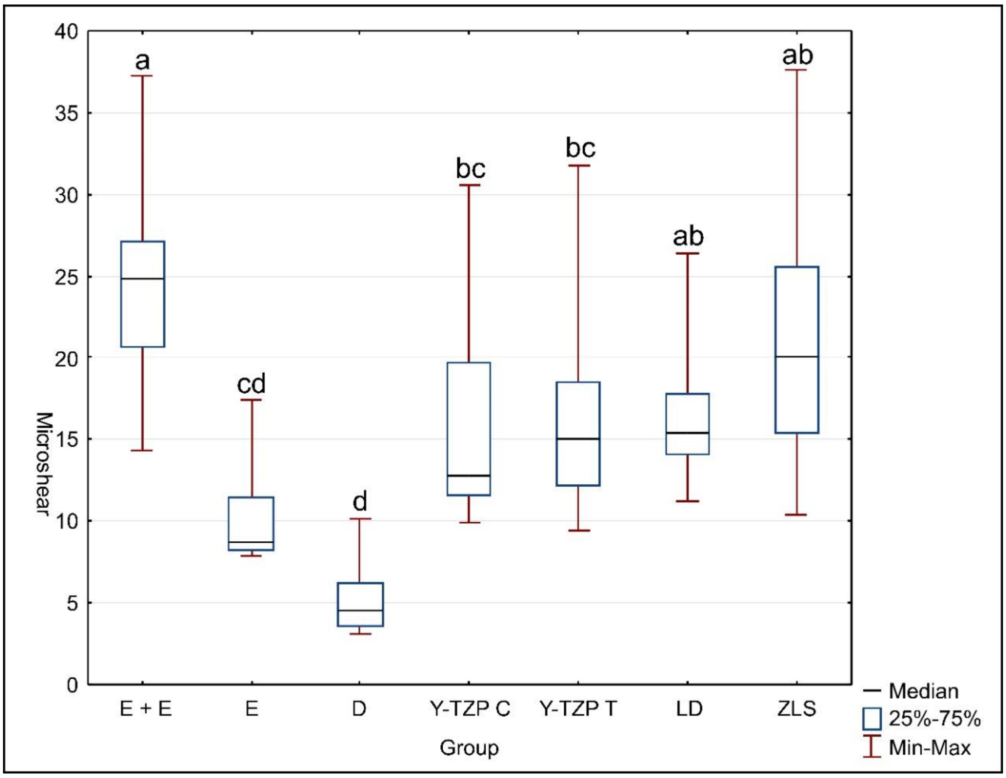

Figure 1 –Microshear bond strength data. Medians and quartiles of, (25%/ median / 75%): microshear teste in each group (E + E, E, D, LD, ZLS, Y-TZP C e Y-TZP T). Different letters indicate statistically significant differences.

The microshear strength of cement to conditioned enamel (E + E) had the highest median (24.88 MPa) showing statistical similarity to the groups of glass ceramics analyzed ZLS (Celtra Duo) (20.07 MPa), LD (e-max) (15.35 MPa), as well as a statistically significant difference in relation to groups E (8.69 MPa) and D (4.51 MPa). Those groups who showed lower bond strength did not receive any previous surface treatment and showed significant similarity to each other. The D group obtained the lowest bond strength value in relation to all the analyzed groups. Group E also exhibited statistical similarity to the Y-TZP T (14.99 MPa) and Y-TZP C (12.78 MPa) groups. these groups, classified as polycrystalline ceramics, did not present statistical differences among themselves or among the other ceramic groups analyzed.

Weibull Analysis

The probability of failure of the groups under tension (MPa) is shown in Figure 2. The adhesive interface with etched enamel (E + E) had a lower probability of failure with the increase in the generated tension. That group had no significant difference in relation to ZLS ceramic, which showed the second lowest probability of failure. This one was similar to DL, which presented a higher probability of failure in relation to the ZLS and E + E groups and without difference in relation to Y-TZP C and Y-TZP T. The D group showed the highest probability of failure at the lowest tension generated, showing a significant difference in relation to all the other groups, followed by the E group.

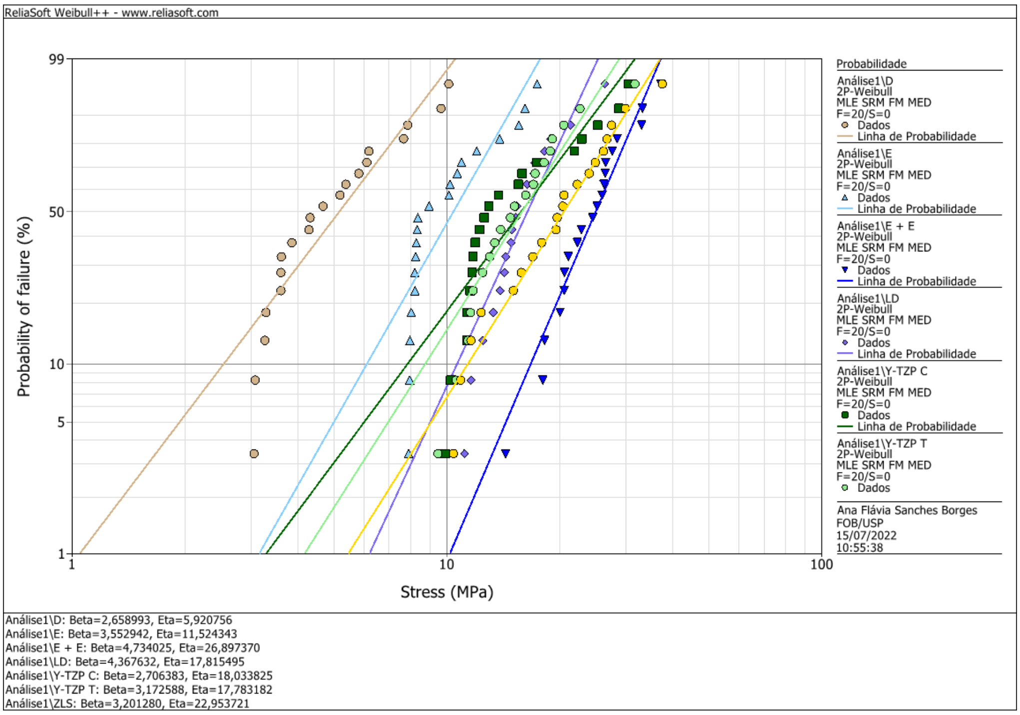

Figure 2 - Probability of failure (%) versus stress (MPa). The E + E group had a lower probability of failure, with no statistical difference to the ZLS group. The DL group had a higher probability of failure compared to the ZLS and E + E groups, but statistical similarity to the ZLS, Y-TZP C and Y-TZP T. The D group, followed by the E group showed a higher probability of failure and a significant difference compared to all other groups.

According to numerical and qualitative analysis by Weibull: Weibull modulus and characteristic strength in MPa of the analyzed groups are shown in Table 5 and Figure 3. The distribution of the study population showed that the E + E group achieved the highest characteristic strength (σ 0 = 26.89), followed by the ZLS (σ 0 = 22.95). The overlap of these two groups shows that 63.2% of their sample would fail under similar stresses and failure types. Thus, the E+E group exhibited the highest Weibull modulus, with lower variability and higher reliability. However, despite the statistical similarity, the SLZ showed high variability in the sample data, making the LD group (m = 4.36) the most reliable, following the E + E group. The unetched enamel group (m = 3.55) showed superior reliability compared to dentin and the two zirconia groups. Y-TZP C and Y-TZP T showed no difference related to characteristic strength. However, Y-TZP T (m = 3.17) was more reliable compared to Y-TZP C (m = 2.70). This last was similar to D (m = 2.65), which showed lower characteristic strength and lower reliability.

The Y-TZP C and Y-TZP T groups showed no difference in terms of characteristic resistance, with the Y-TZP T (m = 3.17) being the most reliable in relation to Y-TZP C (m = 2 .70).

Table 5 - Weibull analysis data. Weibull modulus (*m*) data and characteristic strength (MPa)

Groups	Weibull modulus (*m*)	Characteristic strength (MPa)
E + E	4,73	26,89
E	3,55	11,52
D	2,65	5,92
ZLS	3,20	22,95
LD	4,36	17,81
Y-TZP T	3,17	17,78
Y-TZP C	2,70	18,03

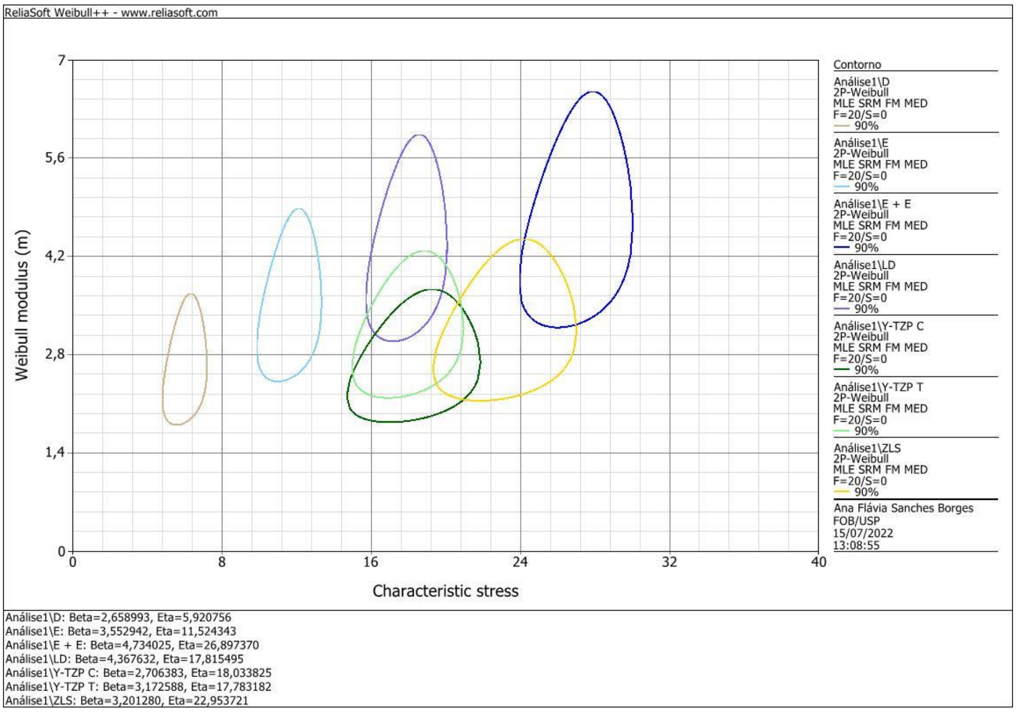

Figure 3 - Contour plots using 90% confidence bounds for the relationship between Weibull modulus (m) and Characteristic strength (σ_0) for all groups tested in microshear, in which 62.3% of the specimens will be failed.

Failure Mode Analysis and Scanning Electron Microscopy (SEM)

The failure modes of the groups are illustrated in Figure 3 and described in Table 6 by SEM analysis. Among the different types of failures, the adhesive type was predominant (73.57%) among all groups. The E group had the highest adhesive failure rate (95%) at its interface with the cement. For mixed failure, the E + E group prevailed

(70%). Cohesive failure in cement occurred in only one specimen from the D group (5%).

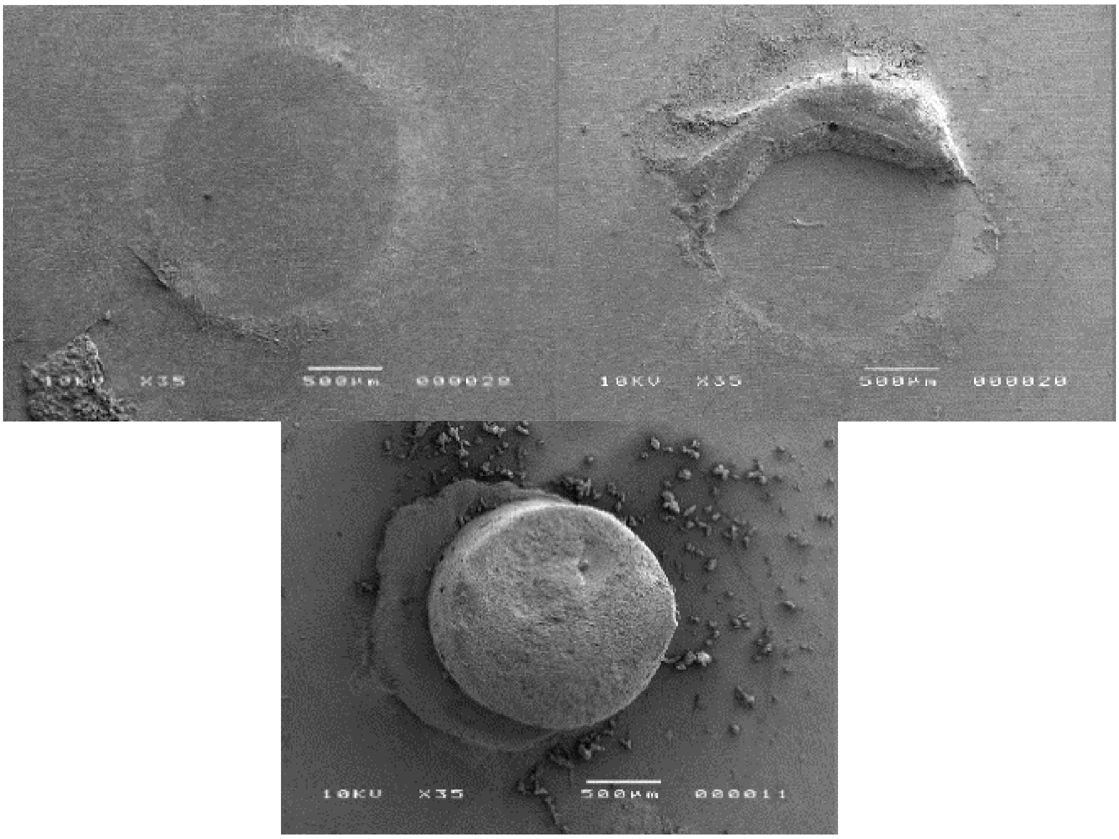

Figure 4 - Scanning Electron Microscopy (SEM) Observation (35x) representing the main types of failures affected (A) LD – adhesive; (B) Y-TZP C- mixed;(C) D- Cohesive in cement.

Table 6 - Failure mode analysis (%). Adhesive (failure at the interface of dental substrate/cement or ceramic/cement), cohesive in dental substrate (failure in dentin or enamel), cohesive in cement (failure in cement) and cohesive in ceramic (failure in ceramic) or mixed (combination between adhesive failure and cohesive failure)

Groups	Adhesive n (%)	Mixed n (%)	Cohesive in tooth n (%)	Cohesive in Cement n (%)	Cohesive in Ceramic n (%)
E + E	6 (30)	14 (70)	0	0	0
E	19 (95)	1 (5)	0	0	0
D	16 (80)	3 (15)	0	1 (5)	0
ZLS	13 (65)	7 (35)	0	0	0
LD	18 (90)	2 (10)	0	0	0
Y-TZP T	15 (75)	5 (25)	0	0	0
Y-TZP C	16 (80)	4 (20)	0	0	0
Total	103 (73,57)	36 (25,71)	0	1 (0,71)	0

DISCUSSION

The hypothesis that there would be a difference in the bond strength between the interface of the different types of ceramics evaluated to the self-adhesive and self-curing cement was rejected. However, when evaluating numerical and qualitative the probability of failure and reliability by Weibull analysis, the glass-ceramics showed similar behavior to enamel with etching. This fact suggests that in the case of adhesive interface with ceramic materials, the Weibull analysis is more complete to more accurately elucidate the characteristics of the population samples of the interfaces studied.

The bonding of glass-ceramics to resin cements, as well as their surface treatment, are well established in the literature. Conditioning with hydrofluoric acid at different concentrations is considered the gold standard, with a concentration of 5% being recommended. This step promotes partial dissolution of the vitreous content and is followed by the application of silane bonding agent and then, resin cement.[10,29,30] The higher strength value obtained by the ZLS group (Celtra Duo®) reflects the bond strength and effective surface treatment of glass ceramics to the satisfactory chemical interactions between their zirconia-based reinforcement particles and the self-adhesive cement through the phosphate monomer in the universal primer used.[31] Figures 2 and 3 show statistical similarity of the ZLS and E + E groups in the probability of failure and bond strength. This result indicates chemical-mechanical and adhesive similarities between these interfaces. Although the ZLS group presented the highest characteristic resistance value, obtained high data variability, not showing the greatest reliability. Among the analyzed ceramics, the most reliable was the LD group. The significant difference between Weibull parameters in the same group was previously identified in the literature.[32] These parameters reflect the distribution of failures and the high variability of data results in lower reliability in the characteristic strength. Thus, interfaces that exhibited low Weibull modulus suggest high variability in bond strength, being more sensitive to this process.[33]

On the other hand, the adhesion of polycrystalline ceramics to resin cement is still a challenge. Several studies have proposed different surface treatments, aiming to optimize adhesive bond strength. These materials are characterized by the absence of a glassy phase, making etching with hydrofluoric acid ineffective.[11-13,34] In our study, conventional surface treatment with aluminum oxide blasting was performed. The similarity exhibited by different zirconia to glass-ceramics could be explained by the strong chemical interaction between the metal oxide present in the zirconia and the phosphoric group of the acidic monomer on the self-adhesive cement[14,17]. Ayyildiz et al.[15] (2015) evaluated the shear bond strength of lithium disilicate and zirconia ceramics, performing the same surface treatment as in this study. When comparing zinc phosphate cement, conventional resin cement and self-adhesive cement, this last one achieved the best performance, especially when applied to zirconia. A similar result was also found, with no difference in strength, for this cement combined or not with a MDP (methacryloyloxy-decyl-dihydrogenphosphate) based primer, thus dispensing its use.[16,20] These data reinforce the reliable bond of self-adhesives to zirconia.

The second hypothesis that there would be a difference in the bond strength at the cement/dental substrate interface, with and without selective acid etching used in enamel, was accepted. Bond strength values at the interface between enamel and cement were higher with acid etching prior to cementation, compared to enamel without any treatment. The literature has proposed this strategy because it causes greater surface energy and irregularities in the enamel microstructure promoted by the etching, compared to what is performed by the acidic monomers of the self-adhesive cement itself (weaker).[8,22,25,26,35] Therefore, in this study, the E + E group showed higher Weibull modulus (m) and higher characteristic strength (σ 0). This demonstrates greater effectiveness in the bond strength of this interface, as well as a greater probability of interaction of the E + E substrate with the resin cement. The lower bond strength without prior conditioning can be attributed to the lower micromechanical imbrication and chemical reactivity of the acidic monomers present in the cement itself. These characteristics make it less effective for this surface of higher mineral content.[1,6,22,26] In another study, SEM analysis at the interface of non-etched enamel to self-adhesive cement showed marginal gaps representing areas of adhesion loss. This fact did not occur at the enamel/conventional resin cement interface.[8]

The interface of the Dentin group to the cement presented the lowest value of bond strength, with a significant difference in relation to all the others. This group exhibited the lowest values in all parameters analyzed: reliability, characteristic strength and higher probability of failure. Another study also indicated a lower performance attributed to the shear-bond strength of dentin to different self-adhesive cements.[7] This result can be attributed to the presence of acid-modified methacrylate monomers of self-adhesive cements performing a surface demineralization and simultaneous infiltration into the dental substrate. This process causes a zone of irregular interaction between cementing agent and dental tissue, which thickness is below 1µm, called interdiffusion zone[8,36]. The conventional system with acid etching and washing can reach 1–5 µm.[37] Studies attribute low values of bond strength to dentin to the low cohesive strength of the substrate itself, associated with the lack of pH neutralization of some cements.[24] Despite this, other studies have detected a stable bond to dentin.[36] However, the less effective etching of acid monomers in enamel was superior in adhesion to dentin, which, in this study, presented lower reliability (m), due to the high variability of the data. In the present study, dentin was used in its intermediate portion, to avoid a high coefficient of variation of the resistance data.[38,39]

The Weibull allows us to infer that the causes of failures were similar. This data is confirmed by the failure analysis, with a predominance of the adhesive type (73.57%) in all groups. The microshear test is commonly used as a mechanical strength test for adhesive interfaces,[40-43] due to characteristics such as: adhesion area less than 2 mm^2, greater sensitivity and production of more homogeneous data, lower percentage of cohesive failures and lower probability of finding a defect of critical size.[7,40-44] The exception was the selectively etched enamel interface showing more mixed-type failures. In line with the literature, changes in the pattern of adhesive failure to mixed in conditioned enamel[22,26,45,46] could be associated with a greater interaction between self-adhesive resin cement and etched enamel. This can be explained by the greater infiltration of resin tags after effective enamel demineralization by phosphoric acid, creating better micromechanical retention.[26]

CONCLUSION

Within the limitations of this study, we can conclude that the adhesive interface of glass-ceramics presented higher characteristic strength, lower probability of failure and higher reliability, being similar to selectively etched enamel.

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