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A localização do término marginal em coroas cimentadas é um fator que influencia a resistência à tração, adaptação marginal e nanoinfiltração

The finish line location of the cemented crown is an influencing factor for tensile bond strength, marginal adaption and nanoleakage

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Dissertação apresentada à Faculdade de Odontologia de Piracicaba, da Universidade Estadual de Campinas como parte dos requisitos exigidos para a obtenção do título de Mestre em Clínica Odontológica, na Área de Dentística.

Dissertation presented to the Piracicaba Dental School of the University of Campinas in partial fulfillment of the requirements for the degree of Master in Clinical Dentistry, in Restorative Dentistry area.

Orientador: Prof. Dr. Luís Roberto Marcondes Martins

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Resumo

Com o avanço de novas técnicas e dos materiais à base de resina composta, vem se discutindo a real necessidade do término marginal estar localizado em estrutura dental. O objetivo deste estudo foi avaliar a influência da localização do término marginal na cimentação de coroas, na resistência à tração, adaptação marginal e nanoinfiltração, de acordo com os fatores: término marginal (dentina, esmalte e resina composta), e material restaurador (resina composta microhíbrida e cerâmica em dissilicato de lítio). Foram coletados 60 terceiros molares hígidos. Para o teste de resistência à tração, todas as amostras foram praparadas com término em chanfro e um cimento resinoso autoadesivo foi utilizado para a cimentação. Para a avaliação da adaptação marginal, foram confeccionados modelos em resina epóxica da linha de cimentação das amostras, previamente ao teste de tração, e submetidas à avaliação em microscopia eletrônica de varredura (MEV), para obtenção de imagens que posteriormente foram mensuradas. Para a nanoinfiltração, foram confeccionados fragmentos dos substratos e materiais restauradores, que foram cimentados com o mesmo protocolo. As amostras/imagens foram obtidas em MEV e mensurada a área infiltrada. O padrão de fratura foi avaliado através de imagens obtidas no MEV e classificados em: falha adesiva, coesiva em cimento, coesiva em dentina, coesiva em resina composta, coesiva em esmalte e mista. A análise estatística foi feita utilizando o teste de normalidade Shapiro-Wilk e Kolmogorov Smirnov e testes paramétricos Anova dois-fatores e Bonferroni (post-hoc), com nível de significância de 5% ($p<0.05$), e teste de Spearman de correlação. No teste de resistência à tração, não foi observada diferença estatística entre os grupos cimentados com resina composta e cerâmica. Foi observada diferença estatística entre o grupo com término em esmalte/dentina (EC-3,28 MPa e DC-3,14 MPa, respectivamente), e resina composta (RC-2,85 MPa), na cimentação da coroa cerâmica. Na avaliação da adaptação marginal, observou-se diferença estatística entre os grupos cimentados com resina composta/cerâmica e a localização do término, com variação de 175,91 μm (grupo EC), e 433,58μm (grupo RR). Na avaliação de nanoinfiltração, foi observada diferença estatística entre todos os grupos, com exceção dos grupos com localização do término em resina composta RR (9,49%) e RC (9,35%). A avaliação do padrão de fratura apresentou em todos os grupos uma predominância de falha adesiva. Pode-se concluir que o preparo para coroa total pode ser realizado com segurança tanto em esmalte, quanto dentina. O término em resina composta apresentou-se promissor, porém ainda faz-se necessário maiores estudos quanto à sua indicação. Podendo ser uma alternativa para se evitar términos subgengivais. A reabilitação pode ser feita com segurança tanto em coroas a base de resina composta ou cerâmica, porém a cerâmica apresentou melhor desempenho, relacionado a resistência de união, adapatação marginal e nanonifintração.

Palavras-chave: coroa do dente; cimentação; cerâmica; resistência à tração

Abstract

With the advancement of new techniques and resin composite materials based, it has been discussed the real need for the marginal finish line location be on dental structure. The aim of this study was to evaluate the influence of finish line location on crown cementation, on tensile bond strength, marginal adaption and nanoleakage, according to the factors: finish line location (dentin, enamel and resin composite) and restorative material (microhybrid resin composite and ceramic in lithium disilicate). Sixty healthy third molars were collected. For the tensile bond strength test, all samples were prepared with a chamfer finish design and a self-adhesive resin cement was used for cementation. For the evaluation of the marginal adaption, epoxy resin models were prepared from the cementation line of the samples, prior to the tensile bond strengh test, and submitted to scanning electron microscopy (SEM), to obtain images that were subsequently measured. For the nanoleakage, fragments of the substrates and restorative materials were made, which were cemented with the same protocol. Samples / images were obtained in SEM and the infiltrated area was measured. The failure mode was evaluated through SEM images and classified as: adhesive failure, cohesive in cement, cohesive in dentin, cohesive in resin composite, cohesive in enamel and mixed. Statistical analysis was performed using the Shapiro-Wilk and Kolmogorov Smirnov normality test and Anova two-factor and Bonferroni (post-hoc) parametric tests, with a significance level of 5% ($p < 0.05$) and Spearman correlation test. In the tensile bond strength, no statistical difference was observed between the groups cemented with resin composite and ceramic. It was observed a statistical difference between the group with enamel / dentin (EC-3.28 MPa and DC-3.14 MPa, respectively) and resin composite (RC-2.85 MPa) in the cementation of the ceramic crown. In the evaluation of the marginal adaption, it was observed a statistical difference between the groups cemented with resin composite / ceramic and the finish line location, with a variation of 175.91μm (EC group) and 433.58μm (RR group). In the evaluation of nanoleakage, a statistical difference was observed among all groups, except for the groups with location in resin composite RR (9.49%) and RC (9.35%). The evaluation of the failure mode showed a predominance of adhesive failure in all groups. It can be concluded that the preparation for crown can be performed safely in both enamel and dentin. The composite resin as finish line location presented promising results, however, further studies are still needed to regarding its indication; it may be an alternative to avoid subgingival terms. The restoration can be done safely in crowns made of composite resin or ceramic, with ceramic presented better performance, related to bond strength, marginal adaption and nanoleakage.

Keywords: tooth crown; cementation; ceramics; tensile bond strength

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1 Introdução

A reabilitação de um dente com perda de estrutura dental em grande extensão pode ser feita de diferentes formas, utilizando-se diferentes técnicas, aplicada de acordo com a escolha do material (Arhun N, Celik C, Yamanel K, 2010; Cortellini D, Canale A 2012). Tal reabilitação pode ser feita desde a utilização de restaurações em resina composta realizada diretamente em boca, até restaurações indiretas, como coroas à base de resina composta ou cerâmica, confeccionadas em laboratório (Arhun N, Celik C, Yamanel K, 2010; Cortellini D, Canale A 2012).

Assim como o material e técnica de escolha são importantes, a localização e design do término marginal também é (Cho L, Choi J, Yi YJ etal., 2004). A literatura aponta que o esmalte, sempre apresenta uma melhor capacidade de resistência de união, muito devido ao seu conteúdo mineral e baixa concentração (cerca de 3%), de água (Mine A, De Munck J, Van A, 2017). A dentina por outro lado, apresenta uma complexibilidde de adesão devido à sua composição orgânica/inorgânica e sua alta concentração de água (cerca de 20%) presente em sua composição (El Zohairy AA, De Gee AJ, Mohsen MM etal., 2005). Com o maior desenvolvimento dos materiais à base de resina composta, somado ao preceito de menor desgaste de estrutura dental, a localização e o design do término do prepare para coroa total vem sendo questionado na literatura (Minyé HM, Gilbert GH, Litaker MS etal., 2018). Porém, ainda há um gap de estudos comprovando a eficácia do término localizado em resina composta.

Considerando o preceito de uma maior preservação de estrutura dental, e com a crescente exigência estética dos pacientes, materiais livres de metal estão sendo cada vez mais utilizados, e com uma alta taxa de sucesso clínico (Minyé HM, Gilbert GH, Litaker MS et al., 2018). Reabilitações somente em cerâmicas estão sendo cada vez mais utilizadas, com ótimo resultado estético e funcional (Al-Akhali M, Chaar MS, Elsayed A et al., 2017). Há diversos tipos de cerâmicas que podem ser utilizadas em uma reabilitação, como a zircônia e leucita; uma dos principais e mais utilizada é a cerâmica de dissilicato de lítio (Oh SC, Dong JK, Lüthy H, Schärer P., 2000). Tal cerâmica possui elevado nùmero de cristais de dissilicato de lítio inter-relacionado com a matriz vítrea, que ja está consolidado na literatura com bons resultados de integridade marginal e resistência mecânica (Attia and Kern, 2004, Guess et al., 2013, Kern et al., 2012, Sasse et al., 2015).

Como uma alternativa às cerâmicas, a resina composta é um material que vem sendo utilizado e apresentando na literatura resultados clínicos satisfatórios (Gianordoli-Neto R, Padovani GC, Mondelli J et al., 2016; Karaman E, Keskin B, Inan U. 2016; Schwfinish lineicke F, Krüger H, Schlattmann P et al., 2016). Entretanto, algumas características da resina composta vêm sendo apontados como potenciais problemas: fendas marginais, sensibilidade pós-operatória e fraturas (Ferracane JL, 2011).

A resistência de união às forças de mastigação e a adaptação marginal são fatores importantes para o sucesso da cimentação de coroas; tanto quanto, a resistência às infiltrações (de Alexandre RS, Santana VB, Kasaz AC et al., 2014; Ganapathy D, Sathyamoorthy A, Ranganathan H et al., 2016). O cimento resinoso é um material que ficará em meio bucal, e passará por um processo de envelhecimento durante a vida útil da reabilitação, o que pode comprometer suas características mecânicas com o passar do tempo, degradando a rede de colágeno e de polímeros na interface de cimentação (Medeiros IS, Gomes MN, Loguercio AD et al., 2007). Tal degradação da interface de cimentação levará a criação de fendas marginais, e consequentemente à maior infiltração, o que acarretará em uma menor resistência de união da cimentação (Medeiros IS, Gomes MN, Loguercio AD et al., 2007; Pfeifer CS, 2017).

Problemas podem ser gerados por erros técnicos, durante o processo de cimentação, como maior desadaptação marginal, maior acúmulo de placa, progressão de cárie secundária, trincas e diminuição na resistência de união; por esta razão, o cimento auto-adesivo foi desenvolvido, com o intuiito de diminuir a sensibilidade da técnica, por diminuir o números de passos (de Alexandre RS, Santana VB, Kasaz AC et al., 2014; Mously HA, Finkelman M, Zandparsa R et al., 2014). Os cimentos resinosos são materiais friáveis, por isso faz-se importante estudar a resistência de união, adaptação marginal e resistência a infiltrações do sitema: término-cimento-coroa (Medeiros IS, Gomes MN, Loguercio AD et al.,2007; Blumer L, Schmidli F, Weiger R et al., 2015).

Com isso, o objetivo deste estudo foi avaliar a influência da localização do término marginal na cimentação de coroas, em resina composta e cerâmica, por meio da avaliação da resistência à tração, adaptação marginal e nanoinfiltração. A hipótese testada foi: a localização do término marginal não influenciou na cimentação de coroas, na resistência à tração, adaptação marginal e nanoinfiltração.

2 Artigo

The finish line location of the cemented crown is an influencing factor for tensile bond strength, marginal adaption, and nanoleakage

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ABSTRACT

Statement of Problem: With advances in techniques and resin composite-based materials, determining the influence of the marginal finish line location on dental structure is critical.

Purpose: It was evaluated the influence of the finish line location of crown cementation on the tensile bond strength, marginal adaption, and nanoleakage, according to the following factors: finish line location (dentin, enamel, and resin composite) and restorative material (microhybrid resin composite and ceramic of lithium disilicate).

Material and Methods: Sixty healthy third molars were collected. For evaluation of the tensile bond strength, all samples were prepared with a chamfer finish design and a self-adhesive resin cement was used for cementation. For evaluation of the marginal adaption, epoxy resin models were prepared from the cementation line of the samples. Prior to performing the test for tensile bond strength, images were acquired under scanning electron microscopy (SEM) and subsequently measured. The nanoleakage was determined in premade fragments of the substrates and restorative materials, which were cemented using the same protocol. Images of the samples were obtained under SEM and the infiltrated area was measured. The failure mode was evaluated through SEM image analysis and classified as: adhesive failure, cohesive in cement, cohesive in dentin, cohesive in resin composite, cohesive in enamel, and mixed. Statistical analysis was performed using Shapiro-Wilk and Kolmogorov Smirnov normality tests, two-way ANOVA, and Bonferroni (post-hoc) parametric test, with a significance level of 5% ($P < .05$), and Spearman correlation test.

Results: The tensile bond strength was not statistically different between the groups cemented with resin composite and ceramic. The cementation of the ceramic crown was not statistically different between the groups (enamel (EC), 3.28 MPa; dentin, 3.14 MPa; resin (RC), 2.85 MPa). The marginal adaption was statistically different between the RC and ceramic groups; finish line location varied between the EC and RR groups (175.91 μm vs. 433.58 μm). Nanoleakage rate was statistically different among all groups, except for the groups with finish line location in the resin composite: with resin composite crown (9.49%) and with ceramic crown (9.35%). There was a predominance of adhesive failure in all groups.

Conclusions: The preparation for crown can be performed safely in both enamel and dentin. The composite resin as finish line location presented promising results, however, further studies are still needed to regarding its indication; it may be an alternative to avoid subgingival terms. The restoration can be done safely in crowns made of composite resin or ceramic, with ceramic presented better performance, related to bond strength, marginal adaption and nanoleakage.

Keywords: tooth crown; cementation; ceramic; tensile bond strength

Clinical Implications: The location of the marginal finish line can already be made in resin composite with safety, showing a potential of greater stability in the interface to infiltrations.

INTRODUCTION

The bond strength to withstand chewing forces, marginal adaption, as well as resistance to infiltration are important factors to achieve success in the cementation of the crown.[1,2] The resin cement maintains constant contact with the buccal media and undergoes aging during the lifetime of the rehabilitation instrument, which may result in compromise of its mechanical characteristics over time, and degradation of the collagen and polymer network at the cementation interface.[3] Such degradation leads to the formation of marginal cracks and, consequently, to larger infiltrations, which result in lower union strength of the cementation.[3,4]

Rehabilitation of a tooth with extensive loss of dental structure, can be achieved using different techniques according to the choice of material, such as the use of resin composite directly in the mouth, and indirect restorations with composite resin or ceramic crowns made in the laboratory.[4,5]

The material and technique of choice, as well as the location and design of the marginal term are important considerations.[6] The literature reports indicated that enamel consistently presented superior bond strength, due to its mineral content and low water concentration of up to 3%; whereas, dentin presented difficulty of adhesion, due to its organic and inorganic composition and high water content of about 20%.[8] Through advanced development of resin composite materials coupled with the precept of lesser dental structure wear, studies have focused on the influence of the location and design of the crown;[9] however, there are no studies to determine the effectiveness of that of resin composite.

According to the guidelines for preservation of the dental structure, and increasing aesthetic requirement of the patients, metal-free materials are increasingly being used with high clinical success rate.[10] Rehabilitation has increasingly used ceramics alone, with good aesthetic and functional results[11]; for this purpose, there are several types of ceramics, such as zirconia and leucite, of which lithium disilicate ceramics is a most frequently utilized type,[12] which consists of a high number of lithium disilicate crystals interrelated with the glass matrix and shows good results in terms of marginal integrity and mechanical strength.[13-15] Composite resin is an alternative material to ceramics that presents satisfactory clinical results;[16-18] however, a study demonstrated some potential problems with resin composite such as marginal cracks, postoperative sensitivity, and fractures.[19]

Problems can be generated by technical errors during the cementation process, such as poor marginal adaptation, increased accumulation of plaque, secondary caries' progression, cracks, and decrease in the bond strength.[20] To overcome these limitations, self-adhesive cement was developed to reduce the sensitivity of the technique by reducing the number of steps involved.[1,20] Resin cements are friable materials, hence it is important to study the bond strength, marginal adaptation, and resistance to infiltration of the finish line of the cement-crown.[3,21]

In this study, we aimed to evaluate the influence of the finish line location of crown cementation on the tensile bond strength, marginal adaption, and nanoleakage, according to the following factors: finish line location (dentin, enamel, and resin composite), and restorative material (microhybrid resin composite and ceramic of lithium disilicate). We hypothesized that the marginal finish line location does not influence the crown cementation in terms of the tensile bond strength, marginal adaption, and nanoleakage.

MATERIALS AND METHODS

This work was approved by the research ethics committee (CAAE 66767417.6.0000.5418).

Experimental design

Sixty third molars obtained from human subjects were included. The following factors were evaluated: 1) finish line location for cementation of the crowns: in dentin, enamel, and resin composite; 2) rehabilitation material: crown in composite resin, and ceramic crown of lithium disilicate in the injected system (E.max Press, Ivoclar Vivadent, Schann, Liechtenstein). Variable responses: tensile bond strength (n = 10); marginal adaption (n = 10); nanoleakage (n = 10), as described bellow:

DR: Dentin finish line– Resin composite crown (n=10)

DC: Dentin finish line– Ceramic crown (n=10)

ER: Enamel finish line– Resin composite crown (n=10)

EC: Enamel finish line– Ceramic crown (n=10)

RR: Resin composite finish line – Resin composite crown (n=10)

RC: Resin composite finish line – Ceramic crown (n=10)

Sample Preparation

Sample inclusion

Prior to the inclusion process, the tooth was demarcated at 2-mm distance from the amelo-cementation junction with an overhead pen. (Fig. 1A) The tooth was fixed with sticky wax, through the crown to the stem of a prosthetic parallelometer. A movable table was placed parallel to the long axis of the tooth, on which a polyvinyl chloride (PVC) cylinder of 10-mm central perforation and 2-mm height was positioned. The self-cured polystyrene resin was handled and poured into the PVC cylinder; after polymerization, it was removed from the support and any excess was removed with a scalpel blade.

The tooth was sectioned up to 2-mm length of the clinical crown (amelo-cementation junction) in a cutter. (Fig. 1B) All teeth were morphologically reconstructed as shown in Figure 1C with microhybrid resin composite (Filtek Z 250 XT, 3M ESPE, St. Paul, MN, USA), using phosphoric acid at 37% for 30 seconds on enamel, and 15 seconds on dentin, the acid was washed by water irrigation for 30 seconds, and dried by 30 seconds. Adhesive used as the bonding agent (Adapter Single Bond 2, 3M ESPE, St. Paul, MN, USA) was applied twice for 20 seconds with air evaporation of the solvent between each application, and subsequently photopolymerized by 20 seconds (Valo, Ultradent-Products Inc., South Jordan, UT, USA). All the specimens of dentin, enamel, and resin composite were prepared by the same recalibrated operator using a diamond-shaped conical drill bit (2135 KG Sorensen, São Paulo, Brazil). (Fig. 1C)

A retention loop was made on the occlusal surface of the resin/ceramic unitary crown. The crown of laboratory-made resin was produced following the same characteristics of the ceramic crown, and then polymerized. (Fig. 1D)

Cementation

A self-adhesive resin cement (Relyx U 200, 3M ESPE, St. Paul, MN, USA) was used as follows:

Surface treatment of the crowns

Resin composite unitary crown: Preapplication of phosphoric acid at 37% for 30 seconds, water irrigation for 30 seconds, and complete drying of the surface were performed. Sequentially, a thin layer of universal single-bond adhesive (3M ESPE, St. Paul, MN, USA), and self-adhesive cement was applied. Ceramic unitary crown: Preapplication of hydrofluoric acid at 5% for 20 seconds, water irrigation for 30 seconds, and complete drying of the surface were performed. Sequentially, a thin layer of adhesive single bond universal (3M ESPE, St. Paul, MN, USA) and self-adhesive cement was applied.

Finish line treatment:

Enamel finish line: Preapplication of phosphoric acid at 37% for 30 seconds, water irrigation for 30 seconds, and complete drying of the surface were conducted. Sequentially, the unitary crown was placed with self-adhesive cement, and photopolymerization for 30 seconds was performed. Dentin finish line: Without previous acid etching (following the manufacturer's instructions), water irrigation of the dentin was performed for 30 seconds and only the excess was removed, leaving the dentin wet. Sequentially, the unitary crown was placed with self-adhesive cement, and photopolymerization for 30 seconds was performed. Resin composite finish line: Preapplication of phosphoric acid at 37% for 30 seconds, water irrigation for 30 seconds each, and complete drying of the surface were performed. Sequentially, a thin layer of universal single-bond adhesive (3M ESPE, St. Paul, MN, USA) was applied, the unitary crown was placed with the self-adhesive cement, and photopolymerization for 30 seconds was performed.

The cementation process was done by the same operator who was precalibrated using finger pressure. The samples were polymerized for 30 seconds per face. Subsequently, the cementation region was isolated with wax number 7. (Fig. 1E) The region was isolated, the self-cured polystyrene resin was handled and poured into the region, and a retaining loop was made, enabling testing of the system with the universal testing machine. (Fig. 1D to H)

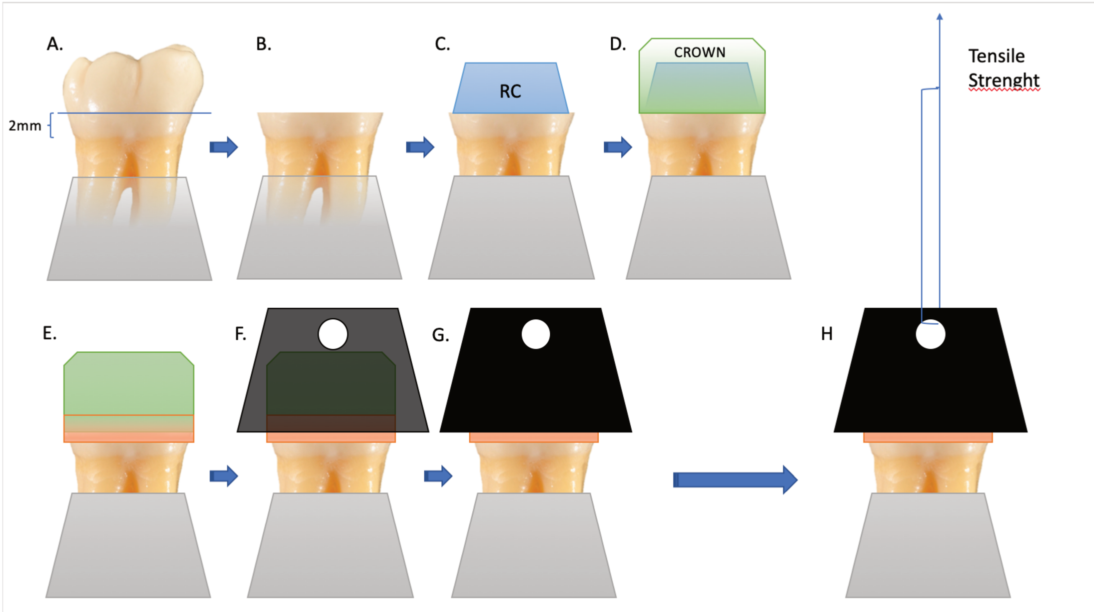

Figure 1. Schedule of sample preparation for the tensile bond strength test. A- inclusion; B- sectioning 2mm of the amelo-cement junction; C- Reconstruction with microhybrid resin composite; D- crown confeccion; E- isolation of cement line; F and G- inclusion of the crown; H - complete system for tensile bond strengh test.

Tensile Bond Strength

Tensile bond strength (Johnson GH, Lepe X, Patterson A et al., 2018) was produced along the long axis of the tooth, at a speed of 1 mm per minute and initial force of 30 N that was increased progressively until rupture of the cementation line. The entire tooth was used as a sample in order to approximate clinical behavior. To evaluate only the influence of the finish line on the cementation process, the clinical crown was sectioned to avoid any influence of the tooth anatomy on the results. For the standardization of results, the following equation was used:

$$RT = F/S.A.$$

where: RT, tensile bond strength (Mpa); F, force of the cementation line (N); S.A., sample area

Failure Mode

After performing the tensile strength test, the failure mode (Sadighpour L, Geramipanah F, Fazel A et al., 2018 - Modified) of the sample was classified through SEM (JEOL JSM-6610LV, MA, US): Samples were coated with a thin layer of carbon (BalTec SCD 050-SputterCoater) for observation under SEM under high vacuum at a power of 15 kV. The mode of failure was classified as: adhesive, cohesive in cement, cohesive in dentin, cohesive in resin composite, cohesive in enamel, and mixed.

Marginal adaption

Before isolating the cementation line in the tensile bond strength test sample, each sample was molded on the palatine and vestibular surfaces by silicone material, and epoxy resin models were made (Al Hamad KQ, Al Rashdan BA, Al Omari WM et al., 2018 – Modified). Subsequently, all samples were assembled in aluminum stubs to receive a thin-layer coating of gold (Balzers-SCD 050 Sputter Coater, Scotia, NY, USA) and evaluated under SEM (LEO 435 VP, LEO Electron Microscopy Ltd, Cambridge, UK) at magnification of 50X and 150X. The evaluation was performed 24 hours after preparing the unitary crowns, and images were measured using ImageJ software (LOCI, University of Wisconsin, USA).

Each image was measured at three points: both borders and center. Subsequently, the mean of each face was calculated, and in sequence, a new media between the palatine and vestibular face was made to obtain the mean value of the sample.

Nanoleakage test

For the nanoleakage test (de Alexandre RS, Santana VB, Kasaz AC et al., 2014), fragments (2 mm × 1 mm × 3 mm) were prepared in a precision metallographic cutter at a speed of 250 rpm under constant cooling of the dentin and enamel, and microhybrid resin composite (Filtek Z 250 XT, 3M ESPE, St. Paul, MN, USA) manufactured in a pre-fabricated Teflon-based matrix, for use as the base of the unitary crown, which was later cemented with resin and ceramics using self-adhesive cement (RelyX U200, 3M ESPE, St. Paul, MN, USA).

The samples of each group were immersed in silver nitrate solution (nitrate crystals 10 g in deionized water 10 mL to which ammonium hydroxide at 28% was applied in dropwise manner) for 24 hours at 37°C in a dark environment. Sequentially, the samples were washed in running water for 2 minutes and immersed in developing solution (developer powder 10.9 g in distilled water 100 mL) for 8 hours under a fluorescent lamp; the samples were washed with distilled water and immersed in polystyrene resin.

After inclusion, the samples were polished with 600, 1200, and 2000 water strips, and felt disks and diamond pastes at decreasing granulation of 3, 0.5, and 0.25 µm. Between polishing with each sandpaper and paste granulation, the samples were placed in an ultrasonic vessel for 10 minutes to remove debris. The samples were dried with absorbent paper and treated with phosphoric acid at 85% for 10 seconds to achieve demineralization, followed by washing with distilled water. Deproteinization was conducted using a 2% solution of sodium hypochlorite for 10 minutes; the samples were washed with distilled water and dried at room temperature. Subsequently, the samples were dehydrated in ethyl alcohol at increasing concentrations of 50%, 75%, 90%, and 100% for 10 minutes per concentration.

The samples were mounted on aluminum stubs and coated with a thin layer of carbon (BalTec SCD 050-SputterCoater) for observation through SEM under high vacuum at a power of 20 kV; images were obtained through backscattered electrons. The images were recorded for evaluation of the infiltrated area using ImageJ software. For each sample, the total area and infiltrated area was calculated, and the percent infiltration was derived.

All data were submitted to analysis of normality and homogeneity of values. Statistical analysis was performed using Shapiro-Wilk and Kolmogorov Smirnov normality test, two-way ANOVA and Bonferroni (post-hoc) parametric tests, with 5% of significance ($P < .05$), and Spearman test of correlation.

RESULTS

The tensile bond strength was not statistically different between the groups of ceramic and resin composite crown, indicating that the material of the crown was not an influencing factor for the performance of cementation; the ceramic crown cementation was statistically different between the groups with enamel/dentin (EC and DC) finish line and resin composite finish line (RC), indicating that the finish line location was an influencing factor for the bond strength. (Table 1)

Table 1. Averages and standard deviation of the tensile bond strength

	Tensile bond strength (Mpa)	
	Resin composite	Ceramic
Dentin	2.95 (0.17)^{Aa}	3.14 (0.35)^{Aab}
Enamel	3.15 (0.30)^{Aa}	3.28 (0.45)^{Aa}
Resin composite	2.81 (0.47)^{Aa}	2.85 (0.17)^{Ab}

Superscript letters indicate statistical difference between the groups (P < .05).

The marginal adaption was statistically different between the groups cemented with ceramic and resin composite crown, and those based on finish line location, indicating that the restoration material and finish line location were influencing factors for the marginal adaption. (Fig. 2 and Table 2)

Table 2. Averages and standard deviation of the marginal adaption

	Marginal adaption (μm)	
	Resin composite	Ceramic
Dentin	342.37 (21.75)^{Ab}	291.15 (17.56)^{Bb}
Enamel	261.42 (8.62)^{Ac}	175.91 (7.42)^{Bc}
Resin composite	433.58 (34.64)^{Aa}	368.68 (30.12)^{Ba}

Superscript letters indicate statistical difference between the groups (P < .05).

The nanoleakage was statistically different among all groups, excepting RR and RC, indicating that the finish line location was an influencing factor for the resistance of the interface to infiltration, independent of the base material of the crown. (Fig. 3 and Table 3)

Table 3. Averages and standard deviation of the nanoleakage

	Nanoleakage (%)	
	Resin composite	Ceramic
Dentin	23.59 (0.65)^{Aa}	22.14 (0.97)^{Ba}
Enamel	15.56 (0.44)^{Ab}	14.66 (0.45)^{Bb}
Resin composite	9.49 (0.55)^{Ac}	9.35 (0.18)^{Ac}

Superscript letters indicate statistical difference between the groups ($P < .05$).

Based on the results through Spearman correlation test, there was a negative and high correlation between the tensile bond strength and marginal adaption (ρ= -0.508; $P <.001$).

Based on the results of failure mode analysis, there were three types of fractures: adhesive, cohesive in resin, and mixed. (Fig. 4A to D) The DR group had a high rate of adhesive failure alone, and the DC group had a high rate of adhesive failure and low percentage of mixed failure cases. The ER and EC groups had a higher rate of adhesive failure followed by mixed failure compared to those of the other groups. The RR group had both adhesive failure and cohesive in resin; whereas, the RC group had a greater percentage of adhesive failure, and lower percentage of cohesive in resin and mixed. (Fig. 5)

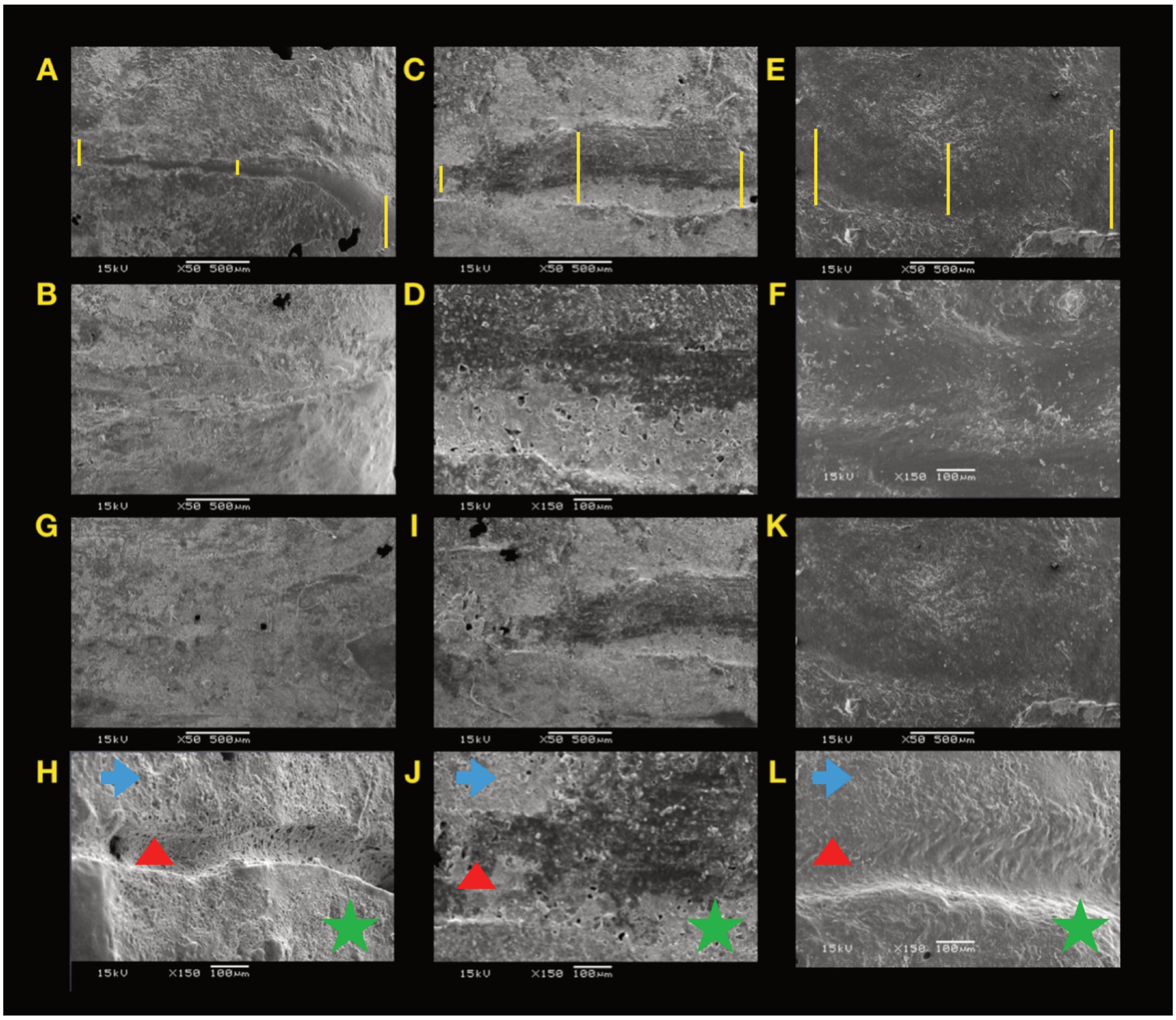

Figure 2 *Marginal adaption test. Figures A and B- group ER on 50x and 150x magnification, respectively. Figures C and D- group DR on 50x and 150x magnification, respectively. Figures E and F- group RR on 50x and 150x magnification, respectively. Figures G and H- group EC on 50x and 150x magnification, respectively. Figures I and J- group DC on 50x and 150x magnification, respectively. Figures K and L group RC on 50x and 150x magnification, respectively. Yellow lines in figures A, C and E representative of the measurement of the marginal adaption. Arrows in blue indicating the crown, red triangle indicating cementation line and green star indicating finish line location.*

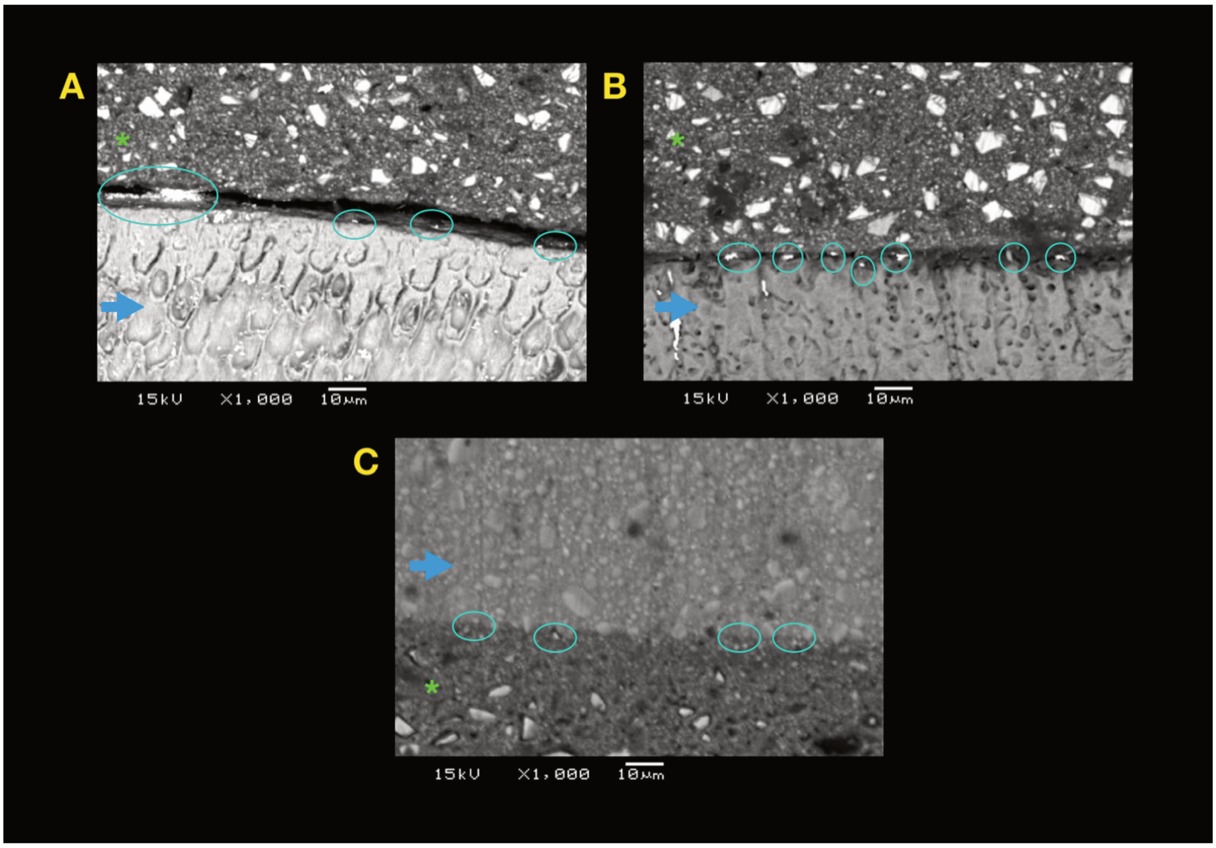

Figure 3 *Nanoleakage test. Figure A- Enamel as finish line location. Figure B- Dentin as finish line location. Figure C-Resin composite as finish line location. Circles in blue point the observed infiltrations. Asterisks in green point cement layer. Arrows in blue point the finish line location.*

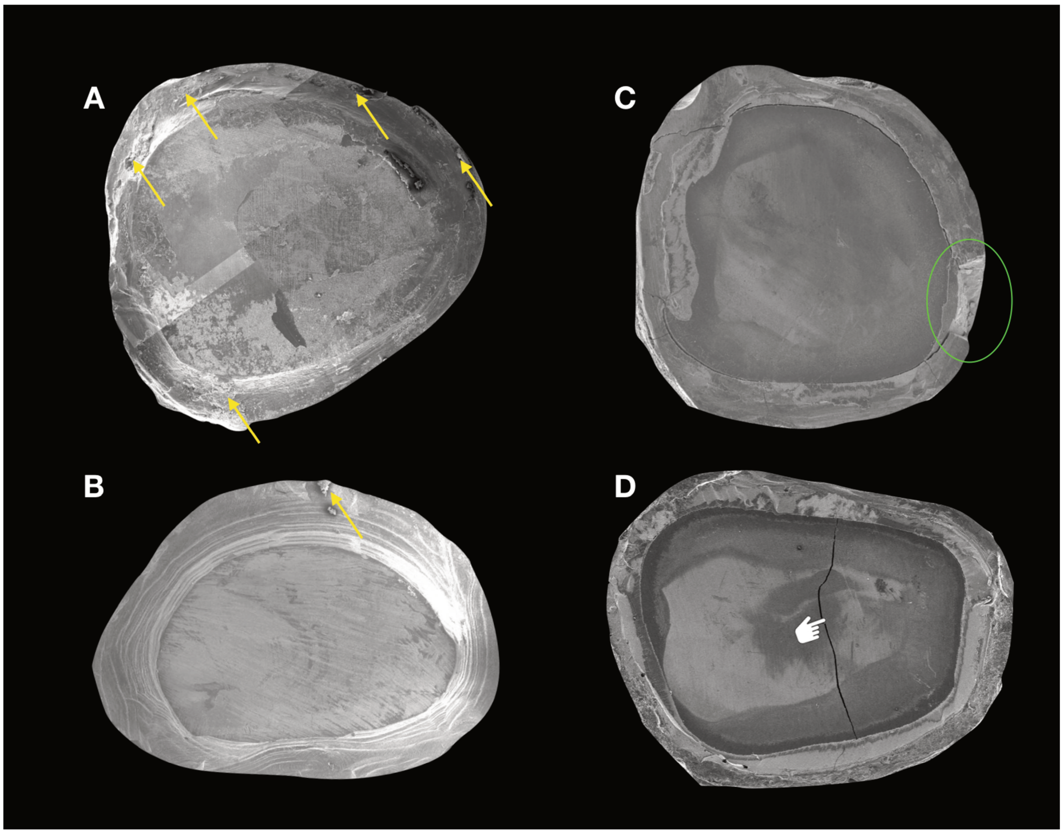

Figure 4 *Representative images of different types of failure mode. A and B- Adhesive failure. C- Cohesive in resin; D- Mixed. Arrow pointing the cement present; green circle pointing the cohesive fracture and hand pointing the teeth fracture.*

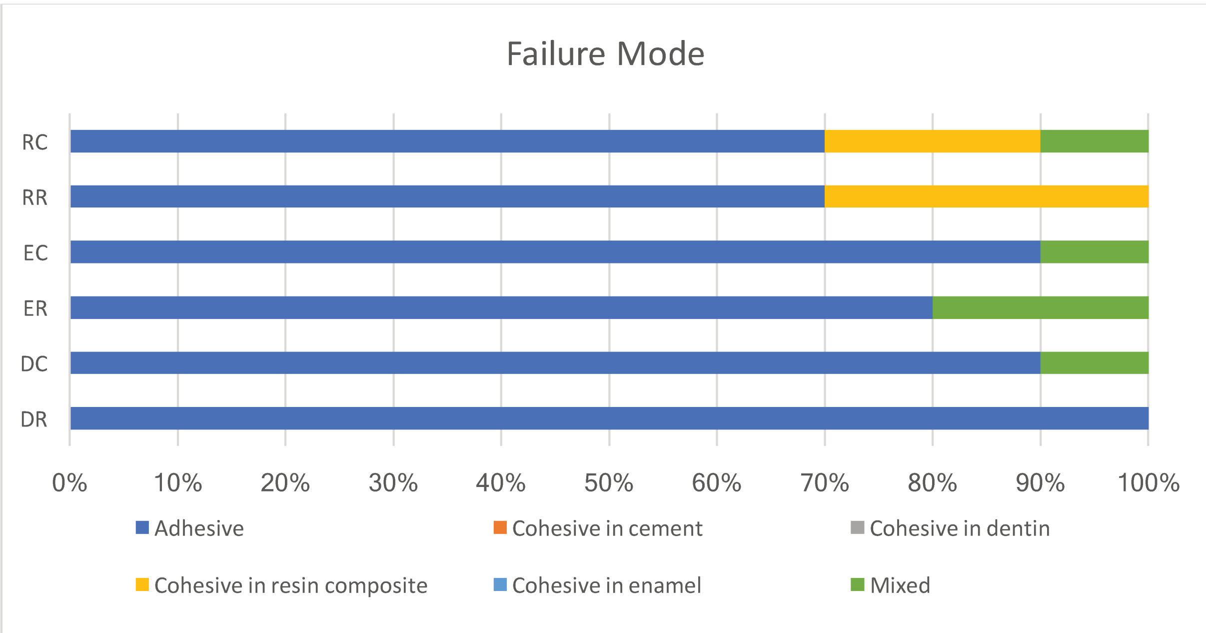

Figure 5 *Percentage of failure mode observed on groups.*

DISCUSSION

The results revealed the presence of group-wise differences indicating that the location of the marginal finish line influenced the tensile bond strength, marginal adaption, and nanoleakage; therefore, our hypothesis was rejected.

The results of the tensile bond strength test indicated that the groups with enamel finish line had better performance, followed in order by those with dentin and resin composite. This phenomenon may be related to the hybridization process involving a network of cross-links (copolymerization) between monomers and those with the hydroxyapatite within the dental structure.[10]

The self-adhesive cements comprise acid monomers, such as carboxylic acid and hydroxyethyl methacrylate (HEMA), which through partial etching of the smear layer surface create micromechanical retentions.[22] In addition, there are other monomers, such as phosphate ester, that mediate chemical bonding with the hydroxyapatite, which remains in the dental tissue after partial removal of the smear layer.[8, 23] Therefore, the bonding process of these materials is more chemical than mechanical.[24] Due to the effect of partial etching and higher viscosity presented by the self-adhesive cement versus that of the conventional cements, it is more difficult for the monomers to penetrate the tubules.[24-26] However, a report has indicated that the treatment of the enamel's surface with phosphoric acid at 37% prior to the application of self-adhesive cement increased the strength of adhesion.[27]

The diffuse orientation of the enamel's prisms presents difficulty in terms of etching and penetration of the substrate for the self-adhesive cement monomers.[27] Surface etching with phosphoric acid at 37% improves the topography, and changes the superficial tension of the surface; therefore, the cement is attracted by capillarity to the pores of the enamel which facilitates mechanical retention in addition to chemical bonding.[26] Moreover, the mineral content of the enamel [inorganic component (96% of weight) comprising calcium phosphate, fluorapatite, carbon apatite; water (3%); organic matrix comprising protein matrix (1%)], necessitate surface etching for adequate adhesion.[25] Results of the tensile bond strength test in our study corroborate those of previous studies, indicating that this protocol increases the resistance of the adhesive interface for self-adhesive cement with enamel.[26-28]

In contrast, the complex composition of dentin [inorganic component comprising intertubular dentin (50% of total composition); organic matrix comprising collagen, phosphorus, and glycosaminoglycans (30%); water (20%)] presents an ongoing challenge for the use of self-adhesive cements.[8] The acid monomers of the self-adhesive cement enable etching of the smear layer and produce microretentions of 2-μm size.[22,23] In addition, removal of the partial smear layer is an advantage, due to the presence of a greater amount of hydroxyapatite in the collagen network, which leads to an increased number of bonds between the monomers, and consequently, an increase in the bond strength of the interface.[23]

A study investigating the retention of three different resin cements reported values of 2.9 - 3.9 MPa, similar to those of the tensile bond strength in the present study using similar methodology; the discrepancy between our results and those of other studies could be due to differences in the methodology and composition of materials used.[29]

With regard to marginal adaption, the groups with enamel finish line also presented better performance, followed in order by the dentin and resin composite. The enamel submitted to acid etching undergoes changes in topography which alters the surface tension; consequently, there is better flow of cement through the surface and better seating of the crown.[30,31]

Some reports have indicated that the preapplication of phosphoric acid at 37% to dentin preceding the application of self-adhesive cement did not improve the mechanical properties, due to the difficulty of the self-adhesive cement of high viscosity to permeate the retentions created in the collagen network,[28] which could explain the result in our study of poorer performance of the groups with dentin as finish line location (DR and DC) compared to those of the enamel groups.

It was used resin cement as a simplified adhesive system of self-etching adhesive in a single vial to mediate bonding to the internal surface of the crown, which are both of acidic nature.[30,31] Due to interaction with oxygen during the cementation process, the acid groups in the unpolymerized layer of the adhesive compete with the peroxides of the cementing agent, for the aromatic tertiary amines, creating an acid-base reaction between the adhesive and cement; this reaction has an effect to decrease copolymerization to below adequate level, resulting in a change in the contraction of the resin cement, and thereby, increased values of the cementation line.[32-34] In addition, the resin composite finish line is without phosphoric acid-mediated surface retentions, and hence, the cement bonding is purely chemical which compromises the seating of the crown[35]; consequently, the marginal adaption for the finish line location of resin composite in the RR and RC groups had the worst performance among all groups.

Internal etching of the crown is another important factor for the success of cementation; for mainly lithium disilicate-based ceramic crowns, application of hydrofluoric acid at 5% for 20 seconds achieves excellent etching on the inner surface, due to the ratio of crystals in the glassy matrix of this material[13,14,36,37]; whereas, the resin composite crown has the poorer settlement during cementation and inferior bond strength compared to ceramic, due to the lack of free radicals that allow chemical bonding with the cement monomers.[38] These findings corroborate the results in the present study of superior tensile bond strength in all groups cemented with ceramic crowns versus those cemented with resin composite crowns.

The values obtained through the marginal adaption test corroborate those previously reported for resin cements (180µm - 380µm) according to the composition of each material and technique used during cementation (finger pressure or constant load of 5 kg)[39-41]; the exception was the group with finish line location and crown in resin composite that presented the highest value indicating the poorest performance and can be explained by the interaction of the self-adhesive cement and self-etching adhesive.

The water content of dentin is necessary for the action of self-adhesive cement to release the middle hydrogen ions allowing demineralization of the smear layer by the acid monomers, which can be reused in the reaction between the monomers of phosphate (multifunctional acid) and particles of alkaline charge.[42-44] The acidic property of self-adhesive cement due to high concentrations of acid monomers during polymerization is neutralized by reaction between phosphate groups and alkaline charged particles and hydroxyapatite.[43,44] When neutralization is complete, the cement becomes more hydrophilic, which leads to increased wettability on the surface, and higher susceptibility of the interface to hydrolysis.[43] The action of self-adhesive cement and water concentration of dentin explains the results obtained through the nanoleakage test of poorest performance for the groups with dentin as finish line location (DR and DC) and better performance for the groups with the enamel finish line location (ER and EC) and resin composite (RR and RC), due to their characteristics of lower water content and absence of water.[22,40]

Previous studies have classified the case of remaining cement in both the tooth and crown as cohesive failure, which was observed in only the RR and RC groups of our study.[45,46] Adhesive failure was the most frequent failure type in our study, which indicates that the bond strength of the cement to surface is less than that of the cement to crown and the presence of debonding on the surface of the ceramics/resin composite such as failure of the bonding crown/cement bond. [45,47] This finding may be due to the lack of silane; reports have indicated that the acid monomer present in the universal adhesive influences the effect of silane to promote instability of the bond.[47-51]

The self-etching adhesive used as a bonding agent in our study comprises a combination of silane, HEMA, 10-methacryloyloxydecyl dihydrogen phosphate (MDP), and bisphenol-A diglycidyl ether dimethacrylate (Bis-GMA) in a single vial.[47] Adhesives with the

MDP component provide a reliable bond between the crown material and tooth surface.[48-50] The combination of silane with MDP monomer increases the level of cross-linkages with the methacrylates groups and also improves wettability of the surface, which results in the improved adhesion mechanism of the system.[50] However, studies have indicated the absence of significant differences between the use of silane alone and single-vial universal adhesive[50]; moreover, single-vial universal adhesives have problems associated with the instability of silane in solution on contact with MDP and bis-GMA.[51] In acidic media with the presence of water and MDP, the reactions of the silanol group would result in a decreased level of bond strength of the interface and resistance to infiltration.[51]

With regard to correlation analysis, the data did not pass the test of homoscedasticity, even though it passed the tests of normality; therefore, Spearman correlation analysis was performed. Spearman's correlation revealed a negative and high correlation between the tensile bond strength and marginal adaption ($\rho = -0.508$; $P < .001$). The groups with the highest tensile bond strength showed the lowest cementation line (group with enamel finish line location), and those with a higher cementation line showed lower bond strength (group with resin composite finish line location).

Although the enamel groups presented better performance in terms of the tensile bond strength and marginal adaption, the resin composite group showed close and satisfactory values. The evaluation of nanoleakage indicated better performance of resin composite versus enamel and dentin due to the difference in water composition of each substrate that has variable effect to lower hydrolysis, and consequently lower infiltration at the interface. This finding suggests higher stability of the interface for composite resin; study including thermomechanical aging of the samples is required to confirm this finding.

The present study highlights that the marginal finish line location has a direct influence on the crucial factors of crown cementation, such as the cement bond strength, marginal adaption, and nanoleakage resistance. The study was conducted using an *in vitro* model; nevertheless, the findings can be correlated with the clinical behavior of dental materials and structures and enable a guideline for clinicians regarding the location of the marginal finish line for cementation of the crowns.

CONCLUSION

It can be concluded that the preparation for crown can be performed safely in both enamel and dentin. The composite resin as finish line location presented promising results, however, further studies are still needed to regarding its indication; it may be an alternative to avoid subgingival terms. The restoration can be done safely in crowns made of composite resin or ceramic, with ceramic presented better performance, related to bond strength, marginal adaption and nanoleakage.

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3 Conclusão

Pode-se concluir que o preparo para coroa total pode ser realizado com segurança tanto em esmalte, quanto dentina. O término em resina composta apresentou-se promissor, porém ainda faz-se necessário maiores estudos quanto à sua indicação. Podendo ser uma alternativa para se evitar términos subgengivais. A reabilitação pode ser feita com segurança tanto em coroas a base de resina composta ou cerâmica, porém a cerâmica apresentou melhor desempenho, relacionado a resistência de união, adapatação marginal e nanonifintração.

O presente estudo foi realizado com o apoio da Coordenação de Aperfeiçoamento de Pesooal de Nível Superior (CAPES)- Código de Financiamento 001.

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Apêndice 1: Metodologia Detalhada

Materiais e Métodos

Delineamento experimental

Unidades experimentais: a unidade experimental foi considerada o elemento dental (60 terceiros molares humanos).

Fatores em estudo: 1) Localização do término do preparo marginal na cimentação de coroas unitárias: término em dentina; término em esmalte; término em resina composta 2) material reabilitador: coroa unitária em resina composta; coroa unitária em cerâmica de dissilicato de lítio em sistema injetado.

Variável resposta: resistência à tração (n=10); adaptação marginal (n=10); resistência à nanoinfiltração (n=10).

O projeto foi encaminhado para a avaliação e aprovação do Comitê de ética em pesquisa da Faculdade de Odontologia de Piracicaba / Universidade Estadual de Campinas, e se encontra aprovado pelo comitê (número do parecer: 2.077.723).

Preparo das amostras

Para a realização do teste de resistência à tração (n=10), teste de adaptação marginal (n=10), e teste de nanoinfiltração (n=10), foram utilizados 60 dentes (terceiro molar hígido humano), no total. O teste de adaptação marginal foi realizado previamente ao teste de tração, permitindo que a mesma amostra tenha sido utilizada para os dois testes. Já para o teste de nanoinfiltração, as amostras utilizadas foram confeccionadas com os fragmentos remanescentes do preparo das amostras para o teste de tração/adaptação marginal. No preparo, os dentes foram seccionados em cortadeira de alta precisão em 2mm de coroa remanescente, acima da unção amelo-cementária (Figura 1).

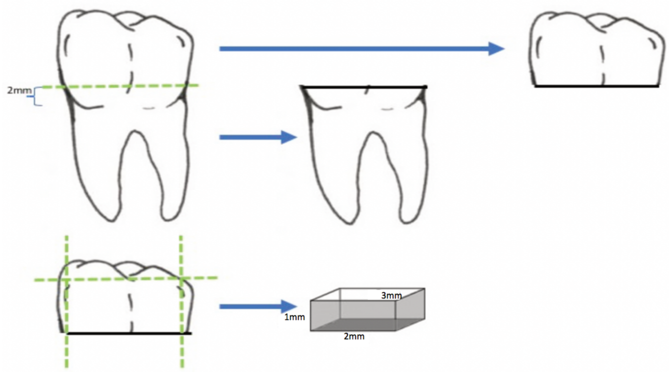

Figura 1 Ilustração da confecção das amostras.

Inclusão

Antes do processo de inclusão, o dente foi demarcado com caneta retroprojetor a 2 mm da junção amelocementária. O dente foi fixado com cera pegajosa, através da coroa, à haste de um delineador protético. A mesa móvel do delineador foi colocada perpfinish lineicularmente ao longo eixo do dente, e sobre esta, foi posicionado um cilindro de PVC com perfuração central de 10 mm, e 2mm de altura. A resina de poliestireno autopolimerizável foi manipulada e vertida no interior do cilindro de PVC. Após sua polimerização, o conjunto foi removido do suporte e os excessos foram removidos com lâmina de bisturi (Figura 2).

Os dentes utilizados foram de até 6 meses após a extração e seu armazenamento foi feito em água destilada 4°C. Utilizando-se ácido fosfórico 37 % por 30 segundos em esmalte e 15 segundos em dentina (Dentsply), seguido de lavagem com água constante por 30 segundos e secagem com papel absorvente. Foram aplicadas duas camadas de adesivo (Adapter Single Bond 2, 3M ESPE, St. Paul, MN, USA) como agente de união, com jato de ar para evaporação do solvente por 10 segundos e fotopolimerizado (Valo, Ultradent- Products Inc; South Jordan; UT; USA). Sequencialmente todos os dentes foram reconstruídos morfologicamente com resina composta microhíbrida (Filtek Z 250 XT, 3M ESPE, St. Paul, MN, USA) e fotopolimerizado por 20 segundos. Todas as amostras foram preparadas com término em chanfrado (confeccionados com broca tronco-cônica diamantada, 2135 KG Sorensen) em dentina, esmalte e resina composta (Tabela 2).

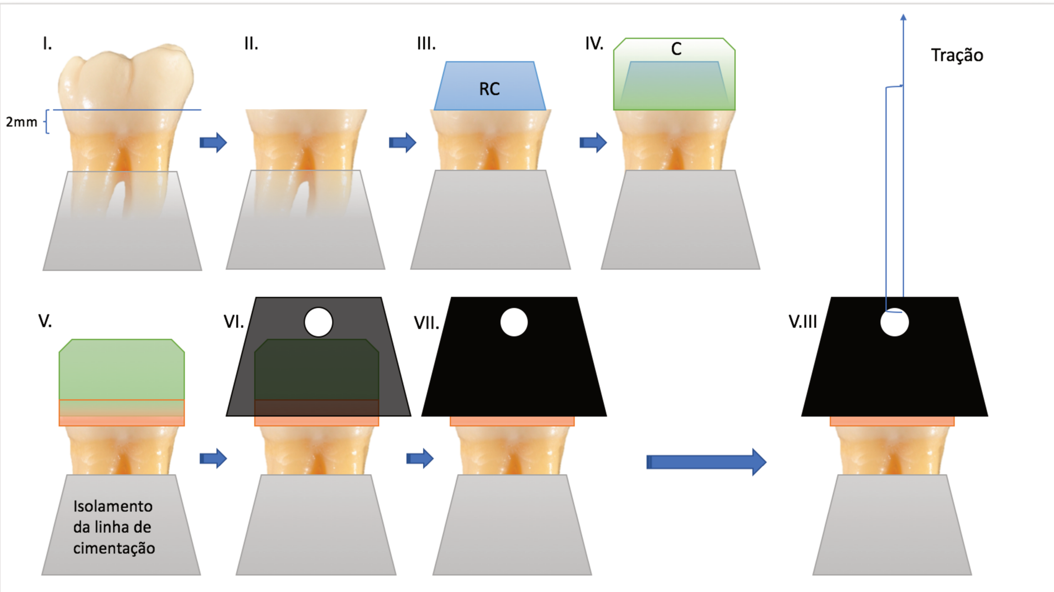

Figura 2 *I-Inclusão; II- seccionamento 2mm da junção amelo-cementária; III- Reconstrução com resina composta convencional; IV- confecção da coroa; V- isolamento da linha de cimentação; VI e VII- inclusão da coroa em resina; VIII- sistema complete para realização da tração.*

Os grupos foram determinados da seguinte maneira (Tabela 1):

Tabela 1 *Organograma da divisão dos grupos.*

DR	Término em dentina - Coroa em resina composta
DC	Término em dentina - Coroa em cerâmica
ER	Término em esmalte - Coroa em resina composta
EC	Término em esmalte - Coroa em cerâmica
RR	Término em resina composta - Coroa em resina composta
RC	Término em resina composta - Coroa em cerâmica

Tabela 2 *Materiais e especificações do fabricante.*

Material	Nome Comercial	Composição	Lote	Fabricante
Resina composta convencional	Filtek Z 250 XT	Bis-GMA, UDMA, BIS-EMA, PEGDMA, TEGDMA.	839067	3M ESPE (St. Paul, MN, USA)
Resina composta	Solidex	Superfície modificada de zircônia / sílica (3 ou menos) não aglomerada / não agregada 20 nanômetros de partículas de sílica modificada em superfície 68% em volume	81230	Shofu (Kyoto, Japan)
Dissilicato de lítio	e.max press	SiO_2 57-80, Li_2O 11-19, K_2O 0-1,3 P_2O5 50-11, ZrO_2 0-8, ZnO 0-8, Al_2O_3 0-5, Mg 0-5,	V38629	Ivoclar Vivadent (AG)
Cimento auto-adesivo	RelyX U200	óxidos corantes 0-8 (em% por peso) Pó de vidro, superfície modificada com ácido 2-propenóico, 2-metil-3-(trietoxissilil), éster propílico,	3191304	3M ESPE (St. Paul, MN, USA)

		material a granel, dimetacrilato substituído, sódio-p-toluenossulfonete, dimetacrilato de 1,12-dodecano, sílica tratada com silano, 2,4 , 6 (1H, 3H, 5H) pirimidinetriona, sal de 5-fenil-1 (fenilmetil) ccio (2: 1), hidrido de ccio, ido 2-propanco, 2-metil [(3-metoxipropil) imino] di-2, Éster 1-etanodiílico, amina metacrilada, dióxido de titânio		
Adesivo auto-condicionante	Single Bond Universal	MDP, resinas dimetacrilato, HEMA, copolímero vitrebond, carga, etanol, água, iniciadores e silano	639516	3M ESPE (St. Paul, MN, USA)
Adesivo convencional	Single Bond Adapter 2	Bis-GMA, UDMA, HEMA, copolímero de ácido acrílico, ácido iônico, partículas de sílica coloidal silanizada, etanol, água e fotoiniciador	N808310	3M ESPE (St. Paul, MN, USA)

* Cor A2 foi utilizada para os materiais resina composta e cimento resinoso auto-adesivo.

A coroa unitária em resina composta laboratorial, foi confeccionada seguindo as mesmas características da coroa unitária feita em cerâmica, e em seguida, levada ao fotopolimerizador de bancada (Figura 3).

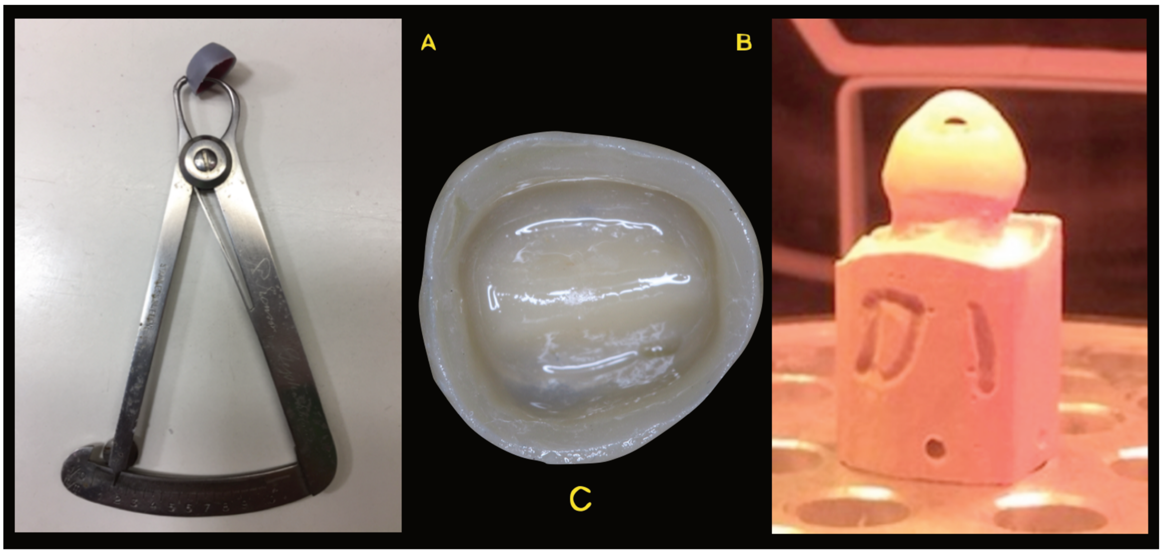

Figura 3 *A- Confecção da coroa unitária em cerâmica (padrão em cera, antes da inclusão); B-Coroa unitária em resina composta (em fotopolimerizador de bancada) C-Preparo (vista oclusal).*

Para a cimentação das coroas, foi utilizado um cimento resinoso autoadesivo (Relyx U 200, 3M Espe), seguindo instruções do fabricante e fotopolimerizando cada face por 30 segundos.

Tratamento de superfície interna das coroas:

- Coroa unitária em resina composta: aplicação de ácido fosfórico a 37%, seguido de irrigação por água, por 30 segundos cada e secagem completa da superfície. Sequencialmente, uma fina camada de adesivo universal, como agente de união (Scothcbond Universal, 3M ESPE, St. Paul, MN, EUA) e depois aplicado o cimento auto-adesivo.
- Coroa unitária em cerâmica de Dissilicato de Lítio: aplicação de ácido hidrofluorídrico a 5%, por 20 segundos, seguido de irrigação por água, por 30 segundos e secagem da superfície completa. Sequencialmente, uma fina camada de adesivo universal, como agente de união (Scothcbond Universal, 3M ESPE, St. Paul, MN, EUA) e depois aplicado o cimento auto-adesivo.

Em seguida a amostra foi incluída em resina de poliestireno autopolimerizável, em duas partes (inclusão da raiz do dente – base; inclusão da coroa - realização da tração), e levada à Máquina de Ensaio Universal - 4411, INSTRON (Figura 4).

- Término em esmalte: aplicação de ácido fosfórico a 37%,por 30 segundos seguido de irrigação com água, por 30 segundos e secagem da superfície completa. Sequencialmente a coroa unitária foi colocada com cimento autoadesivo e fotopolimerizado por 30 segundos.
- Término em dentina: não foi realizado condicionamento ácido prévio (conforme instrução do fabricante). A dentina foi irrigada com água por 30 segundos e apenas o excesso foi retirado com papel absorvente. Sequencialmente a coroa unitária foi colocada com cimento autoadesivo, fotopolimerizado por 30 segundos .
- Término em resina composta: aplicação de ácido fosfórico a 37%, por 30 segundos seguido de irrigação com água, por 30 segundos, e secagem da superfície completa. Sequencialmente, aplicou-se uma camada fina de adesivo autocondicionante universal, como agente de união (Scothcbond Universal, 3M ESPE, St. Paul, MN, EUA), em seguida, a coroa unitária foi posicionada com o cimento autoadesivo, fotopolimerizando por 30 segundos.

A cimentação das amostras foi feita pelo mesmo operador previamente calibrado, através da pressão digital. As amostras foram polimerizadas por 30 segundos cada face durante o processo de cimentação.

Sequencialmente, a região de cimentação foi isolada com cera número 7. A região em seguida foi isolada e a resina de poliestireno autopolimerizável foi manipulada e vertida na região, confeccionando-se uma alça de retenção em sua extremidade, finalizando um sistema que permitu a realização do teste em Máquina de Ensaio Universal - 4411, INSTRON (Figura 4).

Resistência à tração (n=10)

(Johnson GH, Lepe X, Patterson A et al., 2018)

O tracionamento foi feito no longo eixo do dente, em uma velocidade de 1 mm/min e força inicial de 30 N, e aumentando-se progressivamente até ocorrer o rompimento da linha de cimentação.

Foi utilizado o dente inteiro como amostra com o intuito de se chegar ao mais proximo de um comportamento clinico. A coroa clínica da amostra foi seccionada para evitar qualquer influencia da anatomia do dente no resultado, para avaliar apenas a influência do término na cimentação.

$$RT = F/A.M.$$

- *Onde:*
  - *RT = Resistência à tração (Mpa)*
  - *F = Força de ruptura da linha de cimentação (N)*
  - *A.M.= Área da amostra*

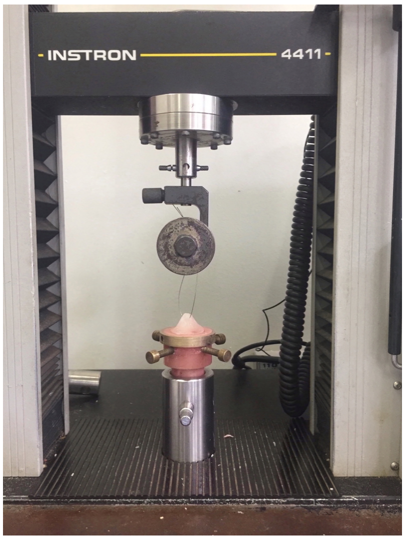

Figura 4 *Teste de resistência à tração.*

Padrão de Fratura

(Sadighpour L, Geramipanah F, Fazel A et al., 2018 - Modificado)

Após o teste de resistência à tração, o padrão de fratura das amostras foi classificado através de imagens obtidas em MEV (JEOL JSM-6610LV, MA, EUA). As amostras foram revestidas com uma delgada camada de ouro (Balzers-SCD 050 Sputter Coater, Liechtenstein) e foram avaliadas em Microscópio Eletrônico de Varredura - MEV (JEOL JSM-6610LV, MA, EUA), operando sob alto vácuo a uma potência de 15 KV. O padrão de fratura foi classificado como:

- Adesiva
- Coesiva em cimento
- Coesiva em dentina
- Coesiva em resina
- Coesiva em esmalte
- Mista

Adaptação Marginal (n=10)

(Al Hamad KQ, Al Rashdan BA, Al Omari WM et al., 2018 – Modificado)

Previamente ao isolamento da linha de cimentação na preparação das amostras para o teste de resistência à tração, as amostras foram moldadas nas faces palatina e vestibular com silicone por adição, e os modelos de resina epóxica foram confeccionados. Sequencialmente, todas as amostras foram montadas em stubs de alumíniob (Figura 5) para receber de revestimento com uma fina camada de ouro (Balzers-SCD 050 Sputter Coater, Liechtenstein) e foram avaliados em Microscópio Eletrônico de Varredura (Figura 6) - MEV (LEO 435 VP, LEO Electron Microscopy Ltd, Cambridge, Reino Unido) com um aumento de 50X e 150X. A avaliação foi realizada 24 horas após a cimentação das coroas unitárias e as imagens medidas no sofwear ImageJ.

Cada imagem (Figura 7) foi medida em 3 pontos (nas bordas e ao centro). Após todas as medidas, foi feita uma média de cada face e, em sequência, uma nova média entre as faces palatina e vestibular foi feita para obter-se a média final da amostra.

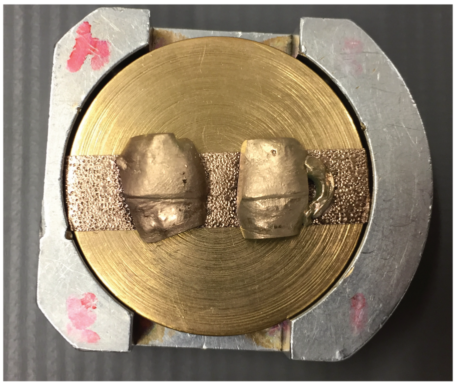

Figura 5 *Amostras metalizadas com ouro-paládio.*

Figura 6 *Microscópio eletrônico de varredura (MEV).*

Figura 7 *Mensuração da linha de cimentação (adaptação marginal).*

Resistência à nanoinfiltração (n=10)

(de Alexandre RS, Santana VB, Kasaz AC et al., 2014)

Para o teste da nanoinfiltração, foram confeccionados fragmentos (2mm x 1mm x 3mm), de dentina e esmalte, em cortadeira metalografica de precisão (à uma velocidade de 250rpm), sob refrigeração constante, e resina composta microhíbrida (Filtek Z 250 XT, 3M ESPE, St. Paul, MN, USA) fabricada em uma matriz (com base de teflon) previamente confeccionada, para serem as bases das restaurações posteriormente cimentadas (com resina e cerâmica) com cimento autoadesivo RelyX U200 – 3M ESPE, St. Paul, MN, USA (Figura 8).

Figura 8 Esquema de confecção das amostras para nanoinfiltração.

As amostras de cada grupo foram imersas em solução de nitrato de prata (composta por 10 gramas de cristais de nitrato, adicionados a 10 mL de água deionizada e posterior aplicação de gotas de hidróxido de amônio a 28%), por 24 horas em ambiente escuro à 37°C (Figura 9). Sequencialmente, foram lavados em água corrente por 2 minutos e imersas em solução reveladora (100 ml de água destilada + 10,9g de pó revelador), por 8h sob luminária fluorescente. Em seguida, as amostras foram lavadas com água destilada e incluídas em resina de poliestireno.

Figura 9 Imersão das amostras em solução de nitrato de prata e hidróxido de amônio.

Após inclusão, as amostras foram desgastadas em politriz com lixas d' água 600, 1200 e 2000, respectivamente, e polidos com discos de feltro e pastas diamantadas em granulação decrescente 3, 0,5 e 0,25µm. Entre cada granulação de lixa e de pasta, as amostras foram levadas à cuba de ultrassom por 10 minutos para remoção de detritos. As amostras foram secas com papel absorvente e, a seguir, receberam aplicação de uma solução de ácido fosfórico a 85% por 10 segundos para desmineralização, seguida de lavagem com água destilada. Para a desproteinização, foi utilizada solução de hipoclorito de sódio a 2% por 10 minutos. Em seguida, foi realizada a lavagem com água destilada e a secagem a temperatura ambiente. Posteriormente, as amostras foram desidratadas em álcool etílico em concentrações crescentes (50%, 75%, 90% e 100%) por 10 minutos em cada concentração.

As amostras foram montadas em *stubs* de alumínio e, depois, receberam um revestimento com uma camada delgada de carbono (Figura 10- BalTec SCD 050-

SputterCoater) para serem observadas em MEV, operando em alto vácuo numa potência de 20 KV, no qual foram obtidas imagens em elétrons retroespalhados (Figura 11). As imagens foram gravadas para avaliação quantitativa da área infiltrada pelo software ImageJ.

Foi medida a área total da amostra e a área infiltrada, em sequência uma porcentagem de infiltração da amostra foi calculada.

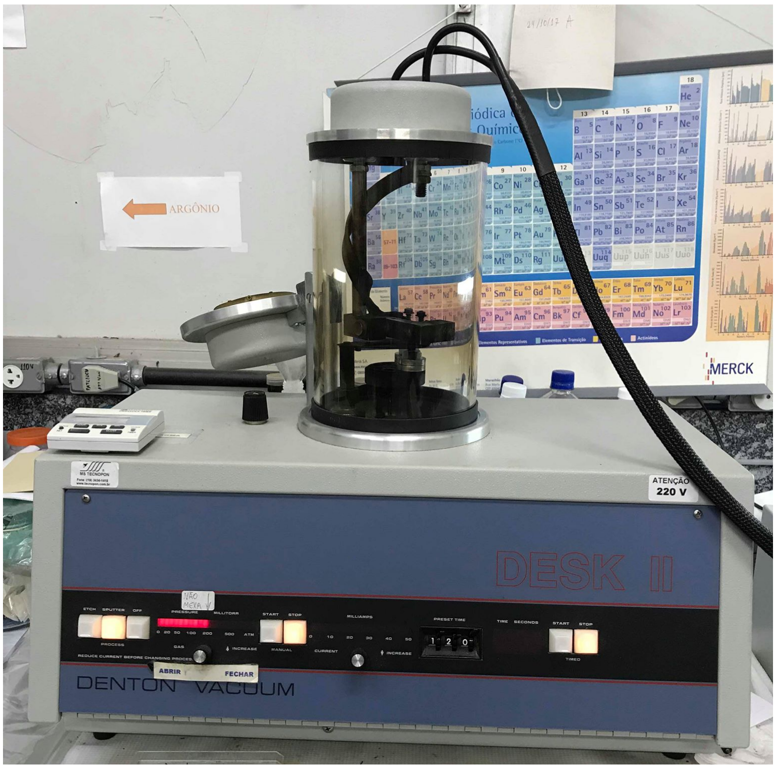

Figura 10 *BalTec SCD 050-SputterCoater.*

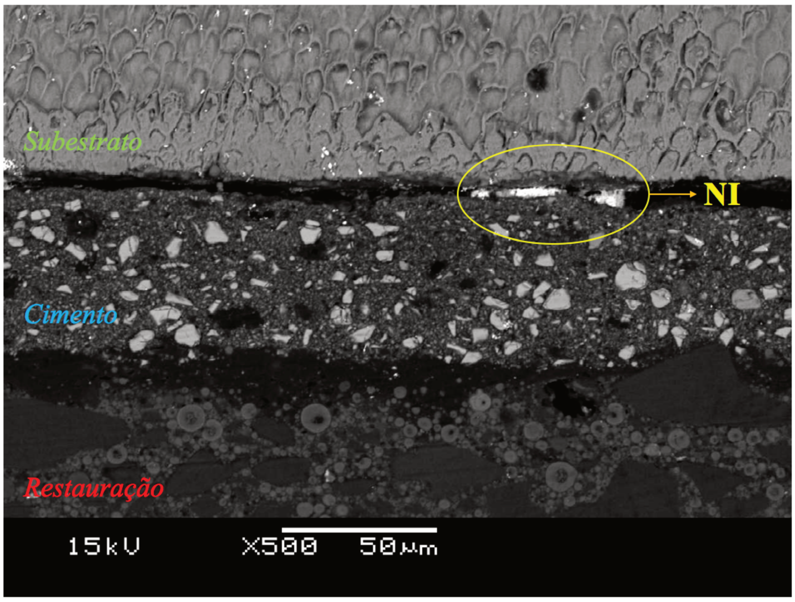

Figura 11 *Avaliação da nanoinfiltração (NI).*

Análise estatística foi feita utilizando teste de normalidade Shapiro-Wilk e Kolmogorov Smirnov e testes paramétricos Anova um-fator e Tuckey (post-hoc), com nível de significância de 5% ($p<0.05$).

Anexos

Anexo 1 – Certificado do Comitê de Ética

COMITÊ DE ÉTICA EM PESQUISA
FACULDADE DE ODONTOLOGIA DE PIRACICABA
UNIVERSIDADE ESTADUAL DE CAMPINAS

CERTIFICADO

O Comitê de Ética em Pesquisa da FOP-UNICAMP certifica que o projeto de pesquisa "Avaliação da adaptação marginal, tração e nanoinfiltração da cimentação de coroas unitárias em diferentes técnicas de preparo marginal", CAAE 66767417.6.0000.5418, dos pesquisadores Enrico Angelo e Luís Roberto Marcondes Martins, satisfaz as exigências das resoluções específicas sobre ética em pesquisa com seres humanos do Conselho Nacional de Saúde – Ministério da Saúde e foi aprovado por este comitê em sua versão original 23/05/2017 e na versão emendada em 24/09/2018.

The Research Ethics Committee of the Piracicaba Dental School of the University of Campinas (FOP-UNICAMP) certifies that research project "Evaluation of the marginal adaption, tensile strength and nanoleakage of the unitary crown cementation on different techniques of marginal preparation", CAAE 66767417.6.0000.5418, of the researcher's Enrico Angelo and Luís Roberto Marcondes Martins, meets the requirements of the specific resolutions on ethics in research with human beings of the National Health Council - Ministry of Health, and was approved by this committee on 23th ofMay of 2017)original version) and 24rd of September of 2018 (amended version).

Profa. Fernanda Miori Pascon	**Prof. Jacks Jorge Junior**
Vice Coordenador	Coordenador
CEP/FOP/UNICAMP	CEP/FOP/UNICAMP

Nota: O título do protocolo e a lista de autores aparecem como fornecidos pelos pesquisadores, sem qualquer edição.
Notice: The title and the list of researchers of the project appears as provided by the authors, without editing.

Anexo 2 – Teste anti-plágio (Turnitin)

Dissertação

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Anexo 3- Comprovante de Submissão do Artigo

Elsevier Editorial System(tm) for Journal of Dentistry
Manuscript Draft

Manuscript Number: JJOD-D-19-00133

Title: The finish line location of the cemented crown is an influencing factor for tensile bond strength, marginal adaption, and nanoleakage

Article Type: Full Length Article

Keywords: Keywords: tooth crown; cementation; ceramics; dental marginal adaptation; composite resins.

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Abstract: Objective: evaluated influence of finish line location of crown cementation on tensile bond strength, marginal adaption and nanoleakage, according to the following factors: finish line location (dentin, enamel, and resin composite) and restorative material (resin composite and ceramic). Methods: Sixty healthy third molars were collected. Tensile bond strength, self-adhesive resin cement was used. For marginal adaption, epoxy resin models were prepared. Prior to tensile bond strength test, images under scanning electron microscopy (SEM) were measured. Nanoleakage was measured using same protocol. Images of samples were measured under SEM. Failure mode was evaluated through SEM and classified: adhesive failure, cohesive in cement, cohesive in dentin, cohesive in resin composite, cohesive in enamel, and mixed. Statistical analysis was performed using Shapiro-Wilk and Kolmogorov Smirnov normality tests, two-way ANOVA, Bonferroni (post-hoc) parametric test, with significance level of 5% ($P < .05$), Spearman correlation test. Results: tensile bond strength was not statistically different between groups cemented with resin composite and ceramic. Cementation of ceramic was not statistically different between the groups (enamel, 3.28 MPa; dentin, 3.14 MPa; resin, 2.85 MPa). Marginal adaption was statistically different between resin and ceramic; finish line location varied between enamel and resin (175.91 m vs. 433.58 m). Nanoleakage rate was statistically different among all groups, except for resin: with resin (9.49%) and ceramic (9.35%). There was a predominance of adhesive failure in all groups. Conclusions: finish line location can be performed safely in enamel, dentin, and resin, as well as restoration material.

Clinical significance: With advances in techniques and resin composite-based materials, determining the influence of the marginal finish line location on dental structure is critical. This study aim to provide for clinicians a guide, based on science, related to preparation and cementation of metal free crowns under different finish line locations.