

PAOLA BERNARDES

**INFLUÊNCIA DOS PROTOCOLOS DE FINALIZAÇÃO DE  
PREPAROS PROTÉTICOS EM DENTINA NA ADESÃO À  
COMPÓSITO VITROCERÂMICO**

*Influence of finalization protocols for dentin prosthetic preparations  
on adhesion to glass-ceramic composite*

Dissertação apresentada à Faculdade de Odontologia da Universidade de Uberlândia, para obtenção do Título de Mestre em Odontologia na Área de Clínica Odontológica Integrada.

Uberlândia, 2023

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Orientador: Prof. Dr. Luís Henrique Araújo Raposo

Banca examinadora:

Prof. Dr. Luís Henrique Araújo Raposo.

Prof. Dr. Marcel Santana Prudente

Prof. Dr. Rafael Pacheco

Uberlândia, 2023



## ATA DE DEFESA - PÓS-GRADUAÇÃO

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## RESUMO

Este estudo teve como objetivo avaliar a influência de diferentes protocolos de acabamento em dentina pós-preparo protético na resistência de união ao compósito vitrocerâmico para CAD-CAM - Brava Block (FGM Dental Group, Joinville, SC, Brasil). Foram utilizados 126 terceiros molares humanos hígidos, nos quais a superfície oclusal foi seccionada e a superfície dentinária exposta foi padronizada com disco de lixas carbetto de silício (#600) e preparada com ponta diamantada cilíndrica (#3145, KG Sorensen, Cotia, SP, Brasil). Os espécimes foram divididos em 7 grupos (n=18) com diferentes instrumentos de acabamento: FB (pontas diamantadas F e FF), MB (brocas multilâminadas), UT (insertos ultrassônicos), MS (pedras montadas de óxido de alumínio e carbetto de silício), SB (pontas diamantadas sinterizadas), APA (jateamento com óxido de alumínio) e RC (selamento dentinário imediato com recobrimento de resina composta). Espécimes representativos foram analisados por microscopia eletrônica de varredura (MEV) e microscopia confocal à laser - LEXT para avaliação da rugosidade superficial e características da camada de smear layer depositada. Blocos do compósito vitrocerâmico para CAD-CAM - Brava Block (FGM Dental Group, Joinville, SC, Brasil) (14x14x5 mm) foram preparados e cimentados com cimento resinoso dual convencional (Panavia V5, Kuraray Noritake Dental, Tóquio, Japão) sob controle de pressão. Os espécimes resultantes foram armazenados sob umidade absoluta por 24 h e posteriormente seccionados em série perpendicularmente à superfície adesiva, obtendo palitos retangulares de cerca de 1 mm<sup>2</sup> de área adesiva. Esses palitos foram fixados em dispositivo específico de microtração e submetidos a uma tensão de tração com velocidade de 0,7 mm/min até a falha dos espécimes. Os resultados da análise de variância revelaram diferenças estatisticamente significantes na resistência adesiva entre os diferentes métodos de finalização do substrato. O grupo RC apresentou maior resistência de união, seguido dos grupos MS, APA e FB. Não houve diferença estatisticamente significativa entre os grupos APA e FB. O grupo com menor resistência de união foi o grupo MB, seguido dos grupos SB e UT. A análise da rugosidade demonstrou diferenças estatisticamente significantes entre os grupos. Os grupos FB, APA e RC apresentaram maior rugosidade e diferiram

estatisticamente dos demais. No entanto, não houve diferenças estatisticamente significantes entre esses três grupos. A análise da espessura de smear layer demonstrou diferenças estatisticamente significantes entre os grupos. O grupo FB apresentou a maior espessura de smear layer depositada na superfície dentinária, seguido pelo grupo APA e MB. Os grupos UT, MS e SB apresentaram menor espessura de smear layer e não apresentaram diferenças significantes entre si, porém diferiram dos demais grupos.

Em conclusão, os métodos de acabamento e finalização do substrato dentinário pós-preparo protético influenciaram a resistência de união, rugosidade superficial e formação de camada de smear layer.

**Palavras-chave:** Aderência Dental; Acabamento Dentário; Restauração Dentária Permanente; Preparo do Dente para Prótese.

## **ABSTRACT**

This study aimed to evaluate the influence of different post-prosthetic preparation dentin finishing protocols on the bond strength of the glass-ceramic composite for CAD-CAM - Brava Block (FGM Dental Group, Joinville, SC, Brazil). 126 sound human third molars were used, in which the occlusal surface was sectioned and the exposed dentin surface was standardized with a silicon carbide sandpaper disc (#600) and prepared with a cylindrical diamond bur (#3145, KG Sorensen, Cotia, SP, Brazil). The specimens were divided into 7 groups (n=18) with different finishing instruments: FB (F and FF diamond burs), MB (multilaminated burs), UT (ultrasonic diamond tips), MS (mounted stones of aluminum oxide and silicon carbide), SB (sintered diamond burs), APA (airborne-particle abrasion), RC (resin coating). Representative specimens were analyzed by scanning electron microscopy (SEM) and confocal laser microscopy - LEXT to evaluate the surface roughness and characteristics of the deposited smear layer. Glass-ceramic composite blocks for CAD-CAM - Brava Block (FGM Dental Group, Joinville, SC, Brazil) (14x14x5 mm) were prepared and cemented with conventional dual resin cement (Panavia V5, Kuraray Noritake Dental, Tokyo, Japan) under control of pressure. The resulting specimens were stored under absolute humidity for 24 h and then serially sectioned perpendicularly to the adhesive surface, obtaining rectangular sticks of approximately 1 mm<sup>2</sup> of adhesive area. These sticks were fixed in a specific microtensile device and subjected to a tensile stress at a speed of 0.7 mm/min until the specimens failed. The results of analysis of variance revealed statistically significant differences in bond strength between different substrate finishing methods. The RC group showed the highest bond strength, followed by the MS, APA and FB groups. There was no statistically significant difference between the APA and FB groups. The group with the lowest bond strength was the MB group, followed by the SB and UT groups. Roughness analysis showed statistically significant differences between groups. The FB, APA and RC groups showed greater roughness and statistically differed from the others. However, there were no statistically significant differences between these three groups. The analysis of the smear layer thickness showed statistically significant differences between the groups. The FB group had the highest thickness of the smear layer deposited on the dentin surface, followed by the APA

and MB groups. The UT, MS and SB groups had lower smear layer thickness and did not present significant differences between themselves, but differed from the other groups.

In conclusion, the methods of finishing and finalizing the dentin substrate after prosthetic preparation influenced the bond strength, surface roughness and formation of the smear layer.

**Key words:** Dental Bonding; Dental Polishing; Dental Restoration, Permanent; Tooth Preparation, Prosthodontic.

## 1. INTRODUÇÃO E REFERENCIAL TEÓRICO

As restaurações indiretas têm um papel crucial na reconstrução de dentes com danos extensos ou que precisam de alteração de forma significativa (Faus-Matoses *et al.*, 2014). Para esse tipo de restauração, é comum preparar o substrato dental utilizando pontas diamantadas ou outros instrumentos, a fim de criar espaço adequado para o material restaurador (Cardoso *et al.*, 2008). No entanto, esses procedimentos podem resultar em irregularidades na superfície preparada, exigindo métodos de acabamento que facilitem a moldagem, adaptação e fixação da restauração indireta. Com isso, métodos de acabamento que favoreçam os procedimentos de moldagem, adaptação e fixação da restauração indireta podem ser necessários (Al-omari *et al.*, 2001; Cardoso *et al.*, 2008; Ayad *et al.*, 2009; Gonzaga *et al.*, 2015).

A textura superficial e a área de preparo do substrato dental para restaurações indiretas desempenham um papel crucial na capacidade de união dos materiais de fixação às microrretenções presentes na superfície (Ayad *et al.*, 2009; Gonzaga *et al.*, 2015). Para garantir uma retenção mecânica adequada da prótese, é essencial que a textura superficial do preparo esteja regularizada, evitando-se, no entanto, um polimento excessivo (Horne *et al.*, 2011; Faus-matoses *et al.*, 2014). A rugosidade na superfície facilita a adesão da prótese ao agente cimentante e a molhabilidade da superfície (Ayad *et al.*, 2009; Lima *et al.*, 2021). No entanto, assim como a superfície não deve ser excessivamente polida para manter as microrretenções, a rugosidade do preparo não deve ser excessiva, pois isso pode resultar na retenção de ar na interface entre o agente cimentante e a estrutura dentária devido a uma camada irregular de adesivo (Al-omari *et al.*, 2001).

Durante o preparo dental, a smear layer é formada pela deposição de detritos na superfície da dentina, obstruindo os túbulos dentinários (Ayad *et al.*, 2009). Nos sistemas adesivos convencionais, o ácido fosfórico é utilizado para remover essa camada, aumentando a permeabilidade dos túbulos e expondo as fibras colágenas (Horne *et al.*, 2011), no entanto, essa maior permeabilidade pode causar sensibilidade pós-operatória. Os adesivos autocondicionantes são

uma alternativa, pois contém monômeros ácidos que removem parcialmente a smear layer, porém sua dissolução é limitada. Em casos de smear layer espessa, o conteúdo mineral pode neutralizar os monômeros ácidos dos adesivos autocondicionantes, resultando em menor resistência de união (Cardoso *et al.*, 2008; Niyomsujarit *et al.*, 2019). Portanto, é recomendado utilizar métodos de finalização do preparo dental que promova a remoção da smear layer sem descalcificar e danificar a superfície dentinária (Cardoso *et al.*, 2008).

## **2. PROPOSIÇÃO**

Avaliar diferentes protocolos de acabamento em dentina pós-preparo protético para determinar sua influência na resistência de união a um compósito vitrocerâmico.



## **1. CAPITULO 1**

*\*Artigo a ser submetido ao periódico Clinical Oral Investigations (Qualis CAPES A1)*

## **Influence of finalization protocols for dentin prosthetic preparations on adhesion to glass-ceramic composite**

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## ABSTRACT

**Objective:** This study evaluated different finishing protocols on dentin after prosthetic preparations to determine their influence on the bond strength to CAD-CAM glass-ceramic composite.

**Materials and methods:** Intact human third molars had their occlusal enamel removed, were standardizedly prepared, and divided into seven groups (n = 18) according to the finishing instruments: FB (F and FF diamond burs), MB (multilaminated burs), UT (ultrasonic diamond tips), MS (mounted stones of aluminum oxide and silicon carbide), SB (sintered diamond burs), APA (airborne-particle abrasion), RC (resin coating). Surface roughness and smear layer formation were evaluated using scanning electron microscopy (SEM) and confocal laser microscopy. Sectioned blocks of glass-ceramic composite (14x14x5 mm) were cemented with conventional dual resin cement (Panavia V5) under controlled pressure. The resulting specimens were stored under absolute humidity for 24 hours and then sectioned to obtain 1 mm<sup>2</sup> cross-sectional sticks, which were tested for microtensile bond strength.

**Results:** Significant differences were observed in bond strength among the tested groups based on the different finishing methods of the post-prosthetic preparation substrate. The RC group showed the highest bond strength values, followed by the MS, APA, and FB groups, while the MB group exhibited the lowest bond strength. Significant differences were also found in surface roughness among the groups, with FB, APA, and RC showing higher roughness values. There were no significant differences among the different regions within each group's specimens.

**Conclusions:** The finishing and finalization methods of the dentin substrate after prosthetic preparation influenced bond strength, surface roughness, and smear layer formation.

**Clinical relevance:** The appropriate selection of finishing protocols on dentin after prosthetic preparations proved to be relevant in improving the characteristics of this substrate, favoring the adhesion to indirect glass-ceramic restorations.

**Keywords:** Dental Bonding; Dental Polishing; Dental Restoration, Permanent; Tooth Preparation, Prosthodontic.

## Introduction

Indirect restorations play a key role in the reconstruction of teeth with extensive structural losses or that require shape changes (Faus-Matoses *et al.*, 2014). For this type of restoration, normally the dental substrate is prepared with diamond burs or other instruments in order to provide adequate space for the restorative material (Cardoso *et al.*, 2008). However, irregularities are created after prosthetic preparations and finishing methods that favor the molding, adaptation and fixation procedures of the indirect restoration may be necessary (Al-Omari *et al.*, 2001; Cardoso *et al.*, 2008; Ayad *et al.*, 2009; Gonzaga *et al.*, 2015).

Fine and ultrafine-grained diamond burs are the most commonly used instruments for finishing prosthetic preparations (Gonzaga *et al.*, 2015). However, due to the morphology of these instruments, which may present irregularities and heterogeneous arrangement of diamonds, a rough surface with high peaks and deep valleys can be created when preparing the dental substrate (Cardoso *et al.*, 2008). The excessive roughness of these substrates can hinder the uniform flow of adhesive systems and fixation agents and result in air trapping at the interface between the fixation material and the tooth structure, which can lead to further weakening of the bond (Al-omari *et al.*, 2001). Some *in vitro* studies, however, have shown that there is no correlation between enamel and dentin surface roughness and bond strength (Gonzaga *et al.*, 2015).

Another relevant aspect to be considered is the presence of a smear layer deposited on the dental substrate after prosthetic preparations, especially after the use of rotary cutting instruments (Cardoso *et al.*, 2008, Conde *et al.*, 2012). The smear layer is a layer composed of organic and inorganic residues from tooth preparation, and its presence can impair the penetration of adhesive agents into dental tissues and the formation of the hybrid layer (Cardoso *et al.*, 2008; Niyomsujarit *et al.*, 2019). When using self-etching adhesive systems, a thick layer of smear layer can buffer acidic monomers, resulting in lower bond strength to dentin (Cardoso *et al.*, 2008; Niyomsujarit *et al.*, 2019).

Aiming at a more homogeneous surface of prepared mineralized tissues, with reduction of the smear layer, studies have evaluated the use of different approaches in this sense (Cardoso *et al.*, 2008; Ayad *et al.*, 2009; Faus-matoses *et al.*, 2014 ; Rirattanapong *et al.*, 2015). Instruments such as ultrasonic inserts, multi-laminated drills and even sandblasting with aluminum oxide were used after cavity preparations (Cardoso *et al.*,

2008; Niyomsujarit *et al.*, 2019; Lima *et al.*, 2021). However, reports on the use of these and other techniques for finishing and finalizing prosthetic preparations for indirect restorations are scarce in the literature (Ayad *et al.*, 2009; Faus-matoses *et al.*, 2014; Gonzaga *et al.*, 2015). Doubts such as whether the use of several instruments can create a highly polished surface and excess smear layer, and whether these factors can influence the bond strength of indirect restorations to dentin.

Therefore, the aim of this study was to evaluate different post-prosthetic dentin finishing protocols to determine their influence on bond strength to a glass-ceramic composite. The null hypothesis generated was that different methods of finishing dentin after prosthetic preparations do not influence surface roughness, smear layer formation and bond strength of indirect glass-ceramic composite restorations to this substrate.

## **Materials and methods**

### *1. Specimen preparation*

A total of 126 freshly extracted sound human third molars for clinical or orthodontic indications were selected (Research Ethics Committee n° 4.938.277-FPM). The teeth were cleaned in running water with periodontal cures, decontaminated in 1.0% chloramine trihydrate for one week and stored in distilled water at 4 °C until the beginning of the experiments (for a maximum of 6 months), according to the ISO 11405/2015 standards. Dentin surfaces were exposed by removing the occlusal third of the teeth (2 mm deep) using a precision cutter (Isomet 1000, Buehler, Lake Bluff, IL, USA). The elements were evaluated under an optical microscope (LeicaMS5; Leica Microscopy Systems Ltda, Heerbrugg, Switzerland) with a 10× magnification and, when necessary, the surface was deepened in 0.3 mm increments until all enamel remnants were removed. Subsequently, the root portions were embedded in epoxy resin, 2 mm below the amelodentinal junction, to facilitate specimen handling. The dentin surface was then flattened and the smear layer standardized with silicon carbide discs (granulometry #600, Norton, São Paulo, Brazil) in a metallographic polisher (Arotec, São Paulo, SP, Brazil) under abundant irrigation, using circle for 60 seconds. All specimens received simulated coronary preparations with cylindrical diamond burs (#3145, KG Sorensen, Cotia, SP, Brazil), mounted in a multiplier contra-angle (1:5, WG-99 LT, W&H, Bürmoos, Austria) and an electric micromotor (EM 12-L, W&H, Bürmoos, Austria) with speed (40,000 RPM

× 5) and torque (0.6 Ncm) controlled. The handpieces were coupled to a device to control and standardize the touch of the instruments on the surface of the specimens, with movements performed 15 times for 60 seconds, keeping the active tip of the instrument in constant contact with the dental surface under abundant cooling. After preparation, the specimens were randomly divided according to the preparation finishing method (n=18): FB- F and FF diamond burs; MB- Multilaminated burs; UT- Ultrasonic diamond tips); MS- Mounted stones of aluminum oxide and silicon carbide; SB- sintered diamond burs; APA- Airborne-particle abrasion; RC- Resin coating. The preparations and steps of the finishing protocols were performed with new instruments for each specimen, as described in Table 1.

## *2. Characterization of instruments*

New instruments from each group were selected (n=1), cleaned in an ultrasonic tank with distilled water for 30 minutes to remove possible debris. Then, they were mounted on an aluminum stub and examined under a scanning electron microscope (VEGA 3 LMU, TESCAN, Libušina, Czech Republic). Photomicrographs were obtained with 100 and 300× magnification to evaluate the surface characteristics of the instrument's active tip and subsequent association with the characteristics of the substrate after use.

## *3. Analysis of surface roughness and smear layer formation*

After preparation and finishing, representative specimens of each experimental group were selected (n=4) and submitted to immediate analysis of the roughness parameters using confocal laser scanning microscope at 10× magnification (LEXT OLS 4100, Olympus, São Paulo – SP , Brazil). The measurement of surface roughness was obtained at 4 different points (A, B, C and D), which were equidistant and similar between the specimens. An average value of Ra (Sa) was obtained for each region, following the parameters defined by the ISO 4287/2002 standard. All specimens were prepared by the same operator and analyzes to calculate the mean surface roughness were carried out automatically using equipment-specific software.

Specimens (n=4) were prepared and cleaned in an ultrasonic tank with distilled water for 30 minutes to remove possible debris, followed by a protocol for dehydration

in ascending concentrations of ethanol (50°, 70°, and 95°) for 10 min in each and finally, the specimens were immersed in absolute alcohol for 30 min. Then, the specimens were stored in a container with silica for 48 hours to remove moisture, mounted on an aluminum stub, covered by spraying with a thin layer of gold and examined under a scanning electron microscope (VEGA 3 LMU, TESCAN). Photomicrographs were obtained with 200 and 1,000× magnification for qualitative evaluation and characterization of surface roughness (n=4). The specimens (n=4) were sectioned perpendicularly to the surface prepared for the characterization of the smear layer according to each group, using 5,000× magnification. The thickness of the smear layer was measured at 10 equidistant points along the cross section of each segment using the software (Image J, v. 1.8.0, NIH, USA).

#### *4. Microtensile bond strength ( $\mu$ TBS)*

Blocks (n=70) of glass-ceramic composite for CAD-CAM (Brava Block, FGM, Joinville, SC, Brazil) were sectioned (14x14x5 mm) in a precision cutter (Isomet 1000, Buehler) and blasted with 50  $\mu$ m aluminum oxide particles at a pressure of 5 Bar. Cleaning was performed in an ultrasonic tank with distilled water for 180 seconds, application of 70% alcohol and etching with phosphoric acid (K-ETCHANT, Kuraray Noritake Dental, Tokyo, Japan) for 5 seconds, followed by washing and drying the surface for 30 seconds. Then, a silane-type bonding agent (Clearfil Ceramic primer plus, Kuraray Noritake Dental) was applied with active friction for 20 seconds and drying with air jet for 30 seconds. For treatment of the dentin surface, the stored specimens were cleaned in an ultrasonic tank with distilled water for 5 minutes and after removing excess moisture, a self-etching adhesive system (Tooth primer, Kuraray Noritake Dental, Tokyo, Japan) was applied with active friction, waiting for 20 seconds and then a soft air jet is applied. Next, dual-cure resin cement (Panavia V5, Kuraray Noritake Dental, Tokyo, Japan) was manipulated with self-mixing nozzles as recommended by the manufacturer, applied to the glass-ceramic blocks and placed in position with continuous pressure in a device simulating digital pressure (20 N) for 5 minutes. Excesses were removed with an explorer probe and then photoactivation was performed for 40 seconds on each face using a wireless multipeak LED unit (VALO Grand, Ultradent, South Jordan, UT, USA).

After completing the fixation procedures, the resulting specimens were stored in distilled water at 37°C for 24 h. Then, they were serially sectioned, perpendicularly to the adhesive surface, using a precision cutter (Isomet 1000, Buehler) (Fig. 3 D) under

constant irrigation in order to obtain rectangular specimens with a cross section (adhesive area) of approximately 1 mm<sup>2</sup> and 8 to 10 mm long (stick-shaped). Only specimens obtained from the central regions of the teeth were used in order to eliminate strategic regional variability. The specimens were fixed at both ends of a specific device for microtensile use with universal adhesive based on cyanoacrylate (Super Bonder, Loctite, Itapevi, SP, Brazil), leaving the adhesive interface free (Fig. 3 E). Immediately after bonding, tensile stress was applied at a speed of 0.7 mm/minute until failure of the specimens in a specific machine for microtensile testing (Microtensile-Tester, Odeme Dental Research, Luzerna, SC, Brazil) (Fig. 3F). In case of loosening of the area where the specimens were attached to the microtraction device, they were fixed and tested again. After specimen failure, the maximum load value (Kgf) was recorded and their adhesive interface was measured with a digital caliper (200 Mm, Mitutoyo Corporation, Tokyo, Japan) and the specimens stored individually in Eppendorf-type flasks filled with distilled water. The maximum load was related to the adhesive area, thus defining the adhesive strength in MPa with the following formula: Tension max.: Load (Strength) max. / Adhesive area. In the case of pre-test failures, records were made and they were considered in the analysis of results.

##### *5. Failure mode*

All fractured specimens were evaluated under an optical microscope (LeicaMS5; Leica Microscopy Systems Ltd, Heerbrugg, Switzerland) with a 10× magnification and the failure mode was classified as: 1) cohesive in dentin, 2) cohesive in resin, 3) adhesive in resin, 4) adhesive in dentin, 5) mixed adhesive and 6) mixed (Raposo et al., 2012). Representative specimens were selected and coated with a gold/palladium layer and the fractured interfaces were analyzed in a high vacuum scanning electron microscope (VEGA 3 LMU, TESCAN) in order to interpret the individualities of the prevalent failure mode for each group. For selection, specimens that presented representative failure modes and with a bond strength close to the average for each group were used.

##### *6. Data analysis*

Normal data distributions for bond strength and roughness (Sa) were evaluated using Shapiro-Wilk and Levene tests. After verification of homoscedasticity, the data were submitted to analysis of variance (ANOVA) in two factors for surface roughness data and ANOVA in single factor for data on bond strength and smear layer thickness.



Next, the Tukey HSD test was used for multiple comparisons. The nominal categorical data of the failure mode of the specimens were submitted to the chi-square test. The significance level used in all analyzes was  $\alpha=0.05$  (95%), using a statistical package for the analyzes (JAMOV v.1.6, The JAMOV Project). Qualitative data obtained from SEM micrographs were analyzed in a comparative way.

## Results

### 1. Characterization of instruments

The comparative analysis of the photomicrographs performed in SEM of the instruments used in each experimental group, demonstrated distinct morphological characteristics for them. At 100 $\times$  magnification, it was possible to verify diamond particles heterogeneously distributed on the metallic rod and duly aggregated to the nickel matrix, which is exposed between the diamond grains for the FB group (Fig.1 - A and B). In the MB group, it was possible to notice that the first instrument used had an ogival-shaped active tip with transverse blades homogeneously distributed (Fig.1 - C); the second instrument has a right-angled end (90 $^\circ$ ) with a greater number of transverse blades, which are also homogeneously distributed (Fig.1-D). Instruments from the UT group have a robust active tip and a rounded active tip end, being integral and evenly coated with diamond crystals (Fig.1 – E and F). In the MS group, the instruments have a rounded shape and the active end is composed of a stone mounted on a metal rod, with a surface showing micro porosities (Fig. 1 – G and H). In the SB Group, the first instrument used has a conical shape with a rounded active tip end, irregularly shaped Diamonds homogeneously distributed on the surface (Fig. 1 – I); the second instrument used has a rounded tip, and diamonds with the same morphological characteristics as the first instrument used in this group (Fig. 1 – J).

In photomicrographs with 300 $\times$  magnification of the instruments, in the FB group (Fig. 2 A and B) an irregular and pointed shape of the diamond particles is observed. In MB (Fig. 2 – C and D), the slides have a right angle with slight slits at the ends and negative microbubbles can be observed on the surface of the second instrument used in this group (Fig. 2 – D). In UT (Fig. 2 E and F) a structure of rounded, robust diamonds

with standardized shape and size is observed, with no areas without diamonds. In the MS group (Fig. 2 – G and H), the surface has porosities with deep valleys. In SB (Fig. 2 – I and J), the instruments have irregularly shaped diamonds and it is not possible to visualize regions without diamonds.

## 2. Surface roughness and smear layer formation

### *Surface roughness (Laser Perfilometry)*

The mean values of surface roughness (Sa) for the experimental groups are described in Table 2. The 2-way ANOVA test showed significant differences in relation to the surface roughness presented by the experimental groups ( $p < .001$ ). There were no significant differences between the regions analyzed in the specimens (A, B, C and D) for each group ( $p = 0.585$ ) and no significant interaction between the groups and regions analyzed ( $p = 0.760$ ). In the post-hoc comparison using the Tukey HSD test, Groups FB ( $1.01 \pm 0.08$ ), APA ( $1.15 \pm 0.08$ ) and RC ( $1.25 \pm 0.53$ ) showed higher means of surface roughness, with no significant differences between them ( $p > 0.05$ ). The MB ( $0.66 \pm 0.12$ ), UT ( $0.74 \pm 0.07$ ), MS ( $0.69 \pm 0.10$ ) and SB ( $0.73 \pm 0.30$ ) groups had lower mean surface roughness and did not show significant differences between them ( $p > 0.05$ ), however, they differed from the other groups ( $p < 0.05$ ).

### *Surface roughness and smear layer formation (Scanning electron microscopy)*

The comparative analysis of the SEM micrographs showed different morphological characteristics on the dentin surfaces according to the post-prosthetic preparation dentin finishing protocols for each experimental group. The FB, UT, APA and RC groups showed greater surface roughness. The FB Group showed irregular grooves caused by the diamond tips, with high peaks and deep valleys. The UT Group demonstrated homogeneously distributed irregularities. The sandblasting carried out in APA promoted a large deposition of aluminum oxide on the surface of the smear layer. In

RC, the dentin covered by resinous material showed some adhered inorganic particles and bubbles on the surface. The MB, MS and SB images showed a smoother, more homogeneous surface with small irregularities (Fig. 3). The findings verified by SEM were similar to those observed by confocal laser microscopy (Fig. 4), with one analysis complementary to the other.

Differences were observed in the thickness of the smear layer in the SEM micrographs for the different experimental groups (Figs. 5 and 6). The finishing protocols used in the FB and APA groups resulted in a thicker smear layer, while the other groups, MB, MS, BS and UT, presented a thin and uniform smear layer with partially obliterated tubules. In the RC Group, the smear layer was not visualized because the dentin was covered by resinous material. Superficial microcracks were observed on dentin surfaces finished in MB. Although these cracks are not very evident in a superficial view, as they are covered by the smear layer, they become clearer in cross-section.

The mean thickness values ( $\mu\text{m}$ ) of the smear layer for the experimental groups are described in Table 2. The single-factor ANOVA test showed significant differences in relation to the smear layer thickness presented by the experimental groups after dentin finishing in the prosthetic preparations ( $p < 0.001$ ). In the post-hoc comparison using the Tukey HSD test, the FB group had a greater thickness of the smear layer deposited on the dentin surface ( $7.32 \pm 0.58$ ), followed by the APA group ( $7.29 \pm 0.52$ ) and MB ( $3.19 \pm 0.39$ ). The UT ( $2.76 \pm 0.19$ ), MS ( $2.47 \pm 0.27$ ) and SB ( $2.30 \pm 0.29$ ) groups had lower smear layer thickness and did not show significant differences between groups ( $p > 0.05$ ), but statistically differed from the other groups ( $p < 0.05$ ).

### 3. Bond strength ( $\mu\text{TBS}$ )

The mean values of bond strength (MPa) for the experimental groups are described in Table 2. The single-factor ANOVA test showed significant differences in bond strength for the different methods of finishing the dentin substrate after prosthetic preparation ( $p < 0.001$ ). In the post-hoc comparison using the Tukey HSD test, the RC group ( $32.03 \pm 3.94$ ) showed the highest bond strength values, followed by the MS ( $27.84 \pm 3.12$ ), APA ( $19.32 \pm 3.02$ ) and FB ( $18.8 \pm 5.13$ ) groups, and APA and FB did not

present significant differences between themselves ( $p>0.05$ ), but differed from the other groups. The MB group ( $15.27\pm 3.02$ ) showed the lowest bond strength values, followed by the SB ( $16.01\pm 3.22$ ) and UT ( $16.92\pm 3.11$ ) groups, with SB and UT not showing significant differences between them ( $p>0, 05$ ), but differed from the other groups.

#### 4. Failure mode

The failure analysis results are graphically summarized in Fig. 7. In the FB (70%), MB (76%) and UT (76%) groups, most of the failures occurred in an adhesive way between the resin fixation agent and the dentin substrate, making it possible, in some cases, to observe the dentin completely cement free. The APA (71%) and SB (65%) groups had a higher incidence of mixed adhesive failures, that is, most failures occurred within the bonding layer, with the cementing agent present in part of the dentin substrate and part in the indirect restoration. In the RC, MS groups, a high incidence of adhesive failures was observed with the indirect restoration, that is, the cementing agent used was released from the glass-ceramic material and was adhered to the dentin substrate. Only the MB group showed no incidence of cohesive failures, neither in dentin nor in resin.

## Discussion

The proposed null hypothesis that different methods of finalizing prosthetic preparation do not influence surface roughness, smear layer formation and bond strength of indirect glass-ceramic composite restorations to dentin was rejected. Significant differences were observed in bond strength, surface roughness and smear layer formation between the evaluated experimental groups.

Prosthetic preparations for indirect restorations are usually finished with fine-grained diamond burs or with polishing discs/rubbers (Gonzaga *et al.*, 2015). Theoretically, smoother surfaces are favorable in the molding step, and avoid the entrapment of bubbles in the adhesive interface, however there is still no consensus in the literature about how much the surface should be polished and whether the surface characteristics generated can influence the bond strength to indirect restorations (Al-Omari *et al.*, 2001; Cardoso *et al.*, 2008; Ayad *et al.*, 2009; Gonzaga *et al.*, 2015). In the

present study, the use of fine-grained (F) and ultrafine-grained (FF) diamond burs generated greater roughness on the dentin surface, when compared to multi-laminated burs, mounted silicon carbide and aluminum oxide burs, sintered diamond burs and ultrasonic inserts. When the surface was sandblasted with aluminum oxide or immediate dentin sealing was performed, there were no significant differences in terms of roughness when compared to diamond finishing burs (F and FF).

The characteristics of the instruments/methods used showed great influence on the surface characteristics created on the dentin substrate, as demonstrated by previous studies (Cardoso *et al.*, 2008; Horne *et al.*, 2011; Faus-matoses *et al.*, 2014). When instruments with an irregular shape and heterogeneous arrangement of diamonds were used (Group FB, Fig. 2 A and B), irregular grooves with high tops and deep valleys were created and a large amount of smear layer obliterating the entrance of the dentinal tubules was observed (Fig. 5 A). It is believed that the association of these factors may have negatively influenced the bond strength and may explain the high incidence of adhesive failures in dentin (70%). In the MS Group, the instruments used, as they were integral and had microporosities, created a smoother and more polished surface, with a thin and uniform smear layer, factors that possibly favored the formation of the hybrid layer and increased dentin bond strength /glass-ceramic composite.

Preparations finished with rotary instruments can present a surface three times more rough when compared to surfaces that have been finished with ultrasonic inserts and, as a consequence, greater chances of microleakage are reported (Horne *et al.*, 2011; Faus-matoses *et al.*, 2014). In addition to providing the preparation with a smoother and more regular surface, other advantages are reported for ultrasonic finishing, such as minimizing the possibility of iatrogenic damage to the adjacent tooth and gingival tissues (Faus-matoses *et al.*, 2014). However, because the ultrasonic inserts obtained by chemical vapor deposition have diamonds with a rounded and robust shape, they can induce greater surface tension in the dentin and generate microcracks, consequently making the substrate more irregular and prone to adhesive failures in bonding tests when compared to diamond burs (Cardoso *et al.*, 2008).

Sandblasting with aluminum oxide particles, a method also used to finish substrates, has been shown to increase surface roughness is sandblasting with aluminum oxide particles (Castro *et al.*, 2006, Lima *et al.*, 2021). When aluminum oxide particles, with high rigidity and hardness, affect the dentin surface with considerable pressure

generated in a specific propellant, it results in abrasion on the dentin surface (Lima *et al.*, 2021). Its indication is based on the principle that a rougher surface could cause better micromechanical interlocking between the dental substrate and adhesive systems, fixation agents and/or restorative materials or even improve the wettability of dentin surfaces (Lima *et al.*, 2021). However, when the dentin surface is abraded by this method, a thick layer of smear layer is deposited, which can negatively influence the bond strength to this substrate (Castro *et al.*, 2006).

A recent systematic review demonstrated that sandblasting with aluminum oxide had no negative effects on the bond strength of resin materials to dentin when compared to the roughened substrate with diamond burs (Lima *et al.*, 2021). In addition, in some cases the use of sandblasting was able to increase the bond strength to dentin, although only with particle sizes below 30  $\mu\text{m}$  and air pressure below 5 bar (Lima *et al.*, 2021). These findings are in line with the results of the present study, in which aluminum oxide particles of approximately 50  $\mu\text{m}$  and a pressure of 5 bar were used, demonstrating that the bond strength of the groups submitted to sandblasting or diamond burs were similar, as well as surface roughness. However, the APA group had a higher incidence of mixed adhesive failures (71%), that is, most failures occurred within the bonding layer, which may suggest better adhesion to dentin when compared to FB, which had a higher incidence of failures. dentin adhesives.

When the preparation was finished with multi-laminated carbide burs (tungsten carbide), the substrate showed less roughness when compared to the use of diamond burs. However, a significant amount of smear layer deposited on the dentin surface was observed, in addition to lower bond strength compared to all other experimental groups and a high incidence of adhesive failures in dentin (76%). Some studies have shown that the smear layer formed due to the rotation of multi-laminated burs can influence surface wettability and consequently reduce the bond strength to the dentin substrate (Ayad *et al.*, 2009; Cardoso *et al.*, 2015). In addition, the bond strength verified in the present study may have been influenced by the morphology of one of the instruments used in this group, since the confocal laser microscopy images associated with the micrographs performed by SEM showed a surface with specific regions presenting deep valleys with microcracks in peritubular dentin.

Immediate dentin sealing is a technique that seeks to create a hybrid layer by the immediate penetration of monomers into the prepared hard tissues, following the concept

of the resin coating technique with fluid composite (Magne *et al.*, 2005; Akehashi *et al.*, 2019). In this study, the Clearfil SE Bond 2 dentin adhesive system was used, containing the functional monomer 10-MDP (10-methacryloyloxydecyl dihydrogen phosphate), which promotes the modification and partial removal of the smear layer, with part of the smear layer being incorporated into the layer hybrid (Cardoso *et al.*, 2008; Niyomsujarit *et al.*, 2019). Studies have shown that the application of a low fluidity resin as a coating after application of the adhesive system dramatically increased the bond strength to dentin compared to the group where only the adhesive system was used as a dentin sealer (Santos-Daroz *et al.*, 2007; Akehashi *et al.*, 2019).

Dentin sealing aims at protecting dentin against bacterial infiltration and sensitivity during the temporary phase of treatment, based on the fact that temporary restorations may allow microleakage of bacteria and subsequently dentin sensitivity (Magne *et al.*, 2005; Carvalho *et al.*, 2021). Another advantage of immediate dentin sealing compared to the other techniques adopted in this study is that it does not require the use of special instruments or equipment to be used, just using restorative materials and appropriate protocols according to the technique (Carvalho *et al.*, 2021). This protocol is also independent of factors such as degradation of rotary instruments, speed/torque of handpieces or even refined manual skill so that adequate results are obtained, thus being an accessible and reproducible technique (Carvalho *et al.*, 2021).

Future studies should focus on determining the possible association of immediate dentin sealing techniques with resin coating with other methods that present adequate parameters for finishing the preparation for indirect restorations, in order to obtain a surface that is more receptive to bonding procedures.

## **Conclusion**

Within the limitations of this *in vitro* study, it can be concluded that:

1- Surface roughness, smear layer formation and bond strength of indirect glass-ceramic composite restorations to dentin were influenced by the different finalization methods of evaluated prosthetic preparations.

2- The increased roughness and smear layer generated by certain post-prosthetic preparation finishing protocols resulted in reduced bond strength of indirect glass-ceramic composite restorations to dentin.

3- The post-prosthetic preparation finishing protocol using immediate dentin sealing showed the best bond strength results of indirect glass-ceramic composite restorations to dentin.

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**Table 1.** Characterization of the materials and description of the protocols used in the finishing of the dentin substrate after prosthetic preparation.

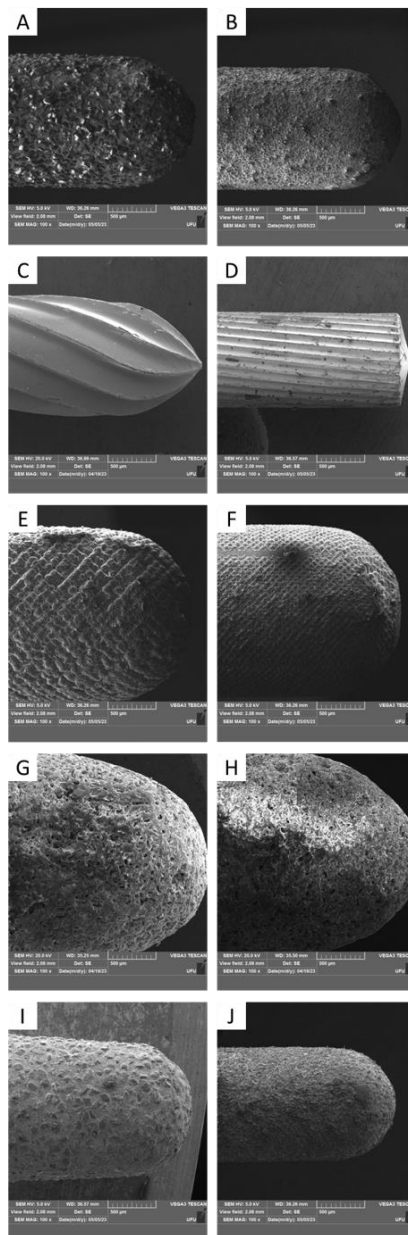
Grupo	Material	Características	Fabricante	Forma de uso
FB	Diamond burs	#3145 F (40µm) #3145 FF (20 µm)	KG Sorensen, Cotia, SP, Brasil	Contra angle (1:5) mounted on an electric micromotor with a speed of 10.000 RPM, coupled to a device to control the cutting force, 30 seconds each instrument under abundant irrigation.
MB	Multilaminated burs	283 Carbide FG (12 lâminas); 9572FF Carbide FG (30 lâminas)	Angelus Prima Dental, Brasil	Contra angle (1:5) mounted on an electric micromotor with a speed of 10,000 RPM, coupled to a device to control the cutting force, 30 seconds each instrument under abundant irrigation.
UT	Ultrasonic diamond tips	CR4 CR4U	CVDentus; São José dos Campos, SP, Brasil	Piezoelectric ultrasound (DentSurg PRO, CVDentus); 20 mL/min water flow rate; 80% of maximum power for CR4 and 50% of maximum power for CR4U, coupled to a cutting force control device, 30 seconds each instrument under abundant irrigation.
MS	Mounted stones of aluminum oxide and silicon carbide	Ponta Montada de Carbetto de Silício (PW1504P) Ponta Montada de Óxido de Alumínio (FG199-016-C)	Dhpro, Paranaguá, PR, Brasil	Contra angle (1:5) mounted on an electric micromotor with a speed of 10,000 RPM, coupled to a device to control the cutting force, 30 seconds each instrument under abundant irrigation.
SB	Sintered diamond burs	# FG199-016-A(40µm) #FG199-016-C (20 µm)	Dhpro, Paranaguá, PR, Brasil	Contra angle (1:5) mounted on an electric micromotor with a speed of 10,000 RPM, coupled to a device to control the cutting force, 30 seconds each instrument under abundant irrigation.

APA	Airborne-particle abrasion	50 µm	BioArt, São Carlos, SP, Brasil	Tip of the device perpendicularly positioned 1 cm from the surface with an approximate pressure of 5 bar, for 30 seconds.
RC	Resin coating	Clearfil SE Bond 2+ Clearfil AP-X Esthetic A2D flowable composite resin	Kuraray Noritake Dental, Tóquio, Japão	<b>Clearfil SE:</b> Active Primer application for 20 seconds, light air blast for 30 seconds from a distance of 10 cm, Bond application with an even film, light air blast. <b>Clearfil AP-X Esthetic:</b> Applied thin layer with brush and light cured for 40 seconds (VALO Grand, Ultradent, South Jordan, UT, USA).

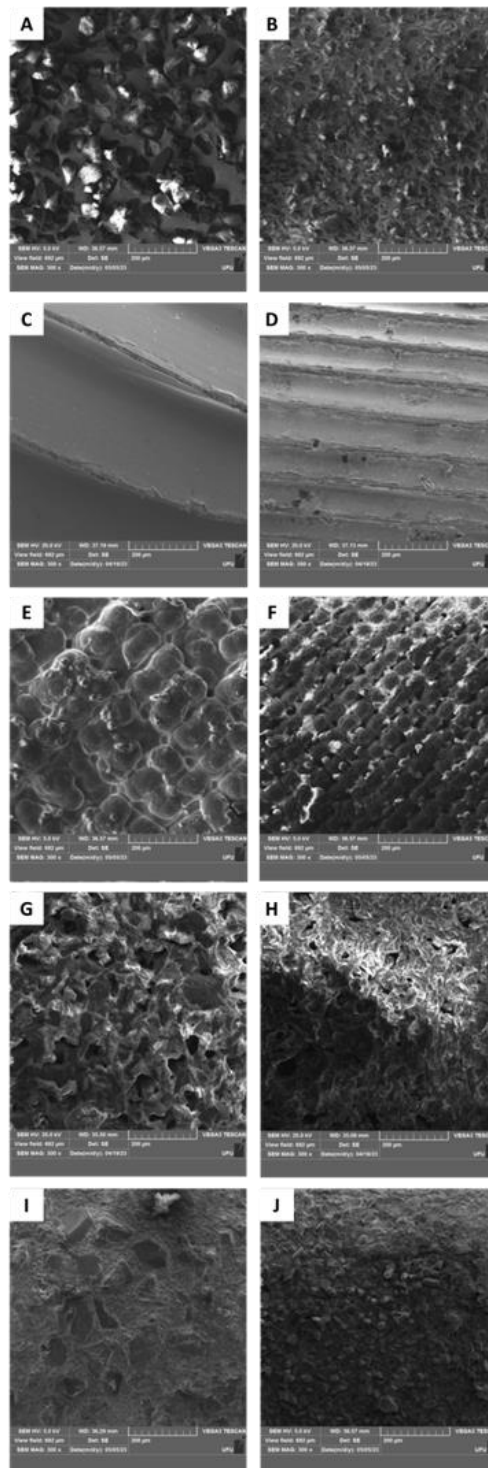
**Table 2:** Mean values of surface roughness (Sa), smear layer thickness ( $\mu\text{m}$ ), bond strength and respective standard deviations ( $\pm$ ) according to the prosthetic preparation finishing protocols for experimental group.

<b>Grups</b>	<b>Surface Roughness (Sa) (n= 4)</b>	<b>Thickness of smear layer (<math>\mu\text{m}</math>) (n=4)</b>	<b>Bond strength (MPa) (n=10)</b>
FB	1.01 $\pm$ 0.08 <sup>a</sup>	7.32 $\pm$ 0.58 <sup>a</sup>	18,8 $\pm$ 5.13 <sup>C</sup>
MB	0.66 $\pm$ 0.12 <sup>b</sup>	3.19 $\pm$ 0.39 <sup>c</sup>	15,27 $\pm$ 3,02 <sup>E</sup>
UT	0.74 $\pm$ 0.07 <sup>b</sup>	2.76 $\pm$ 0.19 <sup>d</sup>	16,92 $\pm$ 3.11 <sup>D</sup>
MS	0.69 $\pm$ 0.10 <sup>b</sup>	2.47 $\pm$ 0.27 <sup>d</sup>	27,84 $\pm$ 3.12 <sup>B</sup>
SB	0.73 $\pm$ 0.30 <sup>b</sup>	2.30 $\pm$ 0.29 <sup>d</sup>	16.01 $\pm$ 3.22 <sup>D</sup>
APA	1.15 $\pm$ 0.08 <sup>a</sup>	7.29 $\pm$ 0.52 <sup>b</sup>	19,32 $\pm$ 3,02 <sup>C</sup>
RC	1.25 $\pm$ 0.53 <sup>a</sup>	-	32,03 $\pm$ 3.94 <sup>A</sup>

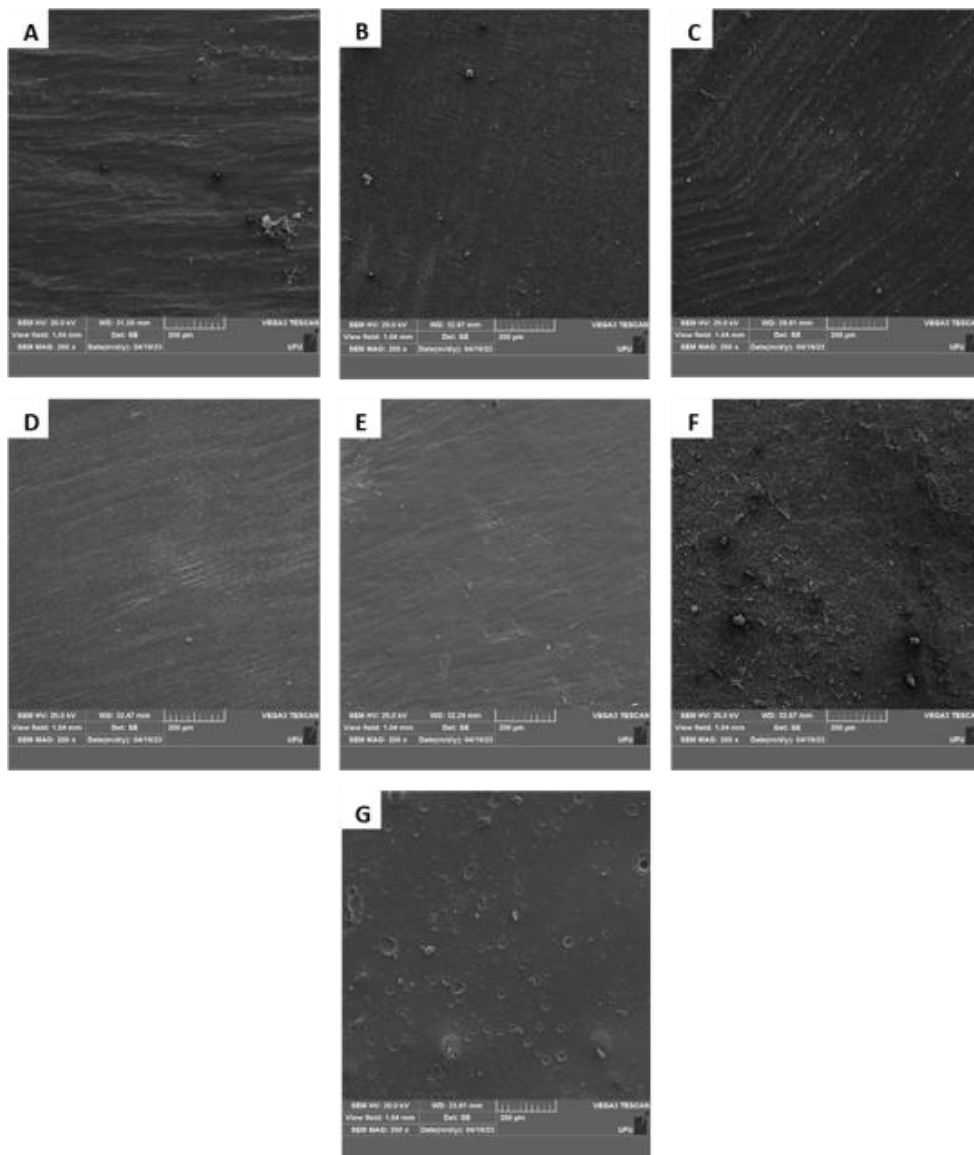
\* Distinct letters indicate statistically significant difference between groups; Tukey HSD test (p<0.05).



**Figure 1.** SEM photomicrographs with 100× magnification of the different instruments used to finish the prosthetic preparations according to the experimental groups: FB (A – diamond tip F and B – diamond tip FF), Group BM (C – multilaminated drill with 12 blades and D – 30-blade multi-blade drill), Group UT (E – ultrasonic insert CR4 and F – ultrasonic insert CR4U), Group MS (G – Mounted aluminum oxide tip and H – Silicon carbide tip); Group SB (I – sintered drill F and J – sintered drill FF).

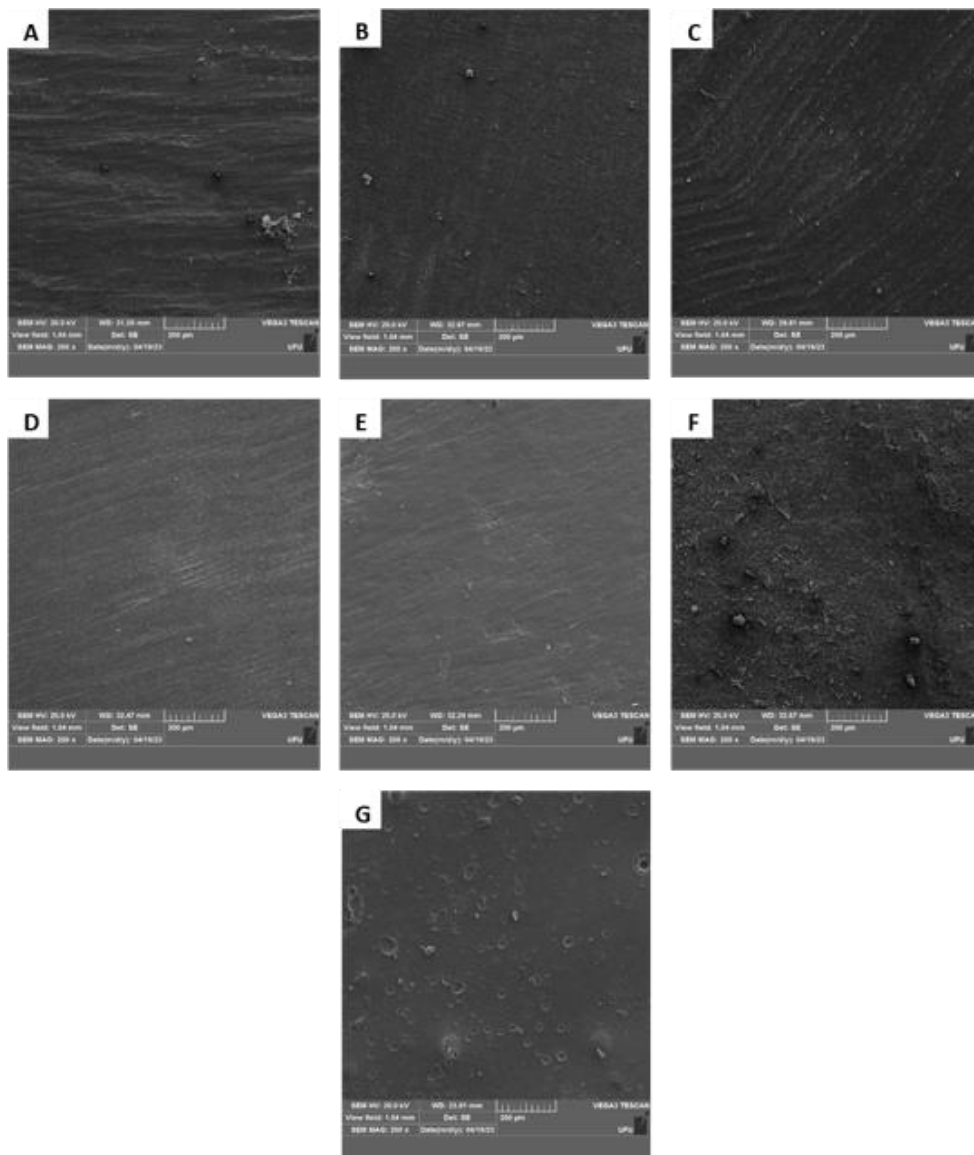


**Figure 2.** SEM photomicrographs with 300× magnification of the different instruments used to finish the prosthetic preparations according to the experimental groups: FB (A – diamond tip F and B – diamond tip FF), Group BM (C – multilaminated drill with 12 blades and D – 30-blade multi-blade drill), Group UT (E – ultrasonic insert CR4 and F – ultrasonic insert CR4U), Group MS (G – Mounted aluminum oxide tip and H – Silicon carbide tip); Group SB (I – sintered drill F and J – sintered drill FF).

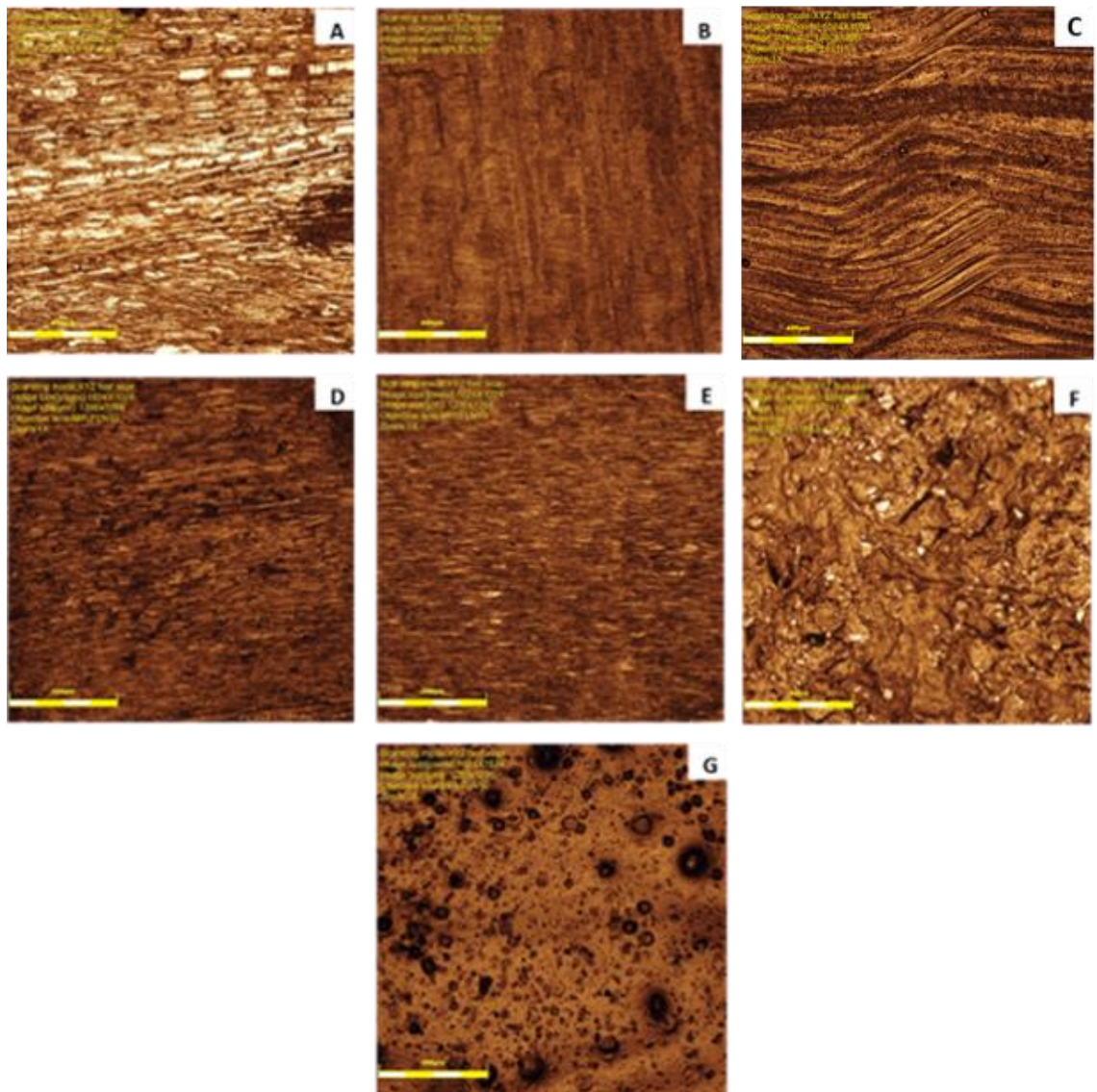


**Figure 3.** SEM photomicrographs with 200× magnification of the dentin surfaces after the different protocols for finishing the prosthetic preparations according to the experimental groups: Group FB (A), Group MB (B), Group UT(C), Group MS(D), SB Groups (E), APA Group (F) and RC Group (G).

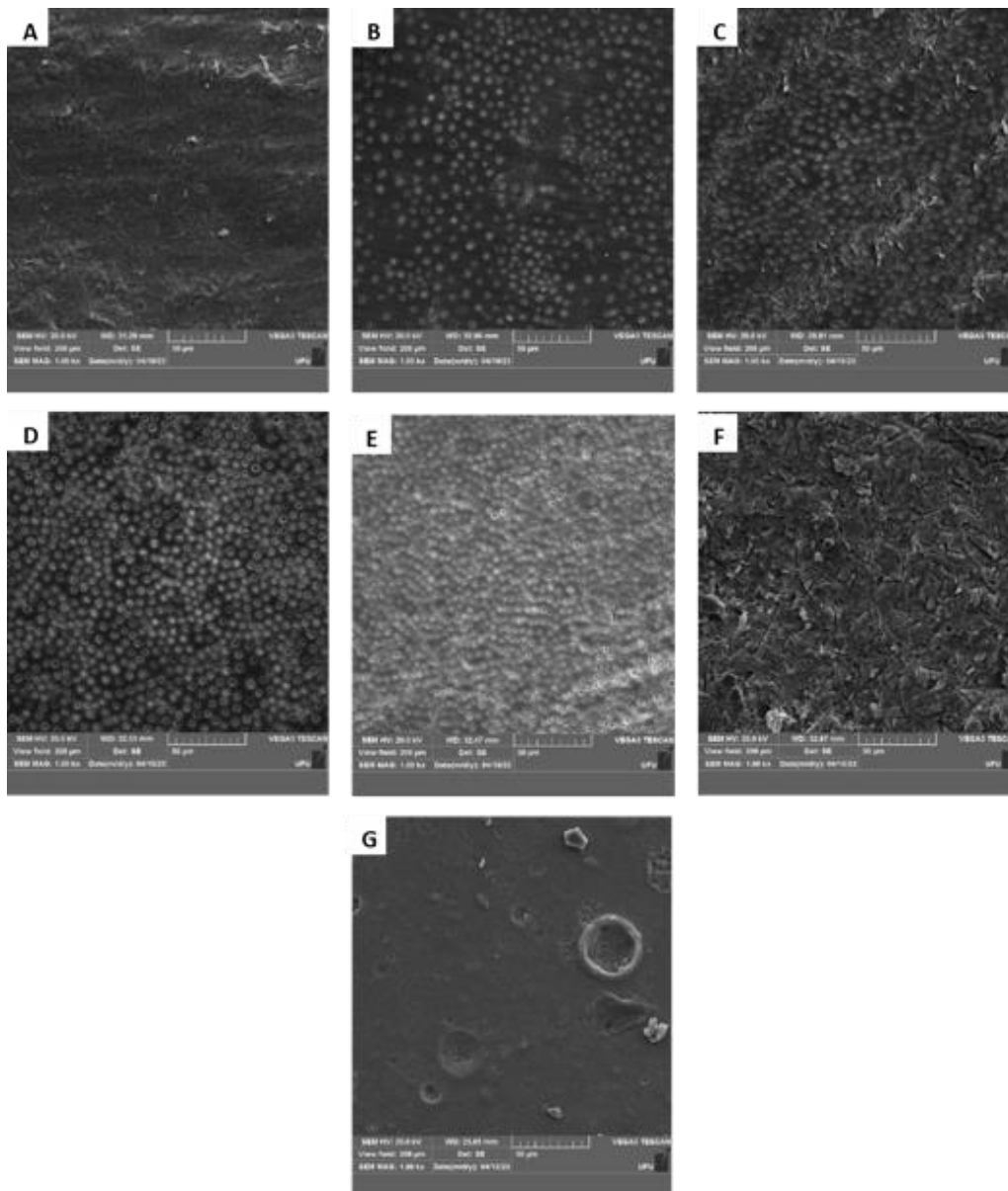




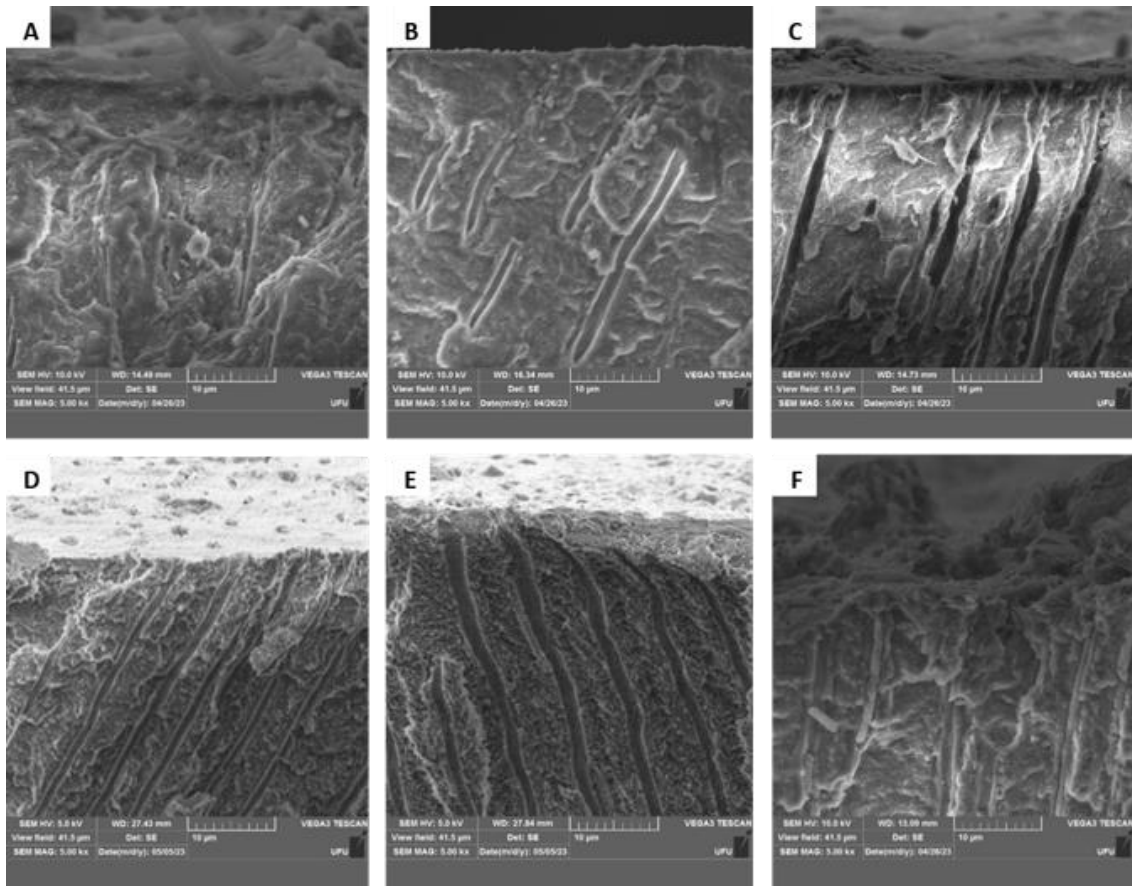
**Figure 3.** SEM photomicrographs with 200× magnification of the dentin surfaces after the different protocols for finishing the prosthetic preparations according to the experimental groups: Group FB (A), Group MB (B), Group UT(C), Group MS(D), SB Groups (E), APA Group (F) and RC Group (G).



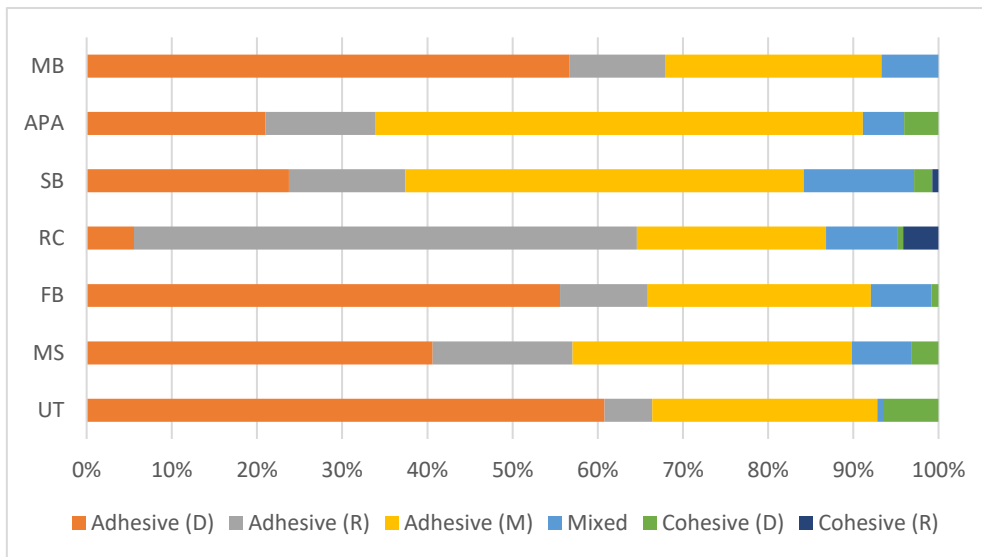
**Figure 4.** Images obtained by laser confocal microscope with 10× magnification of the dentin surfaces after the different finishing protocols of the prosthetic preparations according to the experimental groups: Group FB (A), Group MB (B), Group UT (C), Group MS (D), Group SB (E), Group APA (F) and Group RC (G).



**Figura 5.** Fotomicrografias em MEV com aumento de 1.000× das superfícies de dentina após os diferentes protocolos de acabamento dos preparos protéticos de acordo com os grupos experimentais: PF (A), Grupo BM (B), Grupo IU (C), Grupo PM (D); Grupo PS (E); Grupo AO (F) Gurpo SD (G).



**Figure 6.** SEM photomicrographs with 5,000× magnification of the dentin surfaces after different protocols for finishing the prosthetic preparations according to the experimental groups: FB (A), MB Group (B), UT Group (C), MS Group (D), SB Group (E), APA Group (F).



**Figure 7.** Incidence of failure modes (%) according to prosthetic preparation finishing protocols for each experimental group.

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