UNIVERSIDADE DE UBERABA

ANALIA GABRIELLA BORGES FERRAZ

RESISTÊNCIA DE UNIÃO ENTRE CERÂMICA DE DISSILICATO DE LÍTIO PROCESSADA POR DIFERENTES MÉTODOS E UM CIMENTO RESINOSO SOB DIFERENTES TRATAMENTOS DE SUPERFÍCIE DA CERÂMICA

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Dissertação apresentada como parte dos requisitos para obtenção do título de Mestre em Odontologia, área de concentração: Clínica Odontológica Integrada da Universidade de Uberaba.

Orientador: Prof. Dr. Gilberto Antonio Borges

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Área de concentração: Clínica Odontológica Integrada

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“A menos que modifiquemos a nossa maneira de pensar, não seremos capazes de resolver os problemas causados pela forma como nos acostumamos a ver o mundo.”

(Albert Einstein)

RESUMO

O objetivo deste estudo foi avaliar o efeito de diferentes tratamentos de superfície e do processamento de uma cerâmica à base de dissilicato de Lítio na resistência de união e nas características interfaciais com um cimento resinoso fotoativado. Vinte amostras foram fabricadas de forma prensadas (IPS e.max Press) (10 mm de diâmetro x 1 mm de espessura) e vinte pela técnica CAD/CAM (IPS e.max CAD) (15 mm x 10 mm e 1 mm de espessura). Para cada tipo de processamento, foi dividido em quatro grupos (n = 5) conforme o tipo de tratamento de superfície, totalizando oito grupos experimentais: NT - nenhum tratamento superficial; HFS - ácido fluorídrico a 10% (HF) e aplicação de silano; HFU - HF 10% e adesivo universal e EP - aplicação de primer cerâmico. Depois de preparadas, as amostras foram armazenadas em água destilada por 24h a 37°C e submetidas à ensaio de resistência de união. Os valores de resistência de união, em MPa, foram analisados pelo teste t de Student, Kruskal-Wallis e teste post-hoc de Student-Newman-Keuls (p>0.05) e imagens em microscopia eletrônica de varredura (MEV) foram realizadas para análise qualitativa do padrão de fratura após o ensaio de resistência de união e espécimes exemplares foram realizadas para análise da interface cimento/cerâmica. Os tratamentos HFU (16.8 +/- 3,51 MPa) e EP (12.9 +/- 3.05 MPa) mostraram os melhores valores de resistência de união para cerâmicas prensadas e estatisticamente superiores do que HFU e EP das cerâmicas CAD/CAM. Dentre as cerâmicas CAD/CAM, os melhores valores foram apresentados pelo tratamento HFS (8.17 +/- 4.81 MPa), este estatisticamente semelhante à HFS prensada (5.92 +/- 3.51 MPa). Somente os grupos NT mostraram falhas/gaps na interface de cerâmica/cimento e padrão de fratura adesiva em MEV. Os demais grupos apresentaram um padrão de fratura mista e sem falhas/gaps consideráveis na interface cerâmica/cimento. Os tratamentos HFU e EP mostraram serem os melhores para cerâmicas prensadas e HFS e HFU para cerâmicas CAD/CAM. Imagens MEV não mostraram diferenças significativas entre os tipos de tratamento de superfície, exceto em NTs.

Palavras-chave: Cerâmica. Cimentos de Resina. Resistência ao cisalhamento. CAD-CAM.

ABSTRACT

The aim of this study was to evaluate the effect of different surface treatments and the processing of a Lithium disilicate based ceramic on bond strength and interfacial characteristics with a photoactivated resin cement. Twenty samples were fabricated in press (IPS e.max Press) (10 mm in diameter x 1 mm in thickness) and twenty in the CAD / CAM technique (IPS e.max CAD) (15 mm x 10 mm and 1 mm thickness). For each type of processing, it was divided into four groups (n = 5) according to the type of surface treatment, totaling eight experimental groups: NT - no surface treatment; HFS - 10% hydrofluoric acid (HF) and silane application; HFU - HF 10% and universal adhesive and EP - primer application. After being prepared, the samples were stored in distilled water for 24h at 37 °C and subjected to the bond strength test. The bond strength values, in MPa, were analyzed by Student's t test, Kruskal-Wallis and Student-Newman-Keuls post-hoc test ($p > 0.05$) and scanning electron microscopy (SEM) images were performed for qualitative analysis of the fracture pattern after the bond strength test and specimens were manufactured for cement / ceramic interface analysis. The HFU (16.8 +/- 3.51 MPa) and EP (12.9 +/- 3.05 MPa) treatments showed the best values of bond strength for pressed and statistically better ceramics than HFU and EP of CAD/CAM ceramics. Among the CAD/CAM ceramics, the best values were presented by the HFS treatment (8.17 +/- 4.81 MPa), which is statistically similar to pressed HFS (5.92 +/- 3.51 MPa). Only NT groups showed faults/gaps at the ceramic/cement interface and adhesive fracture pattern in SEM. The other groups presented a mixed fracture pattern with no significant faults/gaps at the ceramic/cement interface. The HFU and EP treatments showed to be the best for pressed ceramics and HFS and HFU for CAD/CAM ceramics. SEM images did not show significant differences between surface treatment types except NTs.

Key-words: Ceramics. Resin cements. Shear Strength. CAD-CAM.

SUMÁRIO

1 INTRODUÇÃO

A utilização de cerâmicas odontológicas é realizada por muitos cirurgiões dentista na prática clínica. Esses materiais têm como função restabelecer de forma total ou parcial a perda de estrutura dental. As cerâmicas foram por muito tempo, exclusivamente, utilizadas sobre uma infraestrutura metálica, mas com o advento das cerâmicas reforçadas e da união à estrutura dental, atualmente as cerâmicas são aplicadas sem o metal (ZHANG & KELLY, 2017), com evidências científicas mostrando a sua longevidade (DELLA BONA et al., 2008; GEHRT et al., 2013; MOBILIO et al., 2018).

Dentre os vários tipos de cerâmicas destaca-se a cerâmica à base de dissilicato de Lítio, a qual é constituída de quartzo, dióxido de lítio, óxido fosfórico, alumina, óxido de potássio e outros componentes. Setenta por cento do seu conteúdo consistem em cristais de dissilicato de lítio, diante disto, estas cerâmicas proporcionam resistência mecânica e estética favorável. Podem ser confeccionadas de forma prensada ou pela técnica Computer-aided Design/Computer-aided Manufacturing (CAD/CAM) e podem ser indicadas para vários casos como laminados, coroas parciais ou totais, próteses fixas, coroas unitárias ou múltiplas e para regiões anteriores ou posteriores (RITTER 2010; GARBOZA et al., 2016; MOUNAJJED et al., 2016; WILLARD & CHU, 2018).

A confecção das cerâmicas prensadas requer variados processos laboratoriais, além de clínicos, para a confecção de algum tipo de estrutura do material, como confecção de modelo de gesso, troquelização, enceramento, fundição, acabamento e polimento (BORGES et al., 2014). Por outro lado, o processo de confecção pelo método CAD/CAM consiste em menos etapas, como digitalização do modelo de gesso (quando não utilizado o Scanner oral), confecção da restauração com auxilio de software específico, usinagem do bloco cerâmico, acabamento e polimento (GJELVOLD et al., 2016). Em particular ao método CAD/CAM, a fase de acabamento é caracterizada pela completa cristalização da restauração cerâmica. Nesta fase, a cerâmica é submetida a temperaturas de 850 ° C, em forno próprio, e passa de 40% de cristalização para 70% (ALKADI & RUSE, 2016).

Cerâmicas de dissilicato de lítio apresentam excelentes propriedades mecânicas, tanto processadas de forma prensada ou como pela CAD/CAM. Existe uma similaridade de resistência à fratura desse tipo de cerâmica, fabricadas por

ambos os métodos e apresentam uma correlação de semelhança na adaptação marginal (OZ & BOLAY, 2018; AZAR et al., 2018). Esses materiais também demonstram resistência à fratura superior quando comparados a outros tipos de materiais, como resinas compostas, cerâmicas híbridas, cerâmicas feldspáticas e cerâmicas reforçadas por leucita (WAFAIE et al., 2018; ANDRADE et al., 2018; SAGSOZ et al., 2018).

Este tipo de material é adesivamente unido à estrutura dental. Desta forma, são utilizados procedimentos e materiais responsáveis por tratar a cerâmica à base de dissilicato de lítio e assim resultar nesta união. Convencionalmente, essas cerâmicas são tratadas com ácido hidrofluorídrico (HF), que condiciona a parte vítrea da cerâmica, criando irregularidades na superfície. E em seguida a cerâmica é revestida com um agente de união, silano, capaz de interagir com parte inorgânica da cerâmica e parte orgânica do agente de cimentação (LISE et al., 2015; GARBOZA et al., 2016; KESHVAD & HAKIMANEH, 2018).

Recentemente foi introduzido no mercado materiais que prometem simplificar essas etapas clínicas em um único passo. Podemos citar como exemplos desses materiais, os adesivos universais e primers cerâmicos, que condicionam e silanizam a estrutura simultaneamente, os fabricantes alegam a sua eficiência pela diminuição de erros provenientes de múltiplos passos (YAO et al., 2017, MORO et al. 2018; TRIBST et al., 2018).

Diante o exposto, vários estudos já foram realizados, porém sem a devida comprovação categórica da superioridade de eficácia desses materiais simplificados no processo de tratamento de superfície das cerâmicas. E observa-se a carência de estudos verificando a ação dos tratamentos de superfície em cerâmicas CAD/CAM. Desta forma, o objetivo deste trabalho foi avaliar o efeito de diferentes tratamentos de superfície e do processamento da cerâmica à base de dissilicato de lítio na resistência de união e nas características interfaciais cerâmica/cimento. O estudo foi realizado sob as seguintes hipóteses: 1) os dados estatísticos não mostraram diferença nos valores de resistência de união entre os tratamentos de superfície da cerâmica; 2) dentre os valores de resistência de união entre os métodos de confecção da cerâmica não mostraram ser estatisticamente diferentes.

2 Capítulo 1: Bond Strength between Lithium Disilicate Ceramic Processed by different Methods and Resin Cement under Different Surface Treatments of the Ceramics.

Operative Dentistry (ANEXO 1).

Manuscript Type: Original Article

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Bond Strength between Lithium Disilicate Ceramic Processed by different Methods and Resin Cement under Different Surface Treatments of the Ceramic

Clinical Relevance

The use of hydrofluoric acid and silane (HF) shows to be the most effective method for treating ceramic surfaces based on CAD/CAM lithium disilicate. As for pressed ceramics, the use of universal adhesive and HF showed to be more efficient.

ABSTRACT

The aim of this study was to evaluate the effect of different surface treatments and the processing of a Lithium disilicate based ceramic on bond strength and interfacial characteristics with a photoactivated resin cement. Twenty samples were fabricated in press (IPS e.max Press) (10 mm in diameter x 1 mm in thickness) and twenty in the CAD / CAM technique (IPS e.max CAD) (15 mm x 10 mm and 1 mm thickness). For each type of processing, it was divided into four groups (n = 5) according to the type of surface treatment, totaling eight experimental groups: NT - no surface treatment; HFS - 10% hydrofluoric acid (HF) and silane application; HFU - HF 10% and universal adhesive and EP - primer application. After being prepared, the samples were stored in distilled water for 24h at 37 °C and subjected to the bond strength test. The bond strength values, in MPa, were analyzed by Student's t test, Kruskal-Wallis and Student-Newman-Keuls post-hoc test ($p > 0.05$) and scanning electron microscopy (SEM) images were performed for qualitative analysis of the fracture pattern after the bond strength test and specimens were manufactured for cement / ceramic interface analysis. The HFU (16.8 +/- 3.51 MPa) and EP (12.9 +/- 3.05 MPa) treatments showed the best values of bond strength for pressed and statistically better ceramics than HFU and EP of CAD/CAM ceramics. Among the CAD/CAM ceramics, the best values were presented by the HFS treatment (8.17 +/- 4.81 MPa), which is statistically similar to pressed HFS (5.92 +/- 3.51 MPa). Only NT groups showed faults/gaps at the ceramic/cement interface and adhesive fracture pattern in SEM. The other groups presented a mixed fracture pattern with no significant faults/gaps at the ceramic/cement interface. The HFU and EP treatments showed to be the best for pressed ceramics and HFS and HFU for CAD/CAM ceramics. SEM images did not show significant differences between surface treatment types except NTs.

Key-words: Ceramics. Resin cement. Shear Strength. CAD-CAM.

INTRODUCTION

The use of dental ceramics is the reality of many dentist surgeons in clinical practice. These materials have the function of restoring totally or partially the loss of dental structure. Ceramics were for a long time exclusively used on a metallic infrastructure, but with the advent of reinforced ceramics and adhesion to dental structure, they are currently applied without the metal, [1] and there is sufficient scientific evidence showing their longevity. [2, 3, 4]

Among the various types of ceramics stand out the ceramics based on lithium disilicate, which consist of quartz, lithium dioxide, phosphoric oxide, alumina, potassium oxide and other components. Seventy percent of its content consists of lithium disilicate crystals, which provide mechanical resistance and favorable esthetic. Restorations of this type of material can be done in a pressed manner or by the technique of Computer Aided Design/Computer Assisted Manufacturing (CAD/CAM) and can be indicated in several cases as laminates, partial or total crowns, fixed prosthesis, single or multiple crowns. [5 - 8]

The preparation of pressed ceramics requires a variety of laboratory and clinical processes for the manufacturing of indirect restorations such as confection plaster model, troquelization, waxing, casting, finishing and polishing. [9] On the other hand, the CAD/CAM method consists of fewer steps, such as digitizing the plaster model (when not using the oral scanner), fabrication the restoration using specific software, machining the ceramic block, finishing and polishing. [10] In particular to the CAD/CAM method the finishing phase is characterized by the complete crystallization of the ceramic restoration. At this stage the ceramic is subjected to temperatures of 850 ° C, in its own furnace, and from 40% crystallization to 70%. [11]

Lithium disilicate ceramics exhibit excellent mechanical properties, both pressed and CAD / CAM processes. There is a similarity of fracture resistance of this type of ceramic, manufactured by both methods and present a correlation of similarity in the marginal adaptation. [12, 13] These materials also show superior fracture toughness when compared to other types of materials, such as composite resins, hybrid ceramics, feldspathic ceramics and leucite-reinforced ceramics. [14, 15, 16]

This type of material is adhesively bonded to the tooth structure. In this way, procedures and materials are used to treat the ceramic based on lithium disilicate and thus to result in this bond. Conventionally, these ceramics are treated with hydrofluoric acid (HF), which conditions the glassy part of the ceramic, creating surface irregularities. The ceramic is then coated with a bonding agent, silane, capable of interacting with the inorganic part of the ceramic and the organic part of the cementing agent. [6, 17, 18]

Recently materials have been introduced that promise to simplify these clinical steps in one step. We can cite as examples of these materials, universal adhesives and ceramic primers, which condition and silanize the structure simultaneously, manufacturers claim their efficiency by reducing errors from multiple steps. [19, 20, 21]

In view of the above, several studies have already been carried out, but without a categorical proof of the superiority of efficacy of these simplified materials in the surface treatment process of the ceramics. It is observed the lack of studies that verify the action of the types of surface treatments in ceramics CAD/CAM and pressed in a single study. In this way, the objective of this work was to evaluate the effect of different surface treatments and the processing of the ceramic based on lithium disilicate in the bond strength and in the ceramic/cement interfacial characteristics. The study was carried out under the following hypotheses: 1) the different types of surface treatment do not influence the values of bond strength between ceramic and resin cement; and 2) ceramic manufacturing methods do not influence the adhesive strength values between ceramic and resin cement.

MATERIALS AND METHODS

Twenty ceramic samples with 10 mm diameter and 1 mm thickness were fabricated for e.max Press (Ivoclar Vivadent, Schaan, Liechtenstein), and twenty samples with 10 mm by 15 mm for 1 mm thickness were fabricated for e.max CAD (Ivoclar Vivadent, Schaan, Liechtenstein). Table 1 describes the materials used in the study and Table 2, gives the manufacturer's instructions.

Pressed ceramic discs

Acrylic resin cylinders (Duralay, Reliance Dental MFG Company, Illinois, USA) with 10 mm in diameter were made in a putty consistency of Poly Dimetil Siloxane

(Zetaplus, Zermack, Italy) and following cut in discs in the thickness of 1.0 mm using a 0.5 mm-diamond saw (Buehler, Lake Bluff, IL, USA) coupled to IsoMet precision machine (Isomet 1000-Buehler, Lake Bluff, IL, USA). After, they were sprued in silicone cylinders, attached to a flask base, invested with phosphate-based material (IPS PressVest Speed, Ivoclar Vivadent, Schaan, Liechtenstein) and eliminated in an automatic furnace (EDG 3000, São Carlos, SP, Brazil) at temperature of 850ºC for 60 min using the lost wax technique. The IPS e.max Press ceramic ingots were pressed into the investment molds in an automatic press furnace (EP 3000, Ivoclar Vivadent, Schaan, Liechtenstein). After removing the disc from the investment material by sandblasting (4 bar to remove the coarse part and 2 bar for removal of coatings near the samples), all samples were ultrasonically cleaned in deionized water (Ultrasonic Cleaner 1440 D, Odontobrás, Ribeirão Preto, SP, Brazil) for 10 min, dried with compressed air. The final disc thicknesses (1.0 mm) were confirmed with a digital caliper (Mitutoyo Corporation, Tokyo, Japan), with accuracy of 0.01 mm.

CAD/CAM ceramic blocks

The CAD/CAM samples were made by cutting the ceramic block with a cutting apparatus (Isomet 1000-Buehler, Lake Bluff, IL, USA) and 0.5 mm diamond disk (Buehler, Lake Bluff, IL, USA). The final thickness was checked the same way as described to the pressed discs. Following, the blocks were crystalized in an automatic press furnace (EP 3000, Ivoclar Vivadent, Schaan, Liechtenstein). The crystallization process takes between 20 to 31 minutes, and the blocks do not shrinkage significantly. The process happens between 840 to 850 º C and it produces microstructure modification, which is a controlled growing of the disilicate crystals.

Surface treatment for cementation

After fabrication, the samples were divided into eight experimental groups, according to the type of surface treatment and processing, as shown in figure 1, where:

- NT = no treatment;
- HFS = hydrofluoric acid and silane
- HFU = hydrofluoric acid and universal adhesive
- EP = monobond Etch & Prime

Cementation procedure

Polyvinyl siloxane molds (Virtual, Ivoclar-Vivadent, Schaan, Liechtenstein), 0.5 mm thick, were fabricated using five cylinder-shaped orifices (0.8 mm in diameter) and were placed on the ceramic disc surface to determine the adhesion area. Before positioning the mold, each surface treatment was applied to the surface of each experimental group.

Surface treatment and cementing procedure were performed by the same operator under controlled temperature (23 ± 2ºC). The resin cement (Variolink Esthetic LC, Ivoclar Vivadent, Schaan, Liechtenstein) was prepared according to the manufacturer's instructions and inserted into the orifice of the mold, with a spoon excavator (Duflex, Juiz de Fora, MG, Brazil). Excess cement was removed using a resin spatula #01 (Duflex, Juiz de Fora, MG, Brazil). The resin cement was photo activated for 40 s, using a continuous mode with a LED Radii Cal (SDI, Victoria, Australia) and an irradiance of 500 mW/cm^2, as verified with radiometer (Kerr, Joinvile, SC, Brazil). After 10 min the silicone matrix was removed and cement cylinders were carefully evaluated with a optical microscope to observe the bonding area. Following, they were stored for 24 h at 37ºC, 100% relative humidity until the bond strength test.

Micro shear bond strength test

Micro shear bond strength (μSBS) testing was performed in a testing machine (EMIC DL 3000 ¯ EMIC - Equipamentos e Sistemas de Ensaios Ltda. São José dos Pinhais, Brazil). A stainless steel chisel was attached to the load cell and the test was carried out at 0.5 mm/min crosshead speed until failure. The average of each resin cement cylinder on the ceramic specimens was calculated to obtain the mean value of the bond strength of each sample. The testing machine software was set to give the results in MPa.

Statistical analysis

The mean of the total samples of each group was submitted to the t student test to normal and homogenous distribution variable among the groups. Following, Kruskal-Wallis test and Student-Newman-Keuls post hoc test were carried out. Differences were considered significant at $p < 0.05$.

Failure mode analysis and cement/ceramic interface

After the rupture of the resin cement cylinders were observed in scanning electron microscopy (SEM) (JSM-5600LV, Jeol Ltd., Tokyo, Japan) at 15Kv. The specimens were mounted on coded brass stubs coated with sputter coating (SCD 050, BAL-TEC, Liechtenstein) for 180 s at 40 mA. And the images were classified as cohesive (COH) (failure within the cement layer), adhesive (ADH) (failure between ceramic and cement), or mixed (MIX) (involving cement and ceramic substrates).

Additional specimens of ceramics-cement-ceramics were obtained for each group; two ceramic samples conditioned with different surface treatment were bonded together using resin cement. The specimens were embedded cross sectionally in epoxy resin in order for the ceramic-cement interfaces to be viewed. After 24 h, the specimens were wet-polished with 600-, 1200- and 2000-grit SiC paper followed by polishing with 3 µm, 1 µm and 0.5 µm diamond compounds. The cross-section profiles were examined by SEM, focusing on the integrity, homogeneity and continuity along the bonding interface.

RESULTS

Statistical analysis

The data are presented in Table 3 and the highest bond strength values in MPa were presented by HFU (16.8 ± 6.26) and PE (12.9 ± 3.05) pressed ceramics groups, followed by HFS groups (5.92 ± 3.51) and NT (2.31 ± 1.66). Among the surface treatments of CAD/CAM ceramics, the highest statistical values of union strength were for the HFS group (8.17 ± 4.81), but were not statistically different in comparison to HFU (7.83 ± 5, 30) and EP (4.34 ± 2.78). Statistically, the lowest bond strength values among the CAD/CAM ceramics were demonstrated by NT 1.24 ± 1.23. Overall, all types of surface treatment of CAD/CAM ceramics were statistically lower than pressed ceramics, with the exception of HFS, which did not present statistically different values among ceramic manufacturing methods.

Failure mode analysis and cement/ceramic interface

The SEM images showed that, regarding the failure mode, only NT groups, both CAD and Press, presented adhesive failures and the other groups presented mixed

failures. The interface between the resin cement and the glass ceramic was continuous without voids or failures for all groups, except NT that showed discontinuity for both CAD and Press, being the CAD group with a larger gap (Fig. 2 - 9).

DISCUSSION

The results showed that there was a statistical difference of bond strength between the types of surface treatment and the types of ceramic manufacture. Therefore, the hypotheses of the study were rejected. In the consulted literature, no study was found comparing the bond strength between the pressed and CAD/CAM systems of the lithium disilicate ceramic, using the same surface treatments in a single research. Both universal adhesives and ceramic primers are materials where the manufacturer brings the proposal to simplify clinical steps, when in only one bottle there are the etching and silane of the ceramic structure. Nevertheless, studies still show that the individual use of the silane coating agent has an important role in bond strength between ceramics, pressed and CAD/CAM, and resin cement. [22-26]

Universal adhesives, as used in the present study, Single Bond Universal (3M ESPE), contains silane and an acidic monomer called 10-methacryloxydecyl phosphate (MDP) in its composition. The silane is an important substance in the bond between lithium disilicate ceramics and resin cement. It is a bifunctional molecule that binds both the organic part of the ceramic and the inorganic one of the resin cement, but it is sensitive to the pH value of the solution. Usually the material presents a pH value between 4 and 5, on the other hand, the universal adhesives the pH is about 2.7. [27] The reaction between silane and MDP promotes the adhesion mechanism, improving surface wettability, [28] but the pH value of an MDP molecule is between 2 and 2.7, which contributes to the low pH value of the universal adhesive, compromising the ideal chemical interaction of the silane with the ceramic. [27] This may have happened in the experiments of this study that led to the lower of results of the samples, treated with universal adhesive in the group of CAD/CAM ceramics, in comparison to the group treated with HF and silane. Furthermore, some studies also show worst results when using universal adhesive, such as Kalavacharla et al. in 2015 [29] and Murillo-Gómez et al. in 2017 [30] which demonstrated bond strength data when used silane plus a statistically better universal adhesive compared to the same

application of the adhesive alone. Advising that, the realization of silane application is necessary for the surface treatment of CAD/CAM ceramics based on lithium disilicate.

The Etch & Prime monobond contains ammonium polyfluoride, which is an acid salt that corrodes glasses and silicates, reaching a porous aspect and resulting in micro-mechanical retentions, but has a softer acidity compared to hydrofluoric acid, leading to a pattern of weak conditioning. [31] Both El-Damanhoury in 2017 [32] and Lyan et al. in 2018 [31] showed that, in comparison to the HF conditioning, the use of EP results in inferior bond strength values between ceramic and resin cement. In the same way, in the present study, better results can also be observed with the use of HFS and HFU in CAD/CAM ceramics than the use of EP.

The results of Press group treated with HFS were different than expected, considering the pertinent literature. [21-25] This factor is related to the concentration of HF used in the study. HF is responsible for removing part of the silica matrix of a glass ceramic, promoting a porous surface, allowing the micromechanical retention, besides providing greater area available for adhesion. [33] This material can be found in concentrations between 1 and 10%. It has already been demonstrated that, HF 10%, resulted in increase of bond strength between ceramic and resin cement because it results in more micro retentions than the other concentrations, [34] however, this high concentration can lead to an extensive removal of the vitreous matrix and the removal of the crystals of lithium disilicate, generating failures as gaps in the bond, acting as initiators of cracks. [33]

With the exception of the HFS CAD group, the remaining within CAD showed statistically lower bond strength values than all groups of pressed ceramics. This can be explained due to the grounding procedure used in CAD/CAM. Grounding by machining of a material is characterized by the process of removal of fragments by a tool (diamond tips and stainless steel burs). In the present study, diamond tips were not used as in clinical reality, but cutting with a diamond blade may have resulted in surface damage associated with the removal of the material, affecting the bond strength between this type procedure and the resin cement. These would induce cracks on the surface of the ceramic, that would propagate and resulting in catastrophic failure. [35] Another relevant explanation for the lower performances of CAD / CAM ceramics was reported in 2016. [11] In this study it was reported that CAD/CAM ceramics

have lower fracture toughness values (K_{IC}) than pressed ceramics and SEM images of ceramic surface characterization, demonstrated that CAD/CAM ceramics present a surface smooth, indicating a crack propagation through the glass matrix, while pressed ceramics present a more rough and irregular surface, with several visible crystals embedded in the glass matrix. The difference between the K_{IC} values between IPS e.max Press and CAD/CAM seems to be related to the higher amount of glass matrix, reduced crystalline phase and the smaller crystal size of the IPS e.max CAD, leading to larger failures of CAD/CAM ceramics.

Within the limitations of this study, in vitro, pressed ceramics resulted in values of bond strength statistically superior to CAD/CAM, when using universal adhesive and ceramic primer. High HF concentration did not show efficacy in pressed ceramics as shown in CAD/CAM ceramics. This leads us to reflect on the choice of the best surface treatment of CAD/CAM ceramics, if there would be a need for a specific treatment for this type of manufacturing method, even the ceramics are of the same composition. More studies are necessary to make clearer if there is a difference between the methods and treatments regarding not only bond strength, but also longevity of the restorations clinically.

CONCLUSION

1) The use of hydrofluoric acid and universal adhesive proved to be the best surface treatment for pressed lithium disilicate ceramics. In contrast, the surface treatment of CAD/CAM ceramics was shown to be more effective when using hydrofluoric acid and silane.
2) SEM images showed significant discontinuity and presence of faults/gaps in ceramics no surface treatment, but the same findings were not found among the other treatments.
3) The fracture pattern between ceramic and resin cement showed both areas of resin cement failure and bonding agents, except in no treatment samples that showed almost total absence of resinous cement residue.

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TABLES AND FIGURES

TABLE 1. DESCRIPTION OF THE MATERIALS USED.

Material	Composition	Manufacturer
IPS e.max Press	SiO2, Li2O, K2O, MgO, ZnO, Al2O3, P2O5 e other oxides.	Ivoclar Vivadent, Schaan, Liechtenstein
IPS e.max CAD	SiO2, Li2O, K2O, MgO, Al2O3, P2O5 e other oxides.	Ivoclar Vivadent, Schaan, Liechtenstein
Variolink Esthetic	Organic matrix: urethane dimethacrylate and other methacrylate monomers. Inorganic matrix: ytterbium trifluoride and mixed spheroid oxide. Initiators, stabilizers and pigments.	Ivoclar Vivadent, Schaan, Liechtenstein
Condac porcelana	10% Fluoridric Acid, water, thickener, surfactant and colorant.	FGM, Joinvile-SC, Brasil
Monobond N	Alcoholic solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate.	Ivoclar Vivadent, Schaan, Liechtenstein
Single Bond Universal	2-hydroxyethyl methacrylate, Bisphenol A diglycidyl ether dimethacrylate (BisGMA), Decamethylene dimethacrylate, Ethanol, Silane treated silica, Water, 1,10-Decanediol phosphate methacrylate, Acrylic copolymer and itaconic acid, Caforquinone, N, N-Dimethylbenzocaine.	3M ESPE, St Paul, MN, USA
Monobond Etch&Prime	Aqueous alcoholic solution of ammonium polyfluoride, silane methacrylate and dye.	Ivoclar Vivadent, Schaan, Liechtenstein

Table 2. Application steps of surface treatment procedures.

Material	Ceramic surface
Condac porcelana	Apply for 20s, wash and dry.
Monobond N	Apply a thin layer with a microbrush and leave to act with 60 sec. Remove any excess with a strong jet of air.
Single Bond Universal	Apply a layer to the pretreated surface with hydrofluoric acid, remove excess and apply a strong jet of air.
Monobond Etch&Prime	Apply with a microbrush for 20 s, leave to act for 40 s. Rinse the surface and dry.

Table 3. Means and standard deviations of the micro shear bond strength (MPa) values of different experimental groups.

Groups	PRESS Mean (sd)	CAD Mean (sd)
NT	2.31 (1.66) ^{Ac}	1.24 (1.23) ^{Bc}
HFS	5.92 (3.51) ^{Ab}	8.17 (4.81) ^{Aa}
HFU	16.8 (6.26) ^{Aa}	7.83 (5.30) ^{Bab}
EP	12.9 (3.05) ^{Aa}	4.34 (2.78) ^{Bb}

Different superscript uppercase letters in the same row indicate significant difference ($P>0.05$). Different superscript lowercase letters in the same column indicate significant difference ($P>0.05$)

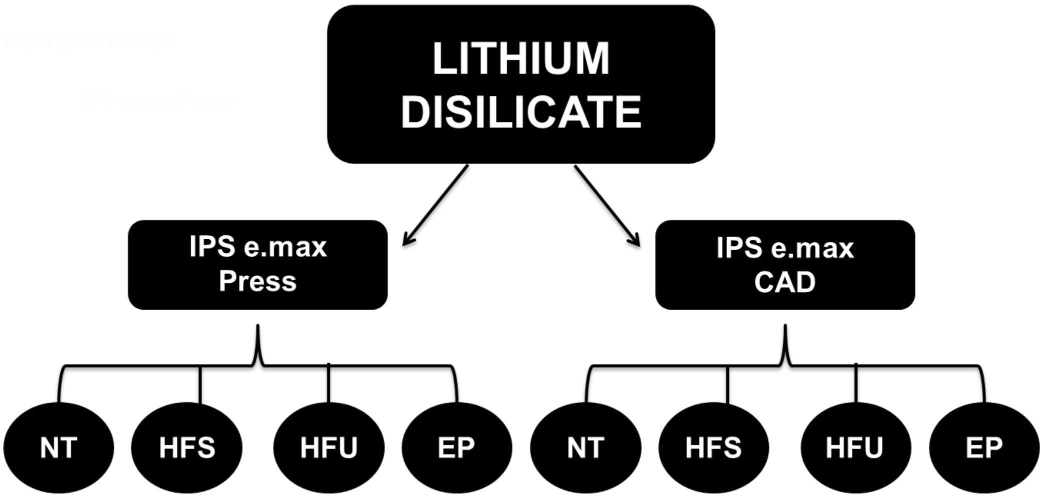

Figure 1 Organization chart of experimental groups.

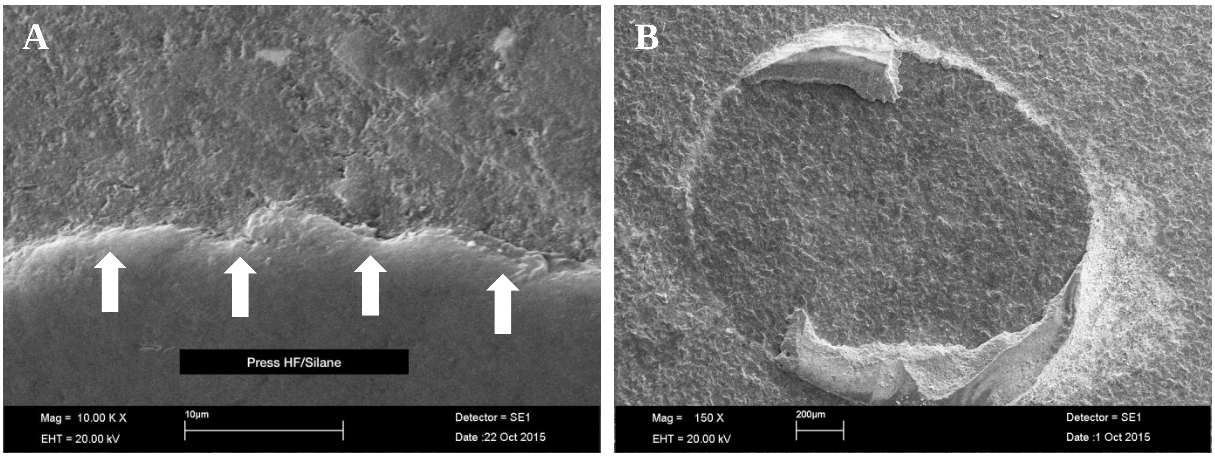

Fig 2 - SEM images. **A** HF/Silane interface (indicated by the white arrows) without discontinuity and failure/gaps (Original magnification X10000) and **B** mixed failure for Press fabrication (Original magnification X150)

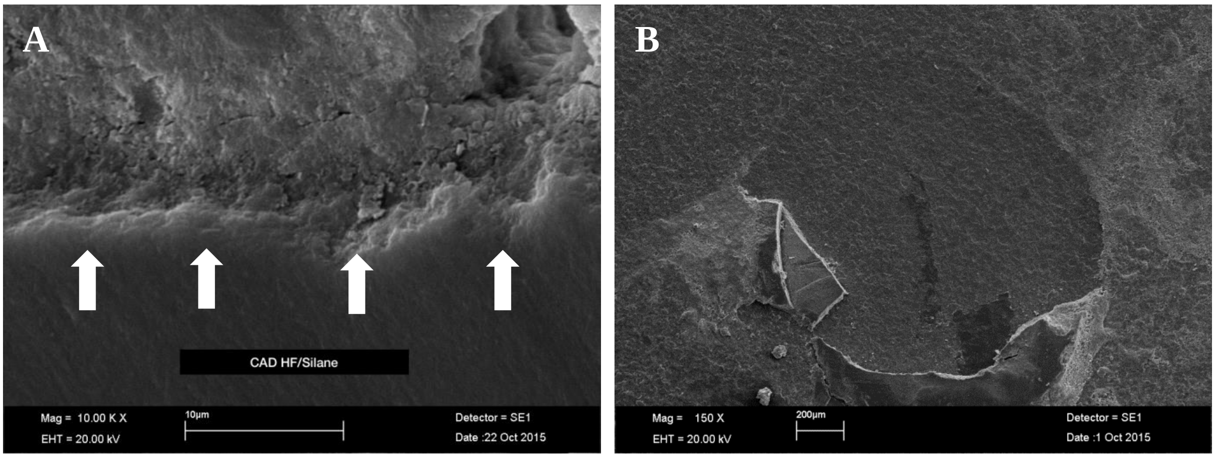

Fig 3 - SEM images. **A** HF/Silane interface (indicated by the white arrows) without discontinuity and failure/gaps (Original magnification X10000) and **B** mixed failure for CAD fabrication (Original magnification X150)

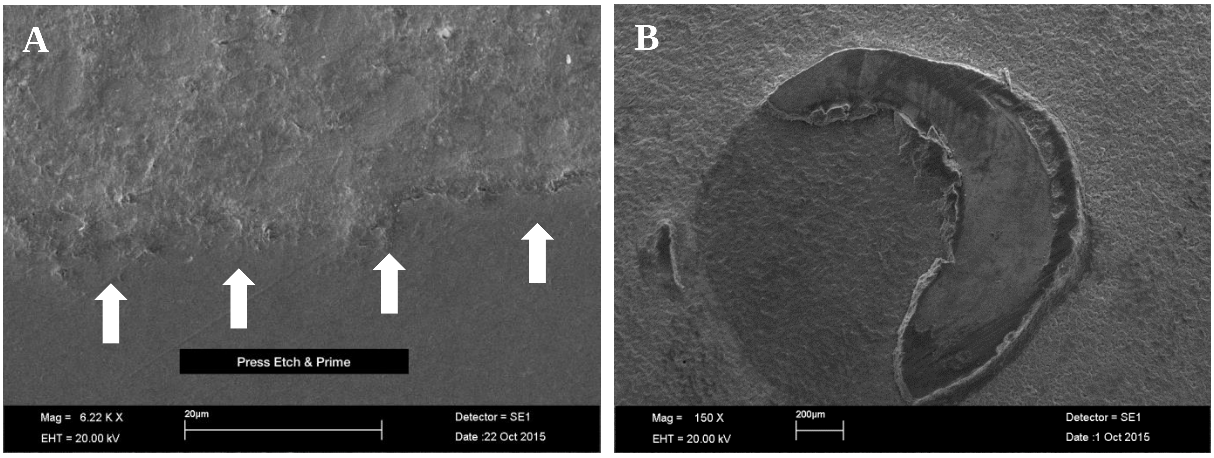

Fig 4 - SEM images. **A** Etch and Prime interface (indicated by the white arrows) without discontinuity and failure/gaps (Original magnification X6220) and **B** mixed failure for Press fabrication (Original magnification X150)

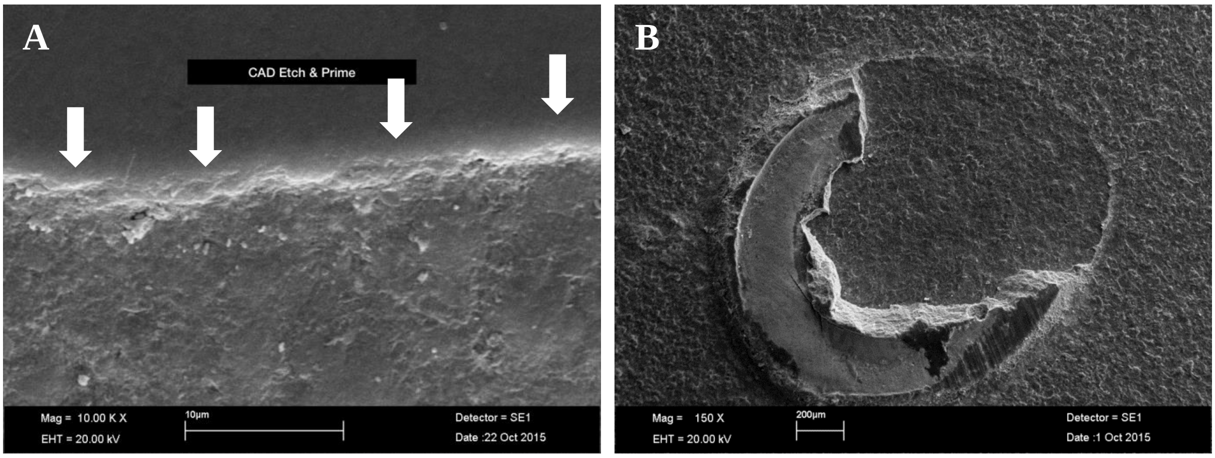

Fig 5 **-** SEM images. **A** Etch and Prime interface (indicated by the white arrows) without discontinuity and failure/gaps (Original magnification X10000) and **B** mixed failure for CAD fabrication (Original magnification X150)

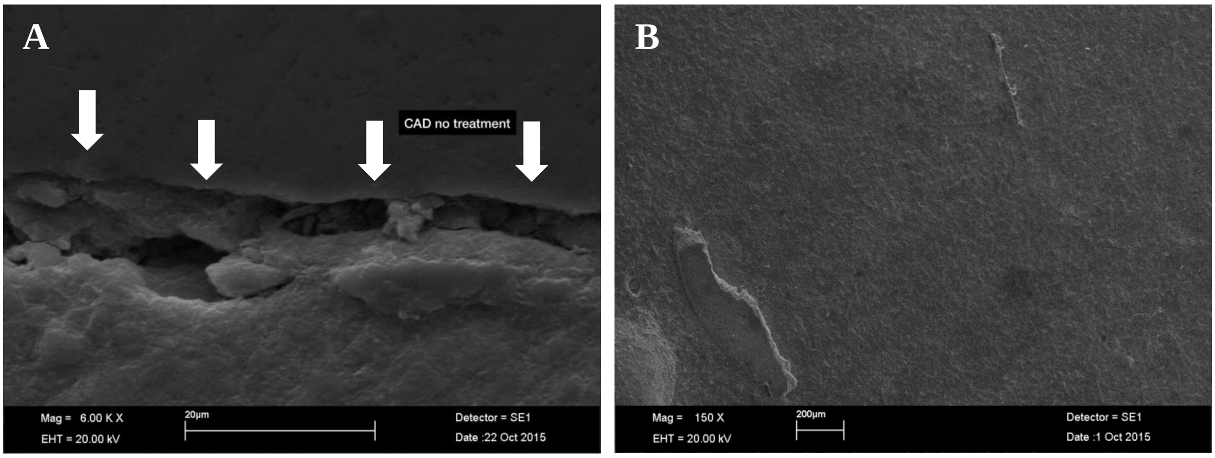

Fig 6 **-** SEM images. **A** No treatment interface (indicated by the white arrows) with discontinuity and failure/gaps (Original magnification X6000) and **B** adhesive failure for CAD fabrication (Original magnification X150)

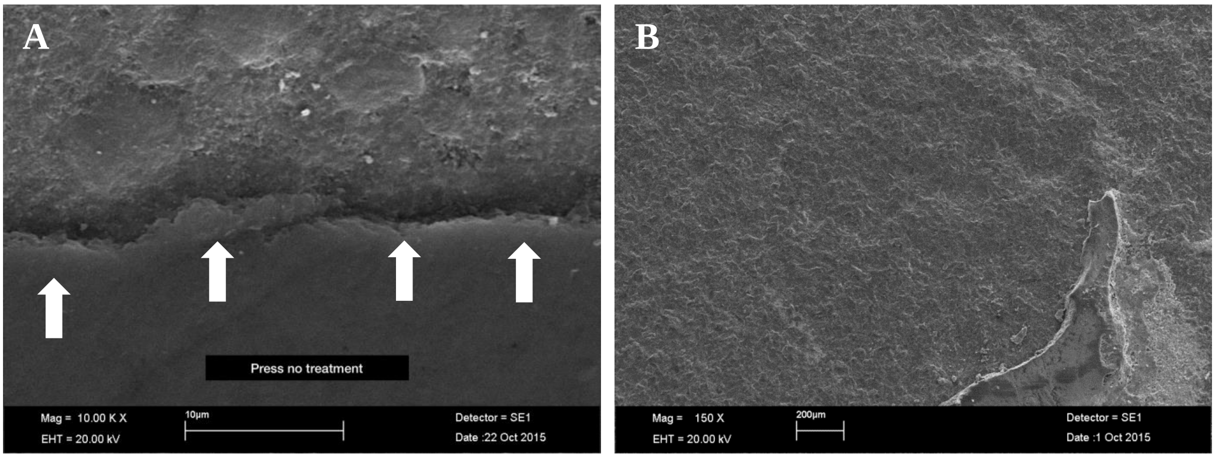

Fig 7 **-** SEM images. **A** No treatment interface (indicated by the white arrows) with discontinuity and failure/gaps (Original magnification X10000) and **B** adhesive failure for Press fabrication (Original magnification X150)

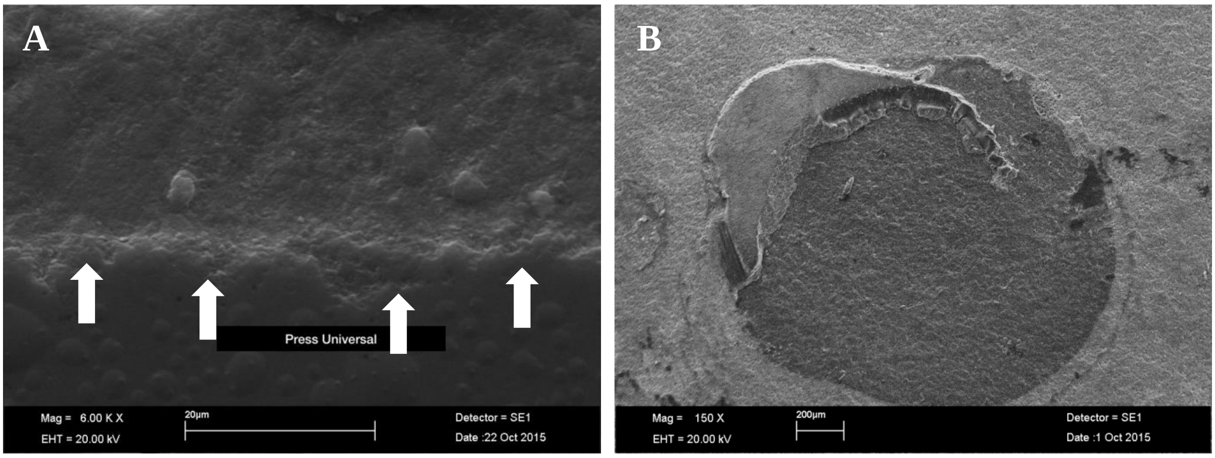

Fig 8 - SEM images. **A** Universal adhesive interface (indicated by the white arrows) without discontinuity and failure/gaps (Original magnification X6000) and **B** mixed failure for Press fabrication (Original magnification X150)

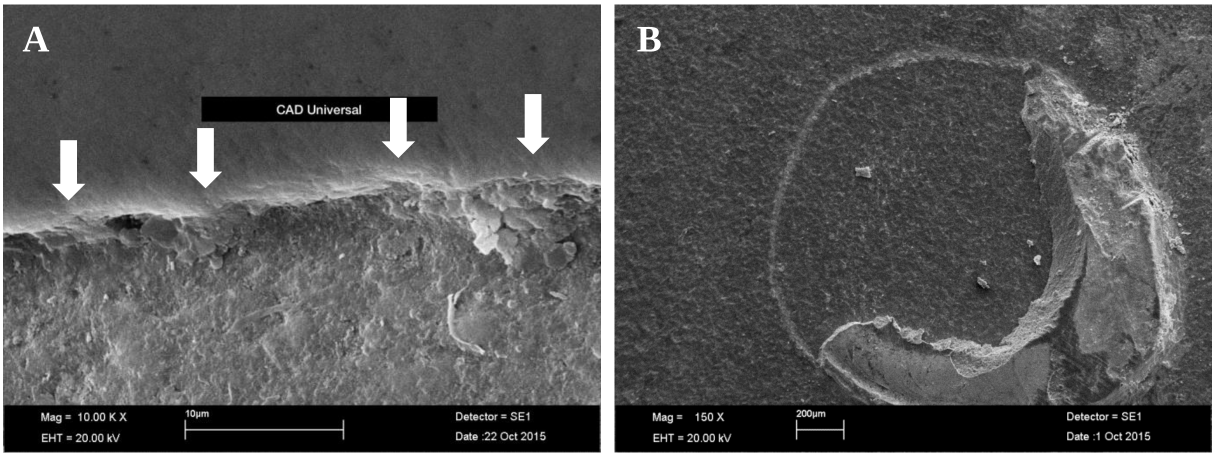

Fig 9 - SEM images. **A** Universal adhesive interface (indicated by the white arrows) without discontinuity and failure/gaps (Original magnification X10000) and **B** mixed failure for CAD fabrication (Original magnification X150)

3 CONCLUSÕES

1) O uso de ácido fluorídrico e adesivo universal provou ser o melhor tratamento de superfície para cerâmicas de dissilicato de lítio prensadas. Em contraste, o tratamento de superfície de cerâmicas CAD/CAM mostrou-se mais eficaz quando se utiliza ácido fluorídrico e silano.
2) Imagens MEV mostraram descontinuidade significativa e presença de falhas/gaps em cerâmicas sem tratamento de superfície, mas os mesmos achados não foram encontrados entre os demais tratamentos.
3) O padrão de fratura entre cerâmica e cimento resinoso mostrou tanto áreas de falha de cimento resinoso como de agentes de união, exceto em amostras sem tratamento que apresentaram ausência quase total de resíduo de cimento resinoso.

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APÊNDICE A – Procedimentos Laboratoriais

Confecção das Cerâmicas Prensadas

Fig. 1 Discos de resina acrílica fixados com cera na base do anel de silicone.

Fig. 2 Proporção para o revestimento IPS Press VEST Speed: 100g de revestimento, 16 mL de líquido e 11 mL de água destilada.

Fig. 3 Manipulação a vácuo.

Fig. 4 Inserção do revestimento no anel de silicone, previamente isolado com vaselina sólida, com suaves vibrações.

FIG. 5 APÓS A SUA PRESSA, O REVESTIMENTO FOI COLOCADO NO FORNO.

FIG. 6 FORNO IVOCLAR VIVADENT PRENSANDO A PASTILHA DE CERÂMICA PARA O INTERIOR DO REVESTIMENTO.

FIG. 7 CORTE DO BLOCO DE REVESTIMENTO PARA DESISNCLUSÃO DAS AMOSTRAS.

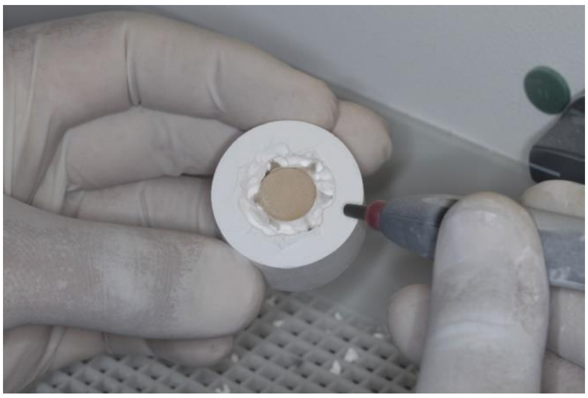

FIG. 8 JATEAMENTO PARA EXPOSIÇÃO DAS AMOSTRAS.

FIG. 9 JATEAMENTO SUAVE PARA REMOÇÃO DE REVESTIMENTO PRÓXIMO AS AMOSTRAS. EM SEGUIDAS PASSARAM PELO PROCESSO DE REMOÇÃO DA CAMADA DE REAÇÃO, ACABAMENTO E POLIMENTO, COM DISCO DIAMANTADO E BORRACHAS ABRASIVAS.

Confecção das Cerâmicas CAD/CAM

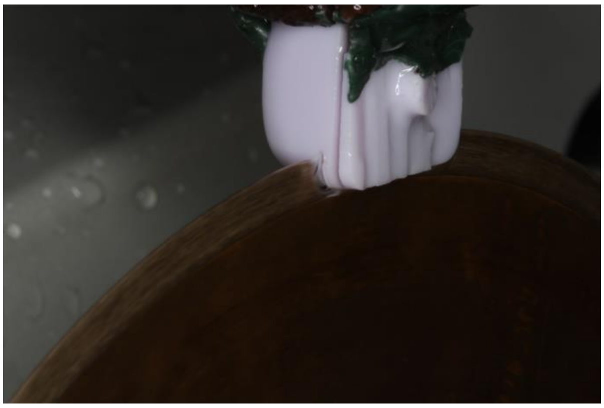

FIG. 10 CORTE DO BLOCO DE CERÂMICA CAD/CAM EM CORTADEIRA DE PRECISÃO COM DISCO DIAMANTADO.

FIG. 11 RESULTADOS DAS AMOSTRAS APÓS CORTE.

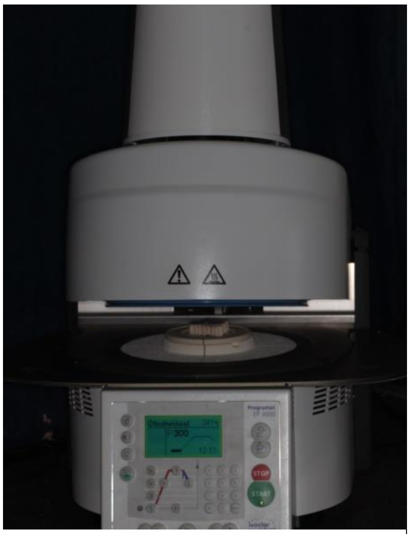

FIG. 12 PROCESSO DE CRISTALIZAÇÃO/SINTERIZAÇÃO.

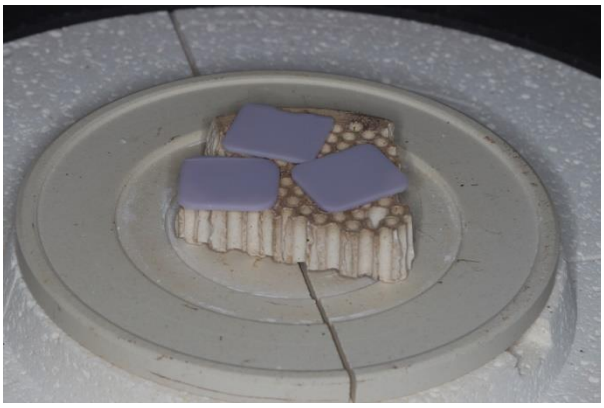

FIG. 13 ANTES DA CRISTALIZAÇÃO/SINTERIZAÇÃO.

FIG. 14 APÓS A CRISTALIZAÇÃO/SINTERIZAÇÃO.

Confecção de matriz de silicone

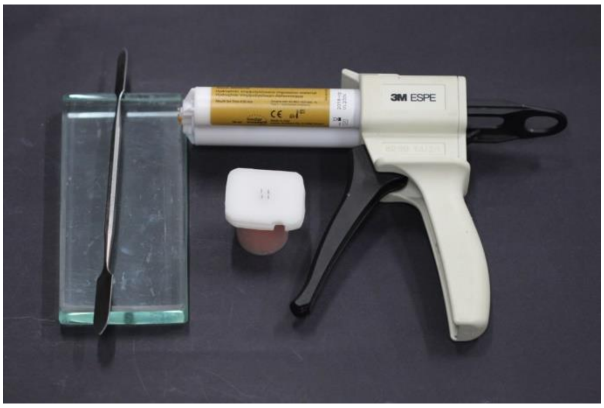

Fig. 15 Materiais envolvidos na confecção: placa de vidro, espátula #74, dispositivo/molde da mtriz, silicone por adição fluido, pistola dispensadora de silicone.

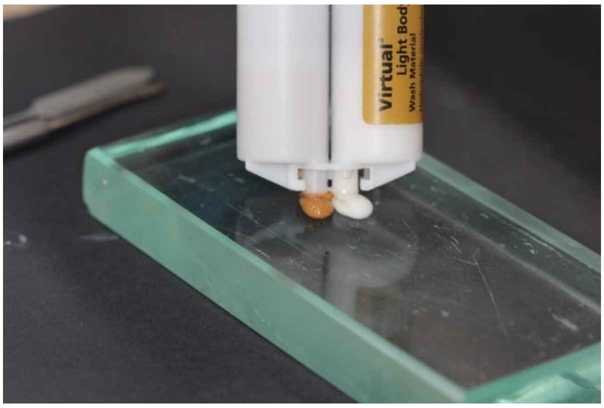

Fig. 16 Dispensa de material na placa.

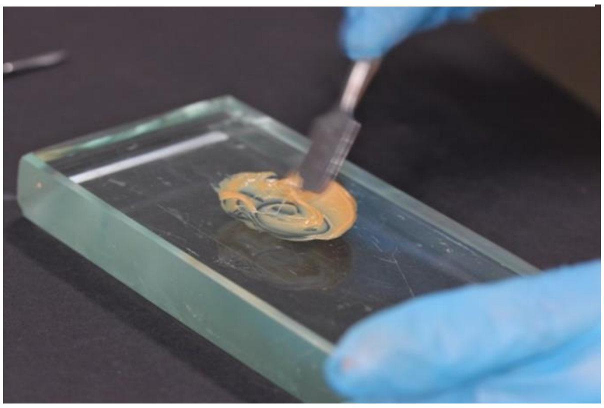

Fig. 17 Manipulação manual do material com movimentos circulares.

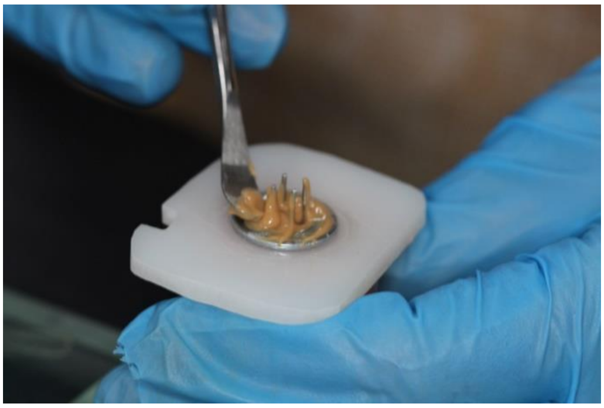

Fig. 18 Inserção de material no dispositivo/molde de matriz.

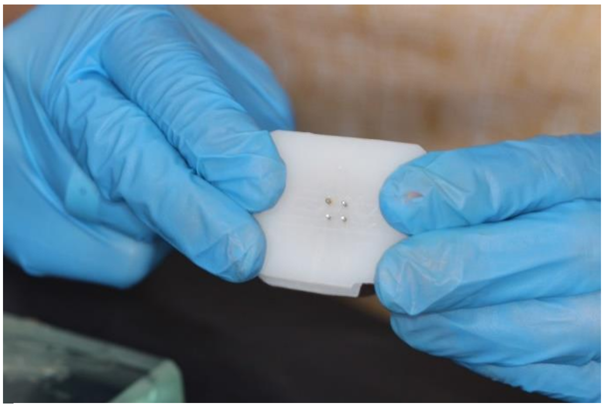

Fig. 19 Pressionamento da tampa do dispositivo.

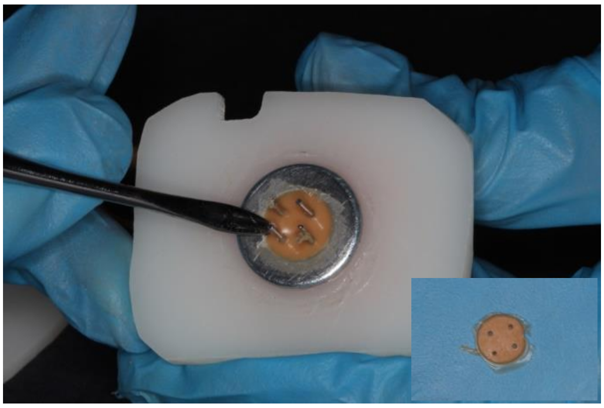

Fig. 20 Matriz removida do molde após a pressa do silicone.

Confecção dos cilindros de cimento resinoso

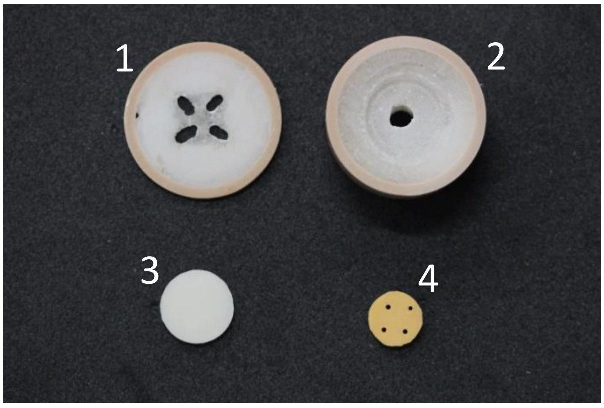

FIG. 21 MATERIAIS ENVOLVIDOS NA CONFECÇÃO DOS CILINDROS DE CIMENTO RESINOSO.1) TAMPA; 2) BASE; 3) AMOSTRA; 4) MATRIZ DE SILICONE.

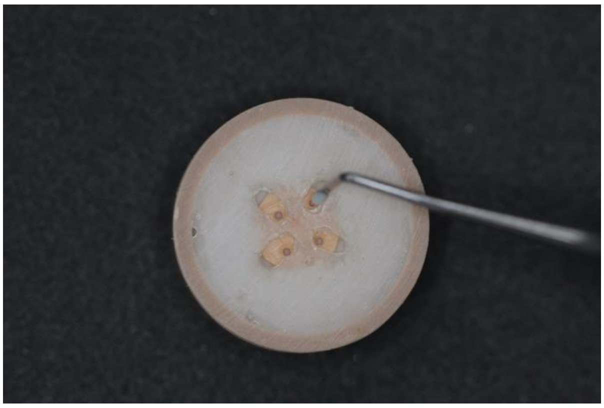

FIG. 22 A AMOSTRA FOI POSICIONADA SOBRE A BASE, A MATRIZ SOBRE A AMOSTRA E A TAMPA SOBRE A MATRIZ. LOGO EM SEGUIDA, O CIMENTO RESINOSO FOI INSERIDO NOS ORIFÍCIOS.

FIG. 23 VERIFICAÇÃO DA INTENSIDADE DE LUZ COM APARELHO RADIÔMETRO.

FIG. 24 FOTOATIVAÇÃO POR 10S COM A TAMPA.

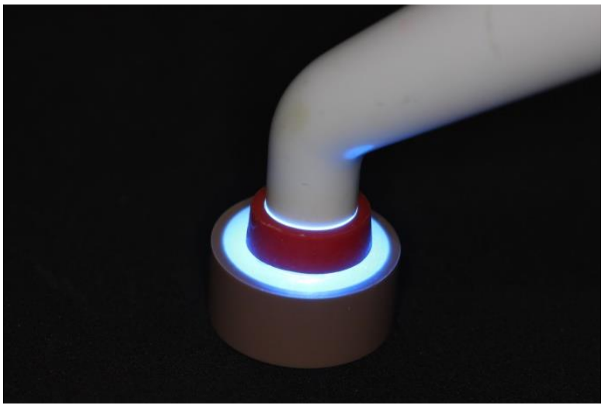

FIG. 25 FOTOATIVAÇÃO POR 40S SEM A TAMPA.

FIG. 26 CILINDROS DE CIMENTO RESINOSO SOBRE A AMOSTRA.

Teste de micro cisalhamento

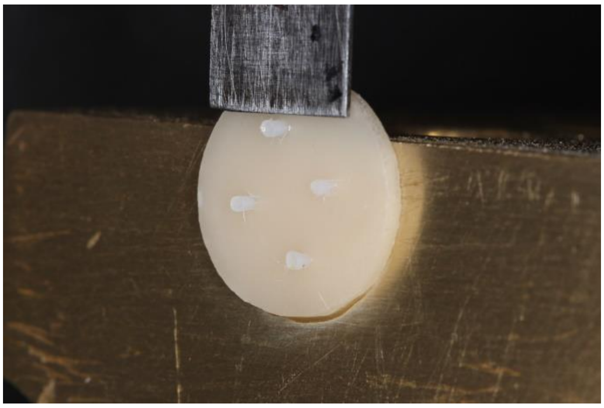

FIG. 27 POSICIONAMENTO DA AMOSTRA EM MÁQUINA EMIC DL 3000 PARA TESTE DE CISALHAMENTO. VISTA FRONTAL.

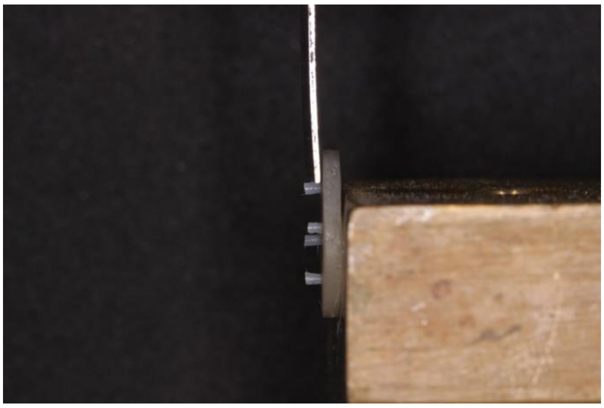

FIG. 28 POSICIONAMENTO DA AMOSTRA EM MÁQUINA EMIC DL 3000 PARA TESTE DE CISALHAMENTO. VISTA LATERAL.

Confecção de amostras para imagens em MEV para análise de interface cerâmica/cimento resinoso

FIG. 29 INSERÇÃO DE CIMENTO RESINOSO NA SUPERFÍCIE DE UMA AMOSTRA PRÉ-TRATADA.

FIG. 30 UNIÃO DE DUAS AMOSTRAS PREVIAMENTE TRATADAS.

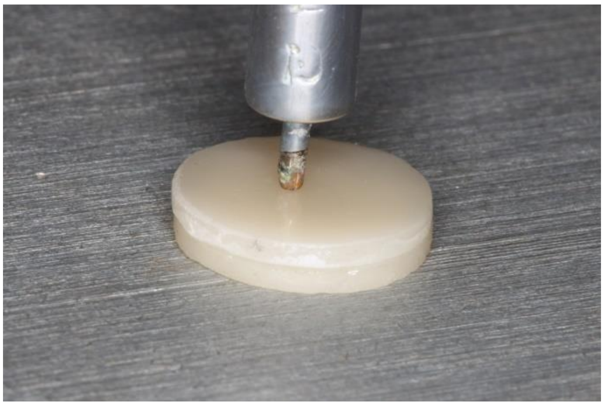

FIG. 31 PRESSIONAMENTO DAS AMOSTRAS.

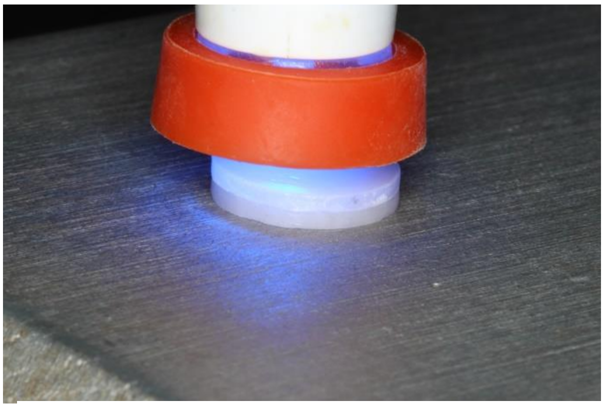

FIG. 32 FOTOATIVAÇÃO POR 40S. VERIFICAÇÃO DA INTENSIDADE DE LUZ PREVIAMENTE À FOTOATIVAÇÃO.

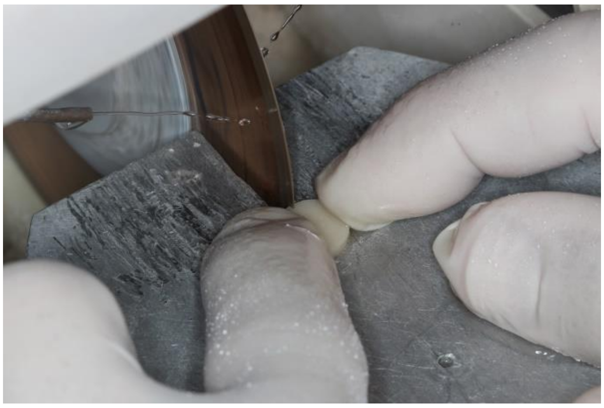

Fig. 33 Corte da amostra em máquina de corte com disco diamantado.

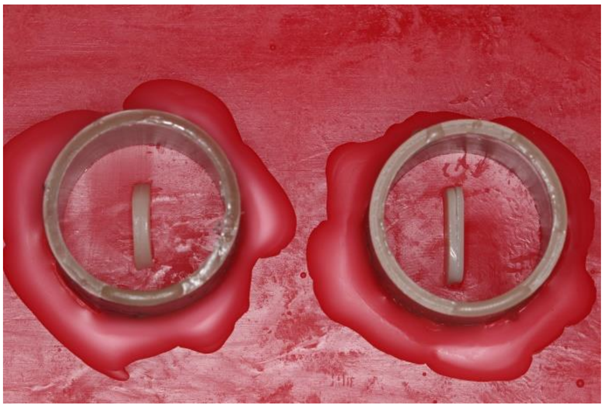

Fig. 34 Posicionamento das amostras em cera.

Fig. 35 Materiais utilizados na inclusão das amostras. A) Reina de poliestireno; B) Monômero; C) Catalizador.

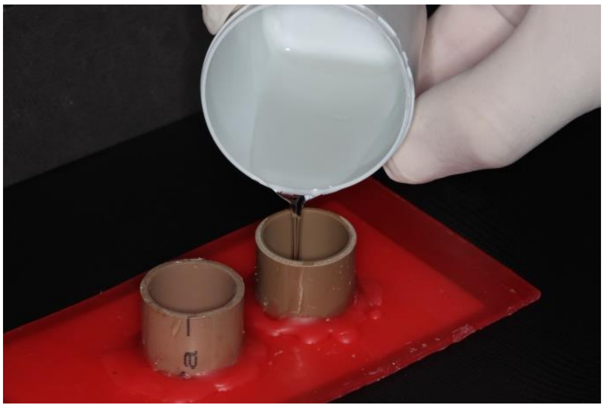

Fig. 36 Inserção da resina de poliestireno nos cilindros de PVC após manipulação.

Fig. 37 Amostras incluídas em resina de poliestireno após pressa do material.

ANEXO A – Operative Dentistry: Intruções para os autores

Novas instruções a partir de 20 de setembro de 2008

Operative Dentistry requires electronic submission of all manuscripts. All submissions must be sent to Operative Dentistry using the Allen Track upload site. Your manuscript will only be considered officially submitted after it has been approved through our initial quality control check, and any problems have been fixed. You will have 6 days from when you start the process to submit and approve the manuscript. After the 6 day limit, if you have not finished the submission, your submission will be removed from the server. You are still able to submit the manuscript, but you must start from the beginning. Be prepared to submit the following manuscript files in your upload:

- A Laboratory or Clinical Research Manuscript file must include:
  - a title
  - a running (short) title
  - a clinical relevance statement
  - a concise summary (abstract)
  - introduction, methods & materials, results, discussion and conclusion
  - references (see Below)
  - The manuscript **MUST NOT** include any:
    - identifying information such as:
      - Authors
      - Acknowledgements
      - Correspondence information
    - Figures
    - Graphs
    - Tables
- An acknowledgement, disclaimer and/or recognition of support (if applicable) must in a separate file and uploaded as supplemental material.
- All figures, illustrations, graphs and tables must also be provided as individual files. These should be high resolution images, which are used by the editor in the actual typesetting of your manuscript. Please refer to the instructions below for acceptable formats.
- All other manuscript types use this template, with the appropriate changes as listed below.

Complete the online form which includes complete author information and select the files you would like to send to Operative Dentistry. Manuscripts that do not meet our formatting and data requirements listed below will be sent back to the corresponding author for correction.

GENERAL INFORMATION

- All materials submitted for publication must be submitted exclusively to Operative Dentistry.
- The editor reserves the right to make literary corrections.
- Currently, color will be provided at no cost to the author if the editor deems it essential to the manuscript. However, we reserve the right to convert to gray

- scale if color does not contribute significantly to the quality and/or information content of the paper.
- The author(s) retain(s) the right to formally withdraw the paper from consideration and/or publication if they disagree with editorial decisions.
- International authors whose native language is not English must have their work reviewed by a native English speaker prior to submission.
- Spelling must conform to the American Heritage Dictionary of the English Language, and SI units for scientific measurement are preferred.
- While we do not currently have limitations on the length of manuscripts, we expect papers to be concise; Authors are also encouraged to be selective in their use of figures and tables, using only those that contribute significantly to the understanding of the research.
- Acknowledgement of receipt is sent automatically. If you do not receive such an acknowledgement, please contact us at editor@jopdent.org rather than resending your paper.
- **IMPORTANT:** Please add our e-mail address to your address book on your server to prevent transmission problems from spam and other filters. Also make sure that your server will accept larger file sizes. This is particularly important since we send page-proofs for review and correction as .pdf files.

REQUIREMENTS

- **FOR ALL MANUSCRIPTS**

1. **CORRESPONDING AUTHOR** must provide a WORKING / VALID e-mail address which will be used for all communication with the journal. **NOTE:** Corresponding authors MUST update their profile if their e-mail or postal address changes. If we cannot contact authors within seven days, their manuscript will be removed from our publication queue.

2. **AUTHOR INFORMATION** must include:
   - full name of all authors
   - complete mailing address for each author
   - degrees (e.g. DDS, DMD, PhD)
   - affiliation (e.g. Department of Dental Materials, School of Dentistry, University of Michigan)

3. **MENTION OF COMMERCIAL PRODUCTS/EQUIPMENT** must include:
   - full name of product
   - full name of manufacturer
   - city, state and/or country of manufacturer

4. **MANUSCRIPTS AND TABLES** must be provided as Word files. Please limit size of tables to no more than one US letter sized page. (8 ½ ” x 11”)

5. **ILLUSTRATIONS, GRAPHS AND FIGURES** must be provided as TIFF or JPEG files with the following parameters
   - line art (and tables that are submitted as a graphic) must be sized at approximately 5” x 7” and have a resolution of 1200 dpi.

- gray scale/black & white figures must have a minimum size of 3.5" x 5", and a maximum size of 5" x 7" and a minimum resolution of 300 dpi and a maximum of 400 dpi.
- color figures must have a minimum size of 2.5" x 3.5", and a maximum size of 3.5" x 5" and a minimum resolution of 300 dpi and a maximum of 400 dpi.
- color photographs must be sized at approximately 3.5" x 5" and have a resolution of 300 dpi.

- **OTHER MANUSCRIPT TYPES**

  1. **CLINICAL TECHNIQUE/CASE STUDY MANUSCRIPTS** must include:
     - a running (short) title
     - purpose
     - description of technique
     - list of materials used
     - potential problems
     - summary of advantages and disadvantages
     - references (see below)

  2. **LITERATURE AND BOOK REVIEW MANUSCRIPTS** must include:
     - a running (short) title
     - a clinical relevance statement based on the conclusions of the review
     - conclusions based on the literature review…without this, the review is just an exercise
     - references (see below)

- **FOR REFERENCES**

REFERENCES must be numbered (superscripted numbers) consecutively as they appear in the text and, where applicable, they should appear after punctuation.

The reference list should be arranged in numeric sequence at the end of the manuscript and should include:

1. Author(s) last name(s) and initial (ALL AUTHORS must be listed) followed by the date of publication in parentheses.
2. Full article title.
3. Full journal name in italics (no abbreviations), volume and issue numbers and first and last page numbers complete (i.e. 163-168 NOT attenuated 163-68)
4. Abstracts should be avoided when possible but, if used, must include the above plus the abstract number and page number..

5. Book chapters must include chapter title, book title in italics, editors' names (if appropriate), name of publisher and publishing address.
6. Websites may be used as references, but must include the date (day, month and year) accessed for the information.
7. Papers in the course of publication should only be entered in the references if they have been accepted for publication by a journal and then given in the standard manner with "In press" following the journal name.
8. **DO NOT** include unpublished data or personal communications in the reference list. Cite such references parenthetically in the text and include a date.

EXAMPLES OF REFERENCE STYLE

- Journal article: two authors
  Evans DB & Neme AM (1999) Shear bond strength of composite resin and amalgam adhesive systems to dentin *American Journal of Dentistry* **12(1)** 19-25.

- Journal article: multiple authors
  Eick JD, Gwinnett AJ, Pashley DH & Robinson SJ (1997) Current concepts on adhesion to dentin *Critical Review of Oral and Biological Medicine* **8(3)** 306-335.

- Journal article: special issue/supplement
  Van Meerbeek B, Vargas M, Inoue S, Yoshida Y, Peumans M, Lambrechts P & Vanherle G (2001) Adhesives and cements to promote preservation dentistry *Operative Dentistry* **(Supplement 6)** 119-144.

- Abstract:
  Yoshida Y, Van Meerbeek B, Okazaki M, Shintani H & Suzuki K (2003) Comparative study on adhesive performance of functional monomers *Journal of Dental Research* **82(Special Issue B)** Abstract #0051 p B-19.

- Corporate publication:
  ISO-Standards (1997) ISO 4287 Geometrical Product Specifications Surface texture: Profile method – Terms, definitions and surface texture parameters *Geneve: International Organization for Standardization* **1st edition** 1-25

- Book: single author
  Mount GJ (1990) *An Atlas of Glass-ionomer Cements* Martin Duntz Ltd, London.

- Book: two authors
  Nakabayashi N & Pashley DH (1998) *Hybridization of Dental Hard Tissues* Quintessence Publishing, Tokyo..

- Book: chapter
  Hilton TJ (1996) Direct posterior composite restorations In: Schwarts RS, Summitt JB, Robbins JW (eds) *Fundamentals of Operative Dentistry* Quintessence, Chicago 207-228.

- Website: single author
  Carlson L (2003) Web site evolution; Retrieved online July 23, 2003 from: http://www.d.umn.edu/~lcarlson/cms/evolution.html

- Website: corporate publication
  National Association of Social Workers (2000) NASW Practice research survey 2000. NASW Practice Research Network, 1. 3. Retrieved online September 8, 2003 from:http://www.socialworkers.org/naswprn/default