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Influence of different photoinitiators on the resistance of union in bovine enamel: an *in vitro* study

Influência de diferentes fotoiniciadores na resistência de união em esmalte bovino: um estudo *in vitro*

Influencia de diferentes fotoiniciadores en la resistencia de unión en esmalte bovino: un estudio *in vitro*

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Abstract

To evaluate the action of photoinitiators on the microtensile resistance of adhesive systems and resins composed in bovine enamel. Forty dental fragments, with flat and smooth enamel surfaces, obtained from the coronary buccal face of healthy bovine incisors, were randomly assigned to 4 experimental groups (n = 10) according to the different adhesive systems and composite resins used: Group 1- Ambar APS + Vittra APS; Group 2- Ambar APS + Opallis; Group 3- Ambar + Vittra APS; Group 4- Ambar + Opallis. After composite restoration, the samples were sectioned to obtain toothpicks that were subjected to the microtensile test (1.0 mm/min). The data obtained were subjected to statistical analysis. The Kruskal-Wallis test revealed no significant differences between groups ($p < 0.05$). Values in MPa were: G1- 18.58 MPa[a]; G2 - 19.83 MPa[a]; G3- 19.87 MPa[a]; G4- 20.99 MPa[a]. The result of the Mann-Whitney test showed no significant differences due to the adhesive (Ambar Universal- 19.58 MPaa, Ambar APS Universal- 19.87 MPaa) and the composite resin used (Vittra APS- 19.52 MPAa, Opallis - 20.56 MPAa). The use of different photoinitiators in the composition of adhesive systems and restorative composites did not influence their adhesive resistance values due to the standardization of the factors that influence their cure pattern.

Keywords: Dental enamel; Adhesives; Synthetic resins; Dental photoinitiators.

Resumo

Avaliar a ação de fotoiniciadores na resistência à microtração de sistemas adesivos e resinas compostas de esmalte bovino. Quarenta fragmentos dentais, com superfícies planas e lisas de esmalte, obtidos da face vestibular coronariana de incisivos bovinos hígidos, foram distribuídos aleatoriamente em 4 grupos experimentais (n = 10) de acordo com os diferentes sistemas adesivos e resinas compostas utilizadas: Grupo 1- Ambar APS + Vittra APS; Grupo 2- Ambar APS + Opallis; Grupo 3- Ambar + Vittra APS; Grupo 4- Ambar + Opallis. Após a restauração do compósito, as amostras foram seccionadas para a obtenção dos palitos que foram submetidos ao teste de microtração (1,0 mm/min). Os dados obtidos foram submetidos à análise estatística. O teste de Kruskal-Wallis não revelou diferenças significativas entre os grupos (p <0,05). Os valores em MPa foram: G1- 18,58 MPa[a]; G2 - 19,83 MPa[a]; G3- 19,87 MPa[a]; G4- 20,99 MPa[a]. O resultado do teste de Mann-Whitney não apresentou diferenças significativas devido ao adesivo (Ambar Universal- 19,58 MPaa, Ambar APS Universal- 19,87 MPaa) e à resina composta utilizada (Vittra APS- 19,52 MPAa, Opallis - 20,56 MPAa). O uso de diferentes fotoiniciadores na composição de sistemas adesivos e compósitos restauradores não influenciou seus valores de resistência adesiva devido à padronização dos fatores que influenciam seu padrão de cura.

Palavras-chave: Esmalte dentário; Adesivos; Resinas sintéticas; Fotoiniciadores odontológicos.

Resumen

Evaluar la acción de los fotoiniciadores en la resistencia a adhesivos microtensiles y resinas compuestas por esmalte bovino. Cuarenta fragmentos dentales, con superficies de esmalte planas y lisas, obtenidos de la cara bucal coronaria de incisivos bovinos sanos, fueron asignados aleatoriamente a 4 grupos experimentales (n = 10) según los diferentes sistemas adhesivos y resinas compuestas utilizadas: Grupo 1- Ambar APS + Vittra APS; Grupo 2- Ambar APS + Opallis; Grupo 3- Ambar + Vittra APS; Grupo 4- Ambar + Opallis. Después de restaurar el composite, las muestras se seccionaron para obtener los mondadientes que fueron sometidos al ensayo de microtensión (1,0 mm / min). Los datos obtenidos se sometieron a análisis estadístico. La prueba de Kruskal-Wallis no reveló diferencias significativas entre grupos (p <0,05). Los valores de MPa fueron: G1- 18,58 MPaa; G2: 19,83 MPaa; G3- 19,87 MPaa; G4 - 20,99 MPaa. El resultado de la prueba de Mann-Whitney no mostró diferencias

significativas debido al adhesivo (Ambar Universal- 19.58 MPaa, Ambar APS Universal- 19.87 MPaa) y la resina compuesta utilizada (Vittra APS- 19.52 MPAa, Opallis - 20 , 56 MPAa). El uso de diferentes fotoiniciadores en la composición de sistemas adhesivos y composites restauradores no influyó en sus valores de resistencia adhesiva debido a la estandarización de los factores que influyen en su patrón de curado.
Palabras clave: Esmalte dental; Adhesivos; Resinas sintéticas; Fotoiniciadores dentales.

1. Introduction

Adhesive systems have significantly revolutionized dentistry, making restorative procedures more conservative and longlasting (Cadenaro et al., 2019; Azad, Ata, Zandi, Shokrollahi & Solhi, 2018). The main objective of adhesive systems is to promote the retention of dental composites to the dental substrate, preventing failures and infiltration along the restoration margin (Cadenaro et al., 2019; Azad et al., 2018; Landuyt et al., 2007).

Their correct polymerization, before inserting the composite resin, provides a higher degree of monomeric conversion, causing satisfactory mechanical properties for the adhesion layer. Triggering this reaction requires the addition of primers, which are molecules with atomic bonds of low dissociation energy that will be consumed during polymerization (Landuyt et al., 2007).

Despite their high clinical acceptance, photoinitiator systems based on camphorquinone and amines have some limitations (Shin & Rawls, 2009; Segreto et al., 2016; Brandt, Schneider, Frollini, Correr-Sobrinho & Sinhoreti, 2010). Camphorquinone is a solid compound with an intense yellowish color and, when presented in high concentrations in composite resin formulations, causes an undesirable effect on the final aesthetic of the material polymerized. This reduces the amount of this photoinitiator in the composites, consequently limiting the degree of monomer conversion and the depth of polymerization (Schneider, Cavalcante, Prahl, Pfeifer & Ferracane, 2012; Brandt, Tomaselli, Correr-Sobrinho & Sinhoreti, 2011).

The degree of conversion is an important parameter that affects the final biological and physical properties of composites. Reduced conversion values result in decreased adhesive strength, hardness, and longevity of the material (Brandt et al., 2010. Schneider et al., 2012). The APS -Advanced Polymerization System was recently launched in the market, presenting a small amount of camphorquinone in its formulation used only to initiate a chain reaction with the multiplication of free radicals as sequential propagation occurs. Therefore, several initiators and co-initiators apart from the traditional ones exchange electrons and protons, producing the free radicals required for polymerization. Camphorquinone is only used to start the process, considering it is sensitized by the wavelength of the light emitted by the usual polymerizing agents. However, the performance of these systems regarding their adhesive properties, especially when used with composites without the same photoinitiation system has not been well established.

Thus, this study aimed to evaluate the microtensile strength of adhesive systems and composite resins with different types of photoinitiators when applied to bovine enamel. The hypotheses tested were: I) The APS photoinitiator system does not affect the microtensile strength of adhesive systems and composite resins in bovine enamel; II) The APS photoinitiator system does not affect the microtensile strength of adhesive systems and composite resins based on the same photoinitiator system or camphorquinone.

2. Methodology

Ethical Aspects

The research project was submitted to and approved by the Ethics Committee on the Use of Animals (CEUA) for Scientific Experimentation from the Health Sciences Center of the Federal University of Rio de Janeiro, RJ, Brazil. It was registered under protocol nº 009/19.

Materials

The following restorative materials were used in this study: 37% phosphoric acid FGM, Ambar and Ambar APS adhesive systems, Opallis and Vittra APS composite resins, and the Valo photoactivator. Table 1 describes the composition and manufacturers of the materials used in the study.

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Table 1. Trademark, components and manufacturers of the materials used in restorative procedures.

BRAND	COMPOSITION	MANUFACTURER	CLASSIFICATION
Opallis	Bis-GMA (Bis-Phenol A di-Glycidyl Methacrylate); BisEMA (Bis-Phenol A di-Glycidyl Ethoxylated methacrylate) TEGDMA (Triethylene glycol dimethacrylate) UDMA (Urethane dimethacrylate), camphorquinone, co-initiator silane. Inactive ingredients: silanized barium-aluminum silicate glass, pigments, and silica	FGM Joinville, SC Brazil	Nanohybrid Restorative Composite
Vittra APS	Mixture of methacrylic monomers, photoinitiator composition (APS), co-initiators, stabilizer and silane. Inactive ingredients: zirconia charge, silica, and pigments	FGM Joinville, SC Brazil	Nanohybrid Restorative Composite
Ambar	Active Ingredients: Methacrylic monomers, photoinitiators, camphorquinone, co-initiators, stabilizer. Inactive Ingredients: Inert charge (nanoparticles of silica), and vehicle (ethanol)	FGM Joinville, SC Brazil	Universal Adhesive System
Ambar APS	Active ingredients: MDP (10Methacryloyloxidecyl dihydrogen phosphate), methacrylic monomers, photoinitiators, co-initiators, and stabilizer. Inactive ingredients: inert filler (silica nanoparticles), and vehicle (ethanol)	FGM Joinville, SC Brazil	Universal Adhesive System

Preparation of dental samples.

Forty healthy bovine incisors (Frigorífico Mondelli, Indústria de Alimentos S/A, Santa Teresa, Bauru, SP, Brazil) were used. They were stored for a maximum of one month in a 0.1% thymol solution (UFRJ– CCMN- Department of Biochemistry, Rio de Janeiro- RJ- Brazil), with a pH of 7, for disinfection until the cleaning of external surfaces started. After cleaning with the Cavitron ultrasound system (Dentsply, RJ, Brazil) and periodontal curettes, the teeth were stored in distilled water until the surfaces were prepared. The root portion was separated from the crown through a section perpendicular to the long axis of the tooth, close to the amelocemental junction, made with a double-sided cutting diamond disc (KG Sorensen Ind. E Com. Ltda, Barueri, SP, Brazil) mounted on a straight piece.

After obtaining the dental fragments, it was necessary to adjust the palatal surface to make it parallel to the buccal surface. The palatal surfaces of the samples were sanded with a #320 silicon carbide (SiC) sandpaper (3M - 411Q 3M do Brasil - Sumaré, SP, Brazil) in a water-cooled rotating electric polisher (Aropol VV, Arotec, Cotia, SP, Brazil), until obtaining a flat surface parallel to the buccal surface. Subsequently, a part of the palatal surface of all teeth was removed through high-speed wear (Kavo do Brasil SA Ind. Com. Ltda, Joinville, SC, Brazil) with a #1014L spherical carbide bur (KG Sorensen Ind. And Com. Ltda, Barueri, SP, Brazil) to access and fill the pulp chamber with pink composite resin through the adhesive procedure. This procedure aimed to increase the thickness of the tooth in the thinner region of the buccal enamel to later facilitate the cutting of toothpicks.

Then, the buccal surfaces of the samples were sanded sequentially with #320 and #400 SiC sandpapers (3M do Brasil - Sumaré, SP, Brazil) in a water-cooled rotating electric polisher (Aropol VV, Arotec, Cotia, SP, Brazil) to obtain a flat enamel surface, with an average of two minutes for each sandpaper. The enamel surfaces were finished through manual wear with a wet #600 SiC sandpaper (3M do Brasil - Sumaré, SP, Brazil) for 15 seconds. The dental fragments remained immersed in distilled and deionized water for a maximum of 24 hours. At the end of this period, they were dried with absorbent paper, identified, and randomly distributed.

Experimental groups

Forty fragments of the teeth were distributed into four experimental groups, with 10 fragments in each group (n=10), and a part of the total number of toothpicks (tooth/resin) from 10 fragments of each group was submitted to the microtensile test.

Group 1: The 10 fragments of bovine teeth received 37% phosphoric acid etching (Condac 37 FGM Produtos Odontológicas Ltda, Joinville, SC, Brazil), Ambar APS Universal adhesive system (FGM Produtos Odontológicas Ltda, Joinville, SC, Brazil), and Vittra APS composite resin (FGM Produtos Odontológicas Ltda, Joinville-SC, Brazil).

Group 2: The 10 fragments of bovine teeth received 37% phosphoric acid etching (Condac 37 FGM Produtos Odontológicas Ltda, Joinville, SC, Brazil), Ambar APS Universal adhesive system (FGM Produtos Odontológicas Ltda, Joinville, SC, Brazil), and Opallis composite resin (FGM Produtos Odontológicas Ltda, Joinville-SC, Brazil).

Group 3: The 10 fragments of bovine teeth received 37% phosphoric acid etching (Condac 37 FGM Produtos Odontológicas Ltda, Joinville, SC, Brazil), Ambar Universal adhesive system (FGM Produtos Odontológicas Ltda, Joinville, SC, Brazil), and Vittra APS composite resin (FGM Produtos Odontológicas Ltda, Joinville-SC, Brazil).

Group 4: The 10 fragments of bovine teeth received 37% phosphoric acid etching (Condac 37 FGM Produtos Odontológicas Ltda, Joinville, SC, Brazil), Ambar Universal adhesive system (FGM Produtos Odontológicas Ltda, Joinville, SC, Brazil), and Opallis composite resin (FGM Produtos Odontológicas Ltda, Joinville-SC, Brazil).

During the experiment, all dental fragments remained in four plastic organizer boxes with 10 divisions each, one for each tooth, immersed in 25 mL of artificial saliva exchanged daily (UFRJ– CCMN- Department of Biochemistry, Rio de Janeiro, RJ, Brazil), and they were removed from this solution only for the application of the surface treatment.

Restorative procedures

Before performing the restorative procedures, which production sequence was randomized by draw, the dental fragments were removed from artificial saliva and maintained in closed plastic recipients under wet gauze (Johnson & Johnson Ind. And Com. Ltda, São José dos Campos, SP, Brazil), thus standardizing the degree of humidity of the dental surface.

a) Application of adhesive systems

Immediately before applying the surface treatments, excess water was removed by capillarity aided by an absorbent paper (Melitta do Brasil-Ind. E Com. Ltda, Avaré, SP, Brazil). Then, the restorative step was started with the proper treatment of enamel depending on the adhesive system used, according to the manufacturer's instructions.

Previously, the enamel was etched with 37% phosphoric acid (Condac 37, FGM - Joinville - SC - Brazil) for 15 seconds. Next, the surface was washed abundantly for 30 seconds and slightly dried with an absorbent paper to remove excess water.

b) Ambar APS Universal and Ambar Universal adhesive systems

The adhesive system was applied with a disposable micro-applicator (Cavibrush - FGM - Joinville - Brazil) - two layers on the enamel surface. The first layer was rubbed vigorously for

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10 seconds and the second, immediately after, for another 10 seconds. A light air spray was applied to evaporate the solvent. Next, polymerization was performed (Valo Cordless, Ultradent - Salt Lake City, Utah, USA) for 10 seconds in standard mode (1,000 mW/cm^2).

c) Insertion of composite resin

Immediately after applying the adhesive system to each dental fragment, the Vittra APS or Opallis composite resin block was produced in color A2, depending on the group. The composite resin was placed in a plastic shell-type meter to standardize the resin for each increment. The first increment, with 2.0 mm of thickness, was produced with the help of a metallic spatula for composite resin (Suprafill - Duflex/SSWhite- Rio de Janeiro, RJ, Brazil) and subsequently photoactivated for 40 seconds with the Valo Cordless device (Ultradent - Salt Lake City, Utah, USA). New increments were added to produce a rectangular block with approximately 10 mm of height, 8 mm of length, and 6 mm of width, with the same initial time and activation mode. This was followed by an additional final polymerization of 20 seconds in the mesial and distal portions at maximum power, always with the active tip of the polymerizer as close as possible to the material. The samples were then immersed in distilled water without the addition of antimicrobials, in which they remained for 24 hours.

Obtaining specimens for the microtensile test

The dental fragments were fixed by the palatal portion, individually, in acrylic plates, using utility wax (Horus, Herpo Produtos Dentários, Petrópolis, RJ, Brazil) to position the buccal surface parallel to the samples worn with the acrylic plate and later, sticky wax (NewWax, Thechnew Com. and Ind. Ltda., Rio de Janeiro, RJ, Brazil) for better fixation. This set was properly fixed and adapted to a precision metallographic cutter (IsoMet, BUEHLER Ltda. Lake Bluff, IL, USA), in which a metal disc (Extec Corp., Enfield, CT, USA), rotating at low speed (300 rpm), under constant water irrigation, sectioned the samples from the external surface of the composite resin block towards the crown, perpendicularly along the tooth axis, in which sections were made to obtain slices with approximately 1 mm of thickness each. Subsequently, sections were made perpendicular to the initial cuts to obtain toothpicks in parallelepiped shape with an adhesive area of approximately 1.0 mm² (Armstrong et al, 2017). The dimension of the prepared interface of tooth and composite resin was constantly assessed with the help of a digital caliper (Utustools Professional MT-00855, USA). Eppendorf tubes filled with distilled water were numbered according to each dental fragment and received all toothpicks obtained from the same tooth.

Mechanical test of microtensile strength

The sequence of fracture of the specimens was performed randomly. Each specimen was individually attached by its ends to the Geraldeli device (Odeme Biotechnology, Joaçaba, SC, Brazil), aided by a cyanoacrylate adhesive glue (Super Bonder - Henkel Loctile Adhesives Ltda, Itapevi, SP, Brazil) for the microtensile test (Armstrong et al, 2017). The apparatus was coupled to a Universal Testing Machine (Emic DL-2000, São José dos Pinhais, PR, Brazil - 20 kN) and the test was conducted at a speed of 1.0 mm/min until the specimens ruptured. At the time of fracture, the movement was immediately stopped. The load required to fracture each specimen, in kilogram-force (kgf), was noted and the dimensions of the adhesive interface of the specimens were measured with the help of a digital caliper (Utustools professional MT-00855, USA) to calculate the area. The values in Mega Pascal (MPa) were calculated by dividing the load at the time of fracture (N) by the area of each toothpick (mm^2).

3. Results

The results obtained in the microtensile test were tabulated and analyzed statistically in the IBM SPSS 22 software (IBM Corporation, Armonk-NY, United States) and the description of the groups was presented in Table 2. For all analyzes, the level of significance was 5% ($\alpha = 0.05$).

Initially, the histogram of the data was observed and the Shapiro-Wilk test was performed to assess the normality of the values (Figure 1).

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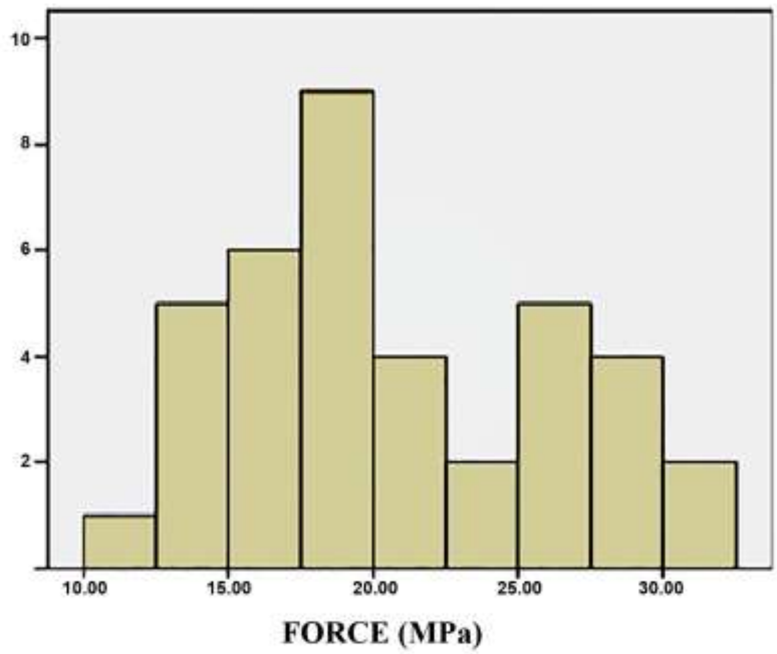
Table 2. Description of the groups evaluated in the study.

GROUP	Mean	Median	Minimum	Maximum	Standard deviation	N
G1- Ambar APS + Vittra APS	20.65	18.58	13.92	31.14	5.75	10
G2- Ambar APS + Opallis	21.03	19.83	14.59	29.90	6.24	8
G3- Ambar + Vittra APS	20.05	19.87	12.43	31.14	6.28	10
G4- Ambar + Opallis	21.55	20.99	16.57	27.37	4.36	10
Total	20.81	19.52	12.43	31.14	5.47	38

Source: Authors.

Figure 1. Strength histogram chart (MPa).



Source: Authors.

The visual evaluation of the histogram of strength results indicated a considerable deviation from normality (Mean = 20.81; Standard deviation = 5.472; N=38).

Furthermore, the result of the Shapiro-Wilk test (Shapiro-Wilk W = 0.93 p = 0.021)

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rejected the null hypothesis. Thus, it can be affirmed that, at 5% significance level, the data are not normally distributed.

Due to the deviation from normality, non-parametric tests were used to assess differences between the groups. When the analysis involved more than two groups, the Kruskal-Wallis test was used (Table 3). Considering there were no significant differences between the groups, no subsequent two-by-two analysis was performed (post-hoc).

The Kruskal-Wallis test ($p < 0.05$) accepted the null hypothesis that the groups have equal strength values. Furthermore, the observation of the Boxplot graph (Figure 2) shows similarities between the groups. For comparing two sets of data, different adhesives (Table 4) and different resins (Table 5), the Mann-Whitney test was used. The Mann-Whitney test ($p < 0.05$) accepted the null hypothesis, thus indicating no differences between the groups.

Table 3. Kruskal-Wallis test to assess the difference in strength between the groups (Force (MPa)).

GROUP	Mean	Median	Standard deviation	N
G1- Ambar APS + Vittra APS	20.65	18.58 a	5.75	10
G2- Ambar APS + Opallis	21.03	19.83 a	6.24	8
G3- Ambar + Vittra APS	20.05	19.87 a	6.28	10
G4- Ambar + Opallis	21.55	20.99 a	4.36	10
Total	20.81	19.52	5.47	38

Equal letters indicate no statistically significant difference between the groups by the Kruskal-Wallis test at 0.05% significance level. Source: Authors.

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Table 4. Mann-Whitney test for the comparison of adhesive systems (Force MPa). Mann-Whitney U = 186; p-value = 0.874.

ADHESIVE	Mean	Median	Standard deviation	N
AMBAR Universal	20.82	19.58 a	5.79	18
AMBAR APS Universal	20.80	19.87 a	5.32	20
Total	20.81	19.52	5.47	38

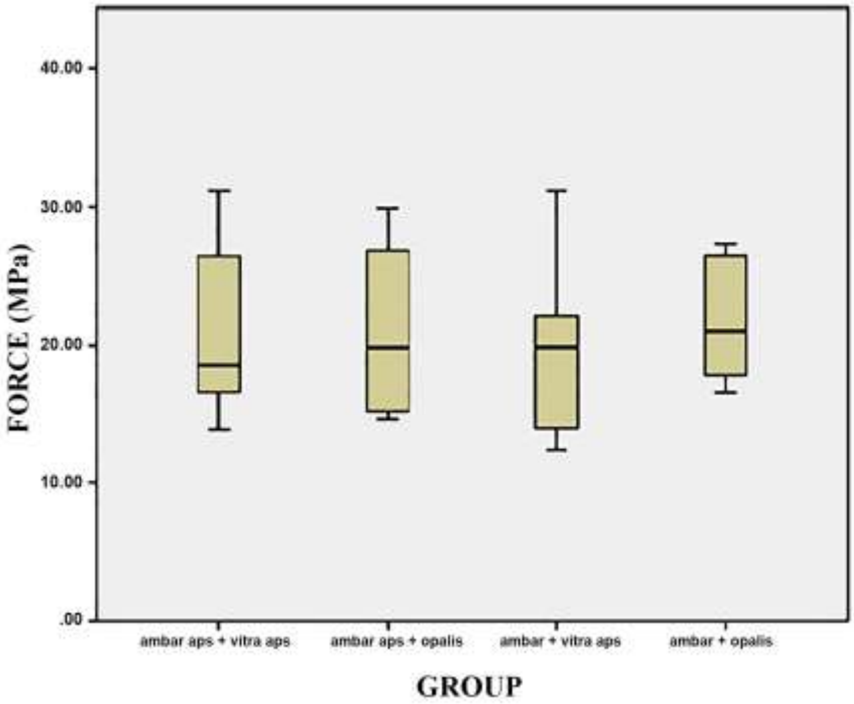
Equal letters indicate no statistically significant difference between the groups by the Kruskal-Wallis test at 0.05% significance level. Source: Authors.

Table 5. Mann-Whitney test for the comparison of composite resins (Force MPa). Mann-Whitney U = 197; p-value = 0.633.

RESIN	Mean	Median	Standard deviation	N
Vittra	20.35	19.52 a	5.87	18
Opallis	21.32	20.56 a	5.11	20
Total	20.81	19.52	5.47	38

Equal letters indicate that no statistically significant difference between the groups by the Kruskal-Wallis test at 0.05% significance level. Source: Authors.

Figure 2. Boxplot graph.



Source: Authors.

4. Discussion

Adhesive systems are materials that can adhere to the dental substrate and restorative composite resin (Cadenaro et al., 2019). The ability to bond restorations to enamel has fundamentally changed the concepts of cavity preparation, providing minimal wear of healthy dental structures, and favoring an infinite number of restorative treatment options (Cardoso, Zarpellon, Madruga, Rodrigues & Arrais, 2011; Armstrong et al., 2017; Yassen, Platt & Hara, 2011). Ideally, adhesion to the tooth structure should be simple to obtain and promote retentive strength, marginal sealing, and, above all, clinical durability. Despite all the developments in this field, bonding to the dental substrate continues to be the subject of several studies (Marshall, Bayne, Baier, Tomsia & Marshall, 2010; Rosa, Piva & Silva, 2015; Szesz, Parreiras, Reis & Loguercio, 2016).

To evaluate the influence of different factors on the bond strength of adhesive systems to enamel and to promote the improvement of adhesion, the application of tests that result in satisfactory data for the development of new materials and/or improvement of existing techniques is required (Armstrong et al., 2017). Mechanical strength tests are commonly used, such as microshear and microtensile tests, and both have advantages and limitations

(Armstrong et al., 2017). The microtensile test was chosen for this study because it uses parallelepiped-shaped specimens with reduced adhesive areas with a lower inclusion of defects, thus promoting a more uniform stress distribution in the interface. Additionally, this test was chosen because it is extensively used in the literature, which facilitates a more comprehensive understanding of the results (Miletic, Pongprueksa, Munck, Brooks & Meerbeek, 2013). However, the tooth was used as an experimental unit and not the toothpick.

Moreover, for *in vitro* studies of adhesive strength, bovine incisors have been used as a substitute for human teeth because they are morphologically similar and present similar values in bond strength tests applicable to the evaluation of the influence of various treatments to dental enamel (Ruse, Smith, Torneck & Titley, 1990). Due to its rough surface, planning was performed in this study to standardize the initial smoothness (Ruse, Smith, Torneck & Titley, 1990).

The most commonly used photoinitiator is camphorquinone associated with tertiary amine, (AlShaafi, 2017; Santini, Gallegos & Felix, 2013; Rueggeberg, Giannini, Arrais & Price, 2017) which is activated by the visible blue light over a wide wavelength spectrum ranging from 360 to 510 nm, with a maximum absorbance peak of 468-470nm, which is compatible with the vast majority of photopolymerizers in the market (Albuquerque, Moreira, Moraes, Cavalcante & Schneider, 2013; Arikawa, Takahashi, Kanie & Ban, 2009). When camphorquinone absorbs light, it interacts with the tertiary amine forming an excited complex. In this state, camphorquinone abstracts the hydrogen atom from the amine, producing free radicals that attack and break the double carbon bonds of the monomers, resulting in polymerization (Salles de Oliveira et al., 2016).

Although it is well accepted, the use of camphorquinone has some limitations, such as toxic effects from residual monomers and the extreme dependency on the tertiary amine content, which has reduced reactivity and can be neutralized by the low pH of self-etching adhesive systems, affecting directly the mechanical and optical properties of the material (Arikawa et al., 2009). One of the main disadvantages is the yellowish color, which affects negatively the final color of the polymerized resinous material, even in small quantities (Hass et al., 2013; Aguiar et al., 2015). This changes esthetic restorations, especially in bleached teeth, which need more translucent materials and less saturated colors. Additionally, camphorquinone is hydrophobic, which can reduce the degree of conversion of hydrophilic monomers (Hass et al., 2013; Aguiar et al., 2015).

More recently, the APS (Advanced Polymerization System) was launched in the market, consisting of a balanced combination of little color, allowing composites to present the same

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color before and after polymerization and longer working time because the APS has little sensitivity to ambient light. It also presents a synergistic combination of different photoinitiators, which allows the polymerization process of methacrylic monomers to occur more efficiently, increasing the degree of conversion of the polymers. Hence, several initiators and co-initiators different from the traditional ones exchange electrons and protons, producing the free radicals required for polymerization. Camphorquinone is used only to start the process because it is sensitized by the wavelength of light emitted by the usual polymerizing agents.

In this study, the hypothesis evaluated was that the APS photoinitiator system does not affect the microtensile strength of adhesive systems and composite resins based on the same photoinitiator system or camphorquinone. This hypothesis was fully accepted because the results obtained after the microtensile test showed no statistically significant difference between the groups tested (G1-18.58 MPa[a]; G2-19.83 MPa[a]; G3-19.87 MPa[a]; G4-20.99 MPa[a]) (Table 2). The same occurred when evaluating separately the different adhesive systems (Table 3) and composites (Table 4) used.

A high degree of monomeric conversion is vital to obtain satisfactory adhesive and mechanical properties of resinous materials. However, several factors affect polymerization. Among them stand out light intensity, exposure time, and adequate wavelength, which were completely controlled in the present study by standardizing the methodology, translated into the results obtained (Cadenaro et al., 2010).

In some situations, the polymerization of monomers does not occur directly on its surface, but indirectly on material or substrate such as enamel and dentin. When light strikes the material, several phenomena may occur, including absorption, reflection, and dispersion. This indirect polymerization significantly reduces the radiance of the light that reaches the monomeric material, and the degree of reduction depends on the composition, thickness, and translucency of this substrate (Cadenaro et al., 2010). In this study, enamel was used as a substrate, considering that, when compared to opaque dentin, the enamel has high translucency and little light refraction, which is more favorable for a greater intensity of light penetration. The high inorganic content of enamel provides unique translucency (Hass et al., 2013; Aguiar et al., 2015).

Moreover, enamel bonding reveals a great uniformity of results, such as the ones hereby presented, considering this fabric has a homogeneous structure composed mostly of minerals and very little water and organic matter (Swift, Perdigão & Heymann, 1997). However, dentin bonding is more difficult and less predictable due to its heterogeneous and physiologically dynamic structure, with 20% water in its total composition (Swift et al., 1997). Self-etching adhesives have a high content of hydrophilic monomers, which cluster before the polymerization reaction, forming hydrophilic areas and channels that allow the infiltration of water present in the dentin through the interdiffusion layer (Cadenaro et al., 2019). This water remains trapped inside the hybrid layer, impairing the monomeric conversion due to the presence of oxygen and the phase separation phenomenon (Hass et al., 2013). reducing adhesion values. Thus, the adhesion to the enamel was chosen so the substrate would not interfere with the results obtained.

Despite the results had not shown a statistically significant difference between the groups, the values presented in group 4 (20.99 MPa - Ambar Universal adhesive system + Opallis composite), which have camphorquinone in their composition, were numerically higher than the result obtained in group 1 (18.58 MPa - Ambar APS Universal adhesive system + Vittra APS composite). Although there is no evidence based on the literature that this was caused by the lower efficiency of the APS photoinitiator system when compared to camphorquinone, authors claim that camphorquinone is more effective in hydrophobic environments such as enamel, besides promoting the continuation of polymerization even in the absence of light, through a curing reaction in the dark (Cadenaro et al., 2019; Pongprueks, Munck, Barreto & Van Meerbeek, 2017), which is insignificant for current photoinitiator systems (Cadenaro et al., 2019). Additionally, an association of factors inherent to the application technique and composition of the Ambar APS Universal adhesive system, which presents water as a solvent in addition to ethanol, associated with the type, shape, and amount of filler in the Vittra APS composite, may have caused this numerical reduction in adhesive strength values.

The curing reaction is affected by the solvent contained in the adhesive. The ethanol in the Ambar Universal system increases the degree of monomeric conversion through the greater mobility of the polymer chain and the greater degree of diffusion of free radicals. In turn, the water contained in the Ambar APS Universal system, even when associated with ethanol, interferes with the polymerization process because it dilutes and reduces the reactivity of resin monomers. This worsens when the photoinitiator is camphorquinone, even in minimal amounts, due to its hydrophobic nature (Cadenaro et al., 2010).

Moreover, after its application, the solvent must be correctly volatilized with air. Any residual solvent not properly volatilized, especially water, interferes in a negative physical way with polymerization (Cadenaro et al., 2010; Hass et al., 2013). Besides the issues aforementioned, the presence of the solvent increases the porosities of the polymerized adhesive layer, causing and propagating microfractures, which reflects in the reduction of bond strength values (Pongprueksa et al., 2017). The manufacturer of the two adhesive systems evaluated suggests the application of two layers on the dental substrate, indicating the volatilization with air only after applying the second layer. This possibly did not affect the values obtained by the Ambar Universal system, considering the solvent in its formulation is ethanol, which has a higher vapor pressure than water and allows better evaporation in a shorter time (Hass et al., 2013). However, it may have interfered with the strength values of the Ambar APS Universal system due to the presence of water in the composition.

This numerical difference in values was not evident when the adhesive systems used were evaluated separately from the composite resins (Table 3, Ambar Universal- 19.58 MPa[a], Ambar APS Universal- 19.87 MPa[a]). Thus, it is suggested that the association with the Vittra APS resin may have affected the achievement of the lowest numerical bond strength value presented in group 1 (Table 2). This could also be observed when the composites used in this study were evaluated separately from the adhesive systems. The values described in Table 4 (Vittra APS composite - 19.52 MPa[a], Opallis composite - 20.56 MPa[a]), showed that, although there was no statistically significant difference, the results obtained by the Vittra APS composite were numerically lower than those obtained by the composite Opallis.

The depth of polymerization and the degree of conversion are closely related to the ability of light to penetrate through the composite resin, which is determined by its translucency and the presence of load (Cardoso et al., 2017). Thus, the characteristics of the inorganic matrix in dental composites have a great impact on the polymeric conversion of these materials (AlShaafi, 2017; Aguiar et al., 2015). The type, size, and concentration of the load may greatly affect the ability of light to be transmitted through the composite resin layer (AlShaafi, 2017; Aguiar et al., 2015). The Vittra APS resin is a nanoparticulate and radiopaque composite with a load composed of the nanospheres of a zirconia complex, with an average particle size of 200 nm and total load content of 52 to 60% in volume. The manufacturer states that this material has a degree of conversion around 50 to 60% of the total, which is considered satisfactory. The Opallis resin, in turn, is a nanohybrid composite, with barium-aluminum silicate glass particles combined with silicon dioxide nanoparticles, ranging from 40 nm to 3.0 μm, with an average size of 0.5 μm and lower load volume when compared to the previous resin, and with unparalleled optical properties according to the manufacturer.

It is suggested that the greater the load size in the composition of composites, the lower its concentration and, consequently, the greater the volume of the resinous matrix, which results in a greater penetration of light through the material, increasing polymerization and providing greater translucency (Lima et al., 2007). Moreover, the presence of radiopaque zirconia nanospheres may have caused a lower degree of monomeric conversion to the Vittra APS resin, resulting in lower numerical retention values when compared to the Opallis resin.

Conversely, the standardization of other factors that directly affect the curing of the material was essential to obtain more homogeneous strength results in all groups evaluated. Thus, translucent enamel composites of color A2 (AlShaafi, 2017). were applied to the substrate in 2-mm thick layers, (Salles de Oliveira et al., 2016) polymerized with standardized exposure time and with the shortest possible distance from the active tip of the photopolymerizer to the material (AlShaafi, 2017). Thus, the lighter color of the resins applied to thin layers may have favored the penetration of light in greater depth and quantity. The high light intensity and the correct irradiance level were also ensured by exposure time, proximity to the light beam, and type of the photopolymerizer used (Aguiar et al., 2015).

Among these factors, perhaps the most important was the wavelength and the light intensity provided by the photopolymerizer used in this experiment. Conventional LED devices have a narrower light spectrum, with a wavelength ranging from 400 to 500 nm, which coincides with the absorbance spectrum of camphorquinone, being more effective, producing less heat, and presenting a longer useful life than halogen light (Salles de Oliveira et al., 2016). However, alternative photoinitiators present the maximum absorbance peak at a shorter wavelength than camphorquinone, making these conventional "monowaves LED" devices unable to activate the polymerization reaction of such resinous materials. To solve this problem, a third generation of LED devices called "polywaves" was introduced in the market. These devices included an additional LED for emitting ultraviolet light, in the electromagnetic spectrum of 380-415nm, besides the LED that emits blue light (Arikawa et al., 2009). They present a greater range of photoinitiator systems, considering the wavelength of the light emitted may vary from 380 to 515 nm, with higher potential for activation.

Based on this information, for the standardization of this experiment and with the perspective of conducting other studies, the Valo Cordless device (Ultradent - Salt Lake City, Utah, USA) was used for the time indicated by the manufacturers of the materials evaluated, in the standard way. This device uses collimated light-emitting diodes of LED light to produce high-intensity light of approximately 1,000 mW/cm^2, with a wavelength ranging between 395 and 480 nm, and it is wide-ranging and sufficiently effective for curing the materials used in this study, greatly affecting the homogeneous pattern of the results obtained. Thus, the need for further studies becomes evident, changing the variables hereby evaluated, especially regarding the light source used, because it does not represent the reality found in the Brazilian public service and most dental offices.

5. Conclusion

Considering the limitations and standardization of this study, it was possible to conclude that:

- The presence of the APS photoinitiator system did not affect the microtensile strength values of adhesive systems and composite resins of bovine enamel when compared to camphorquinone-based systems;

- The APS photoinitiator system included in the adhesive system and the restorative composite did not affect microtensile strength values when associated with the adhesive systems and composite resins based on camphorquinone.

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