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LAUBER JOSE DOS SANTOS ALMEIDA JUNIOR

ESTUDO DO COMPORTAMENTO DE RESINAS BULK FILL EM RESTAURAÇÕES CLASSE I POR MEIO DE MICROTOMOGRAFIA COMPUTADORIZADA E MICROTRAÇÃO

SÃO LUÍS
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Tese apresentada ao Programa de Pós-Graduação em Odontologia da Universidade Federal do Maranhão como parte dos requisitos para a obtenção do título de Doutor em Odontologia.

Orientadora: Profª. Drª. Leily Macedo Firoozmand

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A Comissão julgadora da Defesa do Trabalho Final de Doutorado em Odontologia, em sessão pública realizada no dia / / , considerou o candidato.

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1) Presidente (Orientadora): Profª. Drª. Leily Macedo Firoozmand

____

2) Examinador: Prof. Profª. Drª Viviane Hass

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3) Examinador: Profª. Drª Karina Gama Kato Carneiro

____

4) Examinador: Profª. Drª Daniele Meira Conde

____

5) Examinador: Prof. Dr. Darlon Martins Lima

DEDICATÓRIA

Dedico este trabalho à minha amada Rafaela, aos nossos filhos e a toda minha família.

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"A melhor maneira que o homem dispõe para se aperfeiçoar, é aproximar-se de Deus. "

(Pitágoras)

RESUMO

A contração de polimerização (CP) e seus efeitos prejudiciais vêm sendo o foco de estudos com o intuito de melhorar o desempenho das resinas compostas. A simplicidade da técnica devido a possibilidade de preenchimento único em grande quantidade tem permitido que resinas bulk fill (RBF) sejam indicadas para minimizar essa contração. Esta tese, dividida em dois capítulos, teve por objetivo avaliar o comportamento de resinas bulk fill em restaurações classe I por meio de microtomografia computadorizada (µCT) e resistência de união por microtração (µTBS). O capítulo I avaliou a CP por µCT e a µTBS em restaurações classe I de RBF e convencional, e a correlação entre esses fatores. Cavidades classe I (4x5x4 mm), (fator-C=4,2) foram realizadas em terceiros molares livres de cárie, que foram randomizados e divididos em 4 grupos (n=6): Z350 XT(+): inserção incremental(II) e preenchimento manual (PM); Z350 XT(-): inserção única (IU) e PM; TBF (Tetric N Ceram Bulk Fill: IU e PM); e SFU [SonicFill: IU e preenchimento ultrassônico (PU)]. Os dentes foram escaneados e analisados em µCT em dois tempos: T0- após o preenchimento da cavidade com resinas compostas e T1- depois da polimerização para CP. Após 1 semana os dentes foram seccionados transversalmente no sentido vestíbulo-lingual e mesio-distal para obtenção de espécimes com aproximadamente 1mm² de espessura, e fixados em dispositivo para máquina de ensaio universal para realizar µTBS. Os dados demonstraram diferença estatisticamente significante para a CP entre os grupos Z350 XT(+) e Z350 XT(-) e entre SFU e Z350 XT(-) (Kruskal-Wallis e Dunn, $p<0.05$). A µTBS foi maior para Z350 XT(+) em relação aos demais grupos ($p<0.05$) e não houve correlação entre CP e a µTBS ($p>0.05$). Concluiu-se que RBF apresentaram CP semelhante à resina convencional nanoparticulada inserida de forma incremental. A resistência de união foi maior para a resina convencional inserida pela técnica incremental que apresentou menor falhas pré-teste, quando comparada às RBF. Não foi observada correlação entre a CP e a resistência de união quando utilizadas RBF e convencionais. O capítulo II avaliou o volume de contração de polimerização (VC), gap (VG) e poro (VP) usando µCT em restaurações classe I de RBF e convencional, e suas correlações. Cavidades classe I (4x5x4 mm), fator-C=4,2, foram realizadas em terceiros molares humanos livres de cárie que foram randomizados e divididos em 5 grupos (n=6): Z350 XT(+) (II/PM); Z350 XT (-) (IU/PM); TBF (IU/PM); SFM [(SonicFill (IU/PM)] e SFU (IU/PU). Os dentes foram escaneados e analisados em µCT em dois tempos: T0- após o preenchimento da cavidade com resinas compostas e T1- depois da polimerização para VG e VP, e VC (T1-T0). Após 1 semana os dentes foram seccionados transversalmente no sentido vestíbulo-lingual e mesio-distal para obtenção de espécimes com aproximadamente 1mm² de espessura, e fixados em máquina de ensaio universal para realizar µTBS. Os testes de Kruskal-Wallis e Dunn, demonstraram diferença estatisticamente significante no VC em µCT para os grupos Z350 XT(+) e Z350 XT (-) e entre SFU e Z350 XT(-), e diferença entre o VP para Z350 XT(+) e as RBF ($p<0.05$). Não houve diferença para o VG entre as RBF e convencional ($p>0.05$). Houve correlação positiva entre a VC versus VG e entre VC versus VP (Spearman, $p<0.05$). Concluiu-se que as RBF tanto de inserção sônica quanto manual mostraram contração de polimerização e formação de gap semelhante à resina convencional nanoparticulada inserida de forma incremental. Há uma correlação positiva entre a contração de polimerização e a formação de gap final, assim como entre a contração de polimerização e poro nas resinas compostas. O gap final foi formado principalmente pelo gap inicial gerado durante a inserção/preenchimento da resina na cavidade e não à contração de polimerização.

Palavra-chave: Polimerização, Microtomografia por Raio-X, Resinas Compostas, Resistência à Tração, Porosidade

ABSTRACT

The polymerization shrinkage (PS) and the detrimental effects have been the focus of studies with the aim of improving the performance of composite resins. The simplicity of technique due to the possibility of single fill in large quantities have allowed bulk fill composite resins (BFC) to be indicated to minimize this shrinkage. This thesis, divided in two chapters, aimed to evaluate the behavior of bulk fill composite resins in class I restorations by mans computerized microtomography (μCT) and microtensile bond strength (μTBS). Chapter I evaluated the PS and μTBS in BFC and conventional class I restorations composite resins, and the correlation between these factors. Class I cavities (4 x 5 x 4 mm), (factor-C = 4.2), were created in human third molars that were free of caries, which were randomized and divided into 4 groups (n = 6): Z350 XT(+): incremental insertion (II) and manual fill (MF); Z350 XT(-): single insertion (SI) and MF; TBF (Tetric N-Ceram Bulk Fill: SI and MF); and SFU [SonicFill: SI and ultrasonic fill (UF)]. The teeth were scanned and analyzed in μCT at two times: T0- after the filling of the cavity with composite resins and T1- after the polymerization for PS. After 1 week, the teeth were sectioned crosswise in the buccolingual and mesiodistal directions to obtain specimens with approximately 1 mm² thickness and fixed to universal test machine device to perform μTBS. The data showed a statistically significant difference for PS between the groups Z350 XT(+) e Z350 XT(-), and between SF e Z350 XT(-) (Kruskal-Wallis and Dunn, $p<0.05$). he μTBS was higher for Z350 XT (+) than the other groups ($p <0.05$) and there was no correlation between PS and μTBS ($p> 0.05$). It was concluded that the BFC type present a PS similar to that of the conventional nanoparticulate composite resin inserted using the incremental technique. The bond strength was higher for the composite resin incrementally inserted, which presented a lower number of pre-test failures when compared to bulk fill composites. No correlation was observed between the PS and bond strength when bulk fill and conventional composites were used. Chapter II evaluated the volume of polymerization shrinkage (VS), gap (VG) and void (VV) using μCT in BFC and conventional class I restorations, and their correlations. Class I cavities (4 x 5 x 4 mm), factor-C = 4.2, were created in human third molars that were free of caries, which were randomized and divided into 5 groups (n = 6): Z350 XT(+) (II/MF); Z350 XT(-) (SI/MF); TBF (SI/MF); SFM [(SonicFill: SI and MF)] and SFU (SI/UF). The teeth were scanned and analyzed in μCT at two times: T0- after the filling of the cavity with composite resins and T1- after the polymerization for VG and VV, and for VS (T1-T0). After 1 week, the teeth were sectioned crosswise in the buccolingual and mesiodistal directions to obtain specimens with approximately 1 mm² thickness and fixed in a universal testing machine to perform μTBS. Kruskal-Wallis and Dunn tests showed statistically significant difference in VS for groups Z350 XT(+) and Z350 XT (-), and between SFU and Z350 XT(-), and difference between VV for Z350 XT(+) and BFC ($p <0.05$). There was no difference in VG between the conventional and BFC ($p>0.05$). There is a positive correlation between VS *versus* VG and between VS *versus* VV (Spearman, $p <0.05$). It was concluded that BFCs of both sonic and manual insertion showed polymerization shrinkage and gap formation similar to the incrementally inserted nanoparticulate conventional resin. There is a positive correlation between polymerization shrinkage and final gap formation, as well as between polymerization shrinkage and void in composite resins. The final gap was formed mainly by the initial gap generated during the insertion/filling of the composite in the cavity and not to the polymerization shrinkage.

Key-words: Polymerization, X-Ray Microtomography, Composite Resins, Tensile Strength, Porosity

LISTA DE ILUSTRAÇÕES

Capítulo I

Capítulo II

LISTA DE TABELAS

LISTA DE ABREVIATURAS, SIGLAS E SÍMBOLOS

µCT	Microtomografia computadorizada
µTBS	Resistência de União à Microtração (microtensile bond strength)
ANOVA	Análise de Variância (Analysis of Variance)
CBF	Compósitos Bulk Fill
cm	Centímetros
CP	Contração de Polimerização
FAPEMA	Fundação de Amparo à Pesquisa e ao Desenvolvimento Científico e Tecnológico do Maranhão
Fator-C	Fator de Configuração Cavitária
Fpt	Falhas pré-teste
h	Horas
Kvp	Quilovoltagem
LED	Diódo Emissor de Luz (Light Emitting Diode)
mm	Milímetros
Mpa	Mega Pascal
mW	Megawatt
N	Newton
º C	Grau Celsius
RBF	Resina Bulk Fill
ROI	Região de Interesse (Region of Interest)
VC	Volume de Contração de Polimerização
VG	Volume de Gap
VOI	Volume de Interesse (Volume of Interest)
VP	Volume de Poro
MEV	Microscopia Eletrônica de Varredura
TDLV	Transformador Diferencial Linear Variável

SUMÁRIO

1. INTRODUÇÃO

Resinas compostas têm sido o foco de estudos com o intuito de melhorar o desempenho das restaurações, principalmente pela existência da contração e os seus efeitos prejudiciais após a polimerização (1,2). Há grandes evidências de que o estresse resultante da contração de polimerização nas resinas pode levar a efeitos deletérios, tais como infiltração marginal, deflexão de cúspide, trincas dentais, redução na resistência de união, formação de gaps e cáries secundárias. O controle desses efeitos é de suma importância e faz com que o fenômeno da contração de polimerização seja considerado clinicamente relevante (3).

Durante a polimerização das resinas compostas, ligações carbônicas duplas e simples formam cadeias poliméricas com ligações covalentes, resultando entre 1,5 a 5% de contração volumétrica (4). Quando a contração de polimerização supera a força adesiva induz à formação de fendas marginais, sendo que as incidências dessas fendas variam de 1,67 a 5,68% do volume total da restauração (5) podendo influenciar na resistência de união na interface dente/restauração.

Um dos fatores que exercem grande influência na contração de polimerização é o fator de configuração cavitária (fator-C). Cavidades com alto fator-C, tais como cavidades de classe I (5), levam a uma menor adaptação interna do material (6) e a um maior estresse na interface dente/restauração (7), conduzindo a uma maior formação de gap (8). Todavia, essas falhas podem ser minimizadas com a inserção incremental da resina composta, que promove a diminuição do fator-C (9). Até o momento, o preenchimento pela técnica incremental tem sido indicado a fim de diminuir a formação de gaps devido a contração de polimerização, mantendo uma adequada união da resina ao dente (10,11). Apesar dessa forma de inserção em pequenos incrementos ser empregada para diminuir os efeitos negativos durante a contração, nenhuma técnica é capaz de promover o completo selamento da cavidade (12).

Grandes esforços têm sido realizados para o desenvolvimento de materiais e técnicas que abordem novas estratégias de polimerização e controlem o seu efeito sobre a interface dente/restauração (1,2). Modificações na matriz orgânica das resinas têm permitido a evolução de novos materiais, como a “resina do tipo bulk fill” (RBF). Essa resina tem sido indicada para o uso em uma única camada, em cavidades profundas (4 a 5 mm) e com alto fator de configuração cavitária (Fator-C). Inicialmente, surgiu uma resina bulk fill *flow* (Surefil SDR), seguida de outras com baixa viscosidade e fácil manipulação (Venus Bulk Fill e Filtek Bulk Fill) sugerindo sua utilização como uma base a ser coberta com resina convencional e, também, por permitir uma melhor penetração (elevada fluidez) em cavidades de difícil acesso. Em seguida, as resinas compostas bulk fill “condensáveis” com viscosidade média (Tetric N-Ceram Bulk Fill e X-tra fil) foram desenvolvidas para serem utilizadas como base e até como incremento final da restauração, sendo manuseadas semelhantemente às resinas compostas microhíbridas convencionais. Posteriormente, surgiram as resinas bulk fill sônicas (SonicFill, Kerr, SonicFill 2) inseridas e ativadas sonicamente em única etapa com incrementos de até 5 mm.

Um diferente comportamento de contração de polimerização para as resinas bulk fill tem sido relatado (13), onde a natureza do sistema de monômeros determina a magnitude de contração e a sua tensão resultante. Monômeros, como o TEGDMA e o UDMA modificado são conhecidos por terem maior reatividade nas ligações duplas de carbono e grupos fotoativos no seu sistema, respectivamente, levando a uma menor contração. Ainda, o estresse gerado é dependente da configuração e conformação cavitária, da composição e da natureza visco-elástica do material (13).

Recentes estudos têm sugerido que as RBF podem simplificar (14) e com significativa longevidade (15) os passos operatórios na clínica. É relatado, ainda que o uso das RBF podem promover adequada adaptação marginal (16), baixa contração de polimerização (14), resultados satisfatórios para a resistência de união (9, 17) e baixa porcentagem de poros (18).

Ao considerar técnicas de preenchimento da cavidade com resinas compostas, o aumento da tensão de contração torna-se um fator de grande relevância, pois pode apresentar um efeito negativo sobre a resistência de união do material restaurador/dente (19,20), particularmente em cavidades com alto fator-C (21), onde a tensão de contração pode exceder a resistência de união (22). Nesse caso, resinas de baixa contração de polimerização, como as resinas bulk fill poderiam causar menor estresse sobre a interface dente/restauração.

Apesar do grande número de estudos relacionados à contração de polimerização, a presença de poros no interior da restauração é um aspecto negativo que tem sido amplamente ignorado na literatura (23). A sua presença pode acelerar o processo de deterioração do material, gerando infiltração marginal e descoloração, maior desgaste e menor resistência à flexão (24). Esses espaços podem ser incorporados durante o processo de fabricação ou durante o manuseio na inserção da resina (25,26). Assim, recomenda-se a redução do manuseamento do material, pois essa manipulação pode influenciar na incorporação de ar na matriz conduzindo a uma menor longevidade (25), contrastando entre a técnica incremental *versus* a técnica de preenchimento único (Bulk fill).

Além de influenciar na porosidade do material, sabe-se que a técnica de preenchimento e o tipo de resina podem ter grande impacto na adesão, particularmente em cavidades com alto fator-C (21). Entretanto, os métodos de microscopia utilizados para observar a resina composta, o preenchimento das cavidades e a adaptação marginal precisam submeter as amostras a cortes que geram um severo estresse, ocasionando alterações e formações de pequenas fissuras/fendas (27). Com essa conduta, surgem alguns questionamentos a respeito da fidelidade dos resultados observados, ou seja: se essas falhas ocorrem antes ou após a contração de polimerização, ou se falhas ocorrem durante o preenchimento da cavidade. Dentro deste contexto, recentemente, a microtomografia computadorizada (µCT) tem sido apresentada na literatura (10,27,28) como um método seguro e não destrutível, que pode analisar o comportamento do material em 3D sem deteriorar ou destruir o corpo de prova (29), sendo muito pouco utilizada para quantificar espaços e poros no material restaurador (23) e na interface dente/resina.

Não há evidências que correlacionem se uma menor contração de polimerização está associada a uma maior resistência de união para resinas do tipo bulk fill em restaurações classe I. Também, poucos estudos mostram o comportamento dessas resinas em relação à contração de polimerização, formação de gap e de poro quando inseridas manual e sonicamente. Assim, com o auxílio de um teste não destrutível (microtomografia computadorizada- µCT) e destrutível (resistência de união por microtração - µTBS), este estudo, *in vitro,* dividido em 2 capítulos, teve por objetivo: 1) Analisar a contração de polimerização e a resistência de união de restaurações classe I com alto fator-C, com diferentes resinas bulk fill e resina convencional, correlacionando esses fatores entre si, e 2) Correlacionar a contração de polimerização, presença de gap e poro em restaurações com resinas compostas convencional e bulk fill, por meio de análise em µCT. As hipóteses nulas testadas são de que: (1) não há diferença no volume de contração de polimerização e resistência de união entre resinas bulk fill e convencional; (2) não há diferença para a contração de polimerização, gap e poro entre essas resinas; (3) não há correlação para a contração de polimerização e resistência de união; (4) não há correlação para a contração de polimerização, gap e poros para as resinas bulk fill e convencional.

2. CAPÍTULO I - submetido à revista The Journal of Adhesive Dentistry

Correlation Between Polymerization Shrinkage and Bond Strength in Class I Restorations With Bulk Fill Composites Using Micro-CT

Abstract

Purpose: To evaluate polymerization shrinkage (PS) using computerized microtomography (µCT) and microtensile bond strength (µTBS) in bulk fill composites and conventional class I restorations as well as the correlation between these factors.

Materials and Methods: Class I cavities (4 x 5 x 4 mm), factor-C = 4.2, were created in third molars that were free of caries, which were randomized and divided into 4 groups (n = 6): Z350 XT (+), incremental technique; Z350 XT (-), bulk fill technique; TBF (Tetric N-Ceram Bulk Fill—manual insertion); and SF (SonicFill—sonic insertion). Each tooth was scanned twice in µCT: T0 was after filling the cavity with composite, and T1 was after photopolymerization. The data were analyzed by subtracting the composite volume for each time (T1 - T0). After 1 week, the teeth were sectioned crosswise in the buccolingual and mesiodistal directions to obtain specimens with approximately 1 mm² thickness and fixed in a universal testing machine to perform µTBS.

Results: The Kruskal-Wallis and Dunn tests showed a statistically significant difference for shrinkage in µCT among the Z350 XT (+) and Z350 XT (-) groups and between the SF and Z350 (-) groups ($p < 0.05$). Regarding the µTBS, all the groups differed from Z350 XT (-) ($p < 0.05$).

Conclusions: Bulk fill composites type present a polymerization shrinkage similar to that of the conventional nanoparticulate composite resin inserted using the incremental technique, but the bond strength was higher for the incremental group, which presented a lower number of pre-test failures when compared to bulk fill composites. No correlation was observed between the polymerization shrinkage and bond strength in the studied composites.

Keywords: polymerization; X-Ray Microtomography; composite resins; tensile strength

INTRODUCTION

Modifications in the organic matrix of composites have allowed for the evolution of new materials known as bulk fill composites (BFCs). These materials have been indicated for use in a single layer in deep cavities (4 to 5 mm) and with a high-factor cavity configuration (Factor-C), simplifying the steps in the clinic[33] and with significant longevity.[36] Recent studies have shown that BFCs have presented adequate marginal adaptation,[7] low polymerization shrinkage[33] and satisfactory results in bond strength. [24,32]

Even with the development of new polymeric materials, polymerization shrinkage (PS) of composites is an unavoidable phenomenon because it is an inherent property of these materials.[35] There is significant evidence that the stress of PS in the composites can lead to deleterious effects, such as marginal leakage, gap formation, cuspal deflection, tooth cracking, reduced bond strength, and lowered mechanical properties, which cause great concern about the control of these effects and make the phenomenon clinically relevant.[13]

PS reduces the composite's volume, which may promote a disunion at the dentin interface,[18] but these failures can be minimized by performing the incremental insertion technique, with the decrease of the C-factor.[32] So far, filling using the incremental technique has been recommended to decrease the formation of gaps due to PS and maintain adequate bonding of the composite to the tooth.[22,29] Although incremental insertion is employed to decrease the negative effects of shrinkage, no insertion technique is able to completely seal the cavity.[11]

It is known that the fill technique and the type of composite can have a great impact on adhesion, particularly in cavities with high C-factor.[39] However, the methods used to observe cavity filling and marginal adaptation require the samples to be subjected cuts that generate severe stress, causing changes and the formation of small cracks and creeps.[26] This raises some questions about the fidelity of the observed results, regarding whether these failures occur before or after the PS or if the failures occur during filling of the cavity. Another question is whether the failures, the PS, and inadequate filling could interfere with the bond strength of the restoration. Within this context, recent the microcomputed tomography (μCT) has been presented in the literature[21,26,34] as a safe and non-destructive method of analyzing a material's behavior in 3D without deteriorating or destroying the test body.[8]

When considering cavity-filling techniques with composites, increased shrinkage tension is of a great relevance and may present negative effects on bond strength[27,40]. There is no evidence that a lower PS is associated with higher bond strength for bulk fill composites as well as a slight lack of studies showing this correlation, mainly in class I restorations with the configuration factor, in which a shrinkage tension can exceed the bond strength .[42]

With the aid of non-destructive (microcomputed tomography—μCT) and destructible (bond strength to microtensile—μTBS) testing, the aim of this in vitro study is to analyze the PS and bond strength of class I restorations with high C-factor, with different bulk fill composites and a conventional composite, to correlate these factors with each other. The null hypotheses tested were that (1) there is no difference in the volume of polymerization shrinkage of bulk fill and conventional composites; (2) there is no difference in μTBS between bulk fill and conventional composites; and (3) there is no correlation between PS in μCT and μTBS between these composites.

MATERIALS AND METHODS

This study was submitted to the Research Ethics Committee in Brazil and approved under number 1708531.

One-way ANOVA was used to determine the sample size, considering 4 groups. From the data obtained by Fronza et al.[15] for inter-group variance (147.2) the intra-group variance (104.8), a sample size of 4 units per group was obtained for an alpha of 5% and power of 80%.

Sample Preparation

Twenty-four caries-free third molars were cleaned and stored in thymol at 0.5%, followed by prophylaxis and storage in distilled water at 37 ºC ± 1º C for 24 h.

Standard Class I cavities of 4 mm deep (4 x 5 x 4 mm) and with high C-factor (C = 4.2) were made with diamond tips no. 1090 and 1014 (KG SORENSEN, Cotia, SP, Brazil) at high-rotation refrigeration. The live internal angles were plumped[7] to make the force dissipation and adaptation of the material easier. The prepared teeth were randomly randomized and allocated in 4 experimental groups (n = 6), according to the composites used for restoration of the cavities (Table 1): [Z350 XT(+)]: Filtek Z350XT: incremental technique (positive control); [Z350 XT(-)]: Filtek Z350XT: bulk fill technique (negative control); (TBF) Tetric N-Ceram Bulk Fill, insertion according the manufacturer's instruction; and (SF) SonicFill, insertion according the manufacturer's instructions. The restorative materials and their respective information and filling/insertion techniques are described in Table 1.

Restorative Procedure

After the samples were prepared, the wells were conditioned with phosphoric acid at 37% (FGM, Joinville, Brazil) and the excess water was removed with absorbent paper, followed by application of the adhesive system Single Bond 2 (3M, ESPE, St. Paul, MN, USA) according to the manufacturer's instructions, and photopolymerized for 10 s. The composite was inserted according to Table 1 for each of the groups, and the condensation was performed with a cosmedent SP2 spatula (Cosmedent, Chicago, IL, USA); later, the composites were photopolymerized for 40 s. All of the polymerization procedures were performed with a high-power LED polymerization apparatus (Bluephase, Ivoclar Vivadent AG, Austria), with energy intensity above 1100 mW/cm².[38] The same operator performed the restorative procedures. Then, the specimens were stored in distilled water and maintained at 37 °C ± 1° C for 24 h.

Table 1: Technique and c*ompositions of Materials Investigated*

Materials (acronym) (group)	**Manufacturer**	**Composition**	**Insertion technique**	**Technical and filling**
Filtek Z350 XT [Z350 XT (+)]	3M ESPE, St. Paul, MN, USA	Non-agglomerated silica nanoparticles (20nm), non-agglomerated zirconia (4 to 11nm). 78.5 wt% and 63.3 vol%. Matrix: Bis-GMA UDMA, TEGDMA, PEGDMA and bis-EMA	1,5 mm	IT and MI
Filtek Z350 XT [Z350 XT(-)]	3M ESPE, St. Paul, MN, USA	Non-agglomerated silica nanoparticles (20nm), non-agglomerated zirconia (4 to 11nm). 78.5 wt% and 63.3 vol%. Matrix: Bis-GMA UDMA, TEGDMA, PEGDMA and bis-EMA	4 mm	BT and MI
Tetric N-Ceram Bulk Fill (TBF)	Ivoclar Vivadent, Schaan, Liechtenstein, GE	Barium aluminium silicate glass with two different mean particle sizes, an „Isofiller“, ytterbium fluoride and spherical mixed oxide), ivocerin initiator, 79-81 wt%, 61 vol% and 17vol% “Isofillers”. Matrix: Bis-GMA, Bis-EMA, UDMA	4 mm	BT and MI
SonicFill (SF)	Kerr, Orange, CA, USA	Barium glass, silicon dioxide (5-10%), oxide, chemicals (10-30%), MPS (10-30%), silicon dioxide, EBPDMA (1-5%), bisphenol A bis (2-hydroxy-3-methacryloxypropyl) ether (1-5%), and TEGDMA (1-5%) (Filler 83.5 wt%)	4 mm	BT and SI
Adper Single bond 2 (adhesive is a total etch)	3M ESPE, St. Paul, MN, USA	Bis-GMA, HEMA, dimetthacrylates, photoiniciator, methacrylate functional copolymer of polyacrylic and polyitaconic acids, 10% by weight of 5 nanometer-diameter spherical silica particles, water, ethanol.	Apply two consecutive coats of adhesive to the tooth surface with gentle agitation for 15 seconds; gently air thin; light cure for 10 seconds	

Bis-EMA: Bisphenol-A polyethylene glycol diether dimethacrylate; Bis-GMA: Bisphenol-A diglycidyl ether dimethacrylate; EBPDMA: Ethoxylated Bisphenol-A-dimethacrylate; TEGDMA: Triethylene glycol dimethacrylate; PEGDMA: polyethylene glycol dimethacrylate; UDMA: Urethane dimethacrylate, MPS: 3-(trimethoxysilyl) propyl methacrylate; HEMA: 2-Hydroxyethyl methacrylate. IT: Incremental Technique; BT: Bulk Fill Technique; MI: Manual Insertion; SI: Sonic Insertion.

Microcomputed tomography (µCT) Scanning and Analysis

Images were obtained using microtomography (SkyScan 1176, Kontich, Antwerp, Belgium). The µCT scanning occurred in 2 times: T0 was after the insertion of the composite, and T1 was after the final polymerization (Figure 1). The microtomography acted under the operating conditions for the energy device (70 Kvp—114 microamps) with a resolution of 18 µm. The images were standardized using the DataViewer® software, and analyses were performed in CTAnalyser®. All of the calculations were performed over the volume of interest (VOI) obtained from the region of interest (ROI) centered on the delimitations of the restorative material. The volumetric analyses were aimed at quantify the composite volume at 2 different moments (T0 and T1). The final shrinkage volume was measured by the difference between the times.

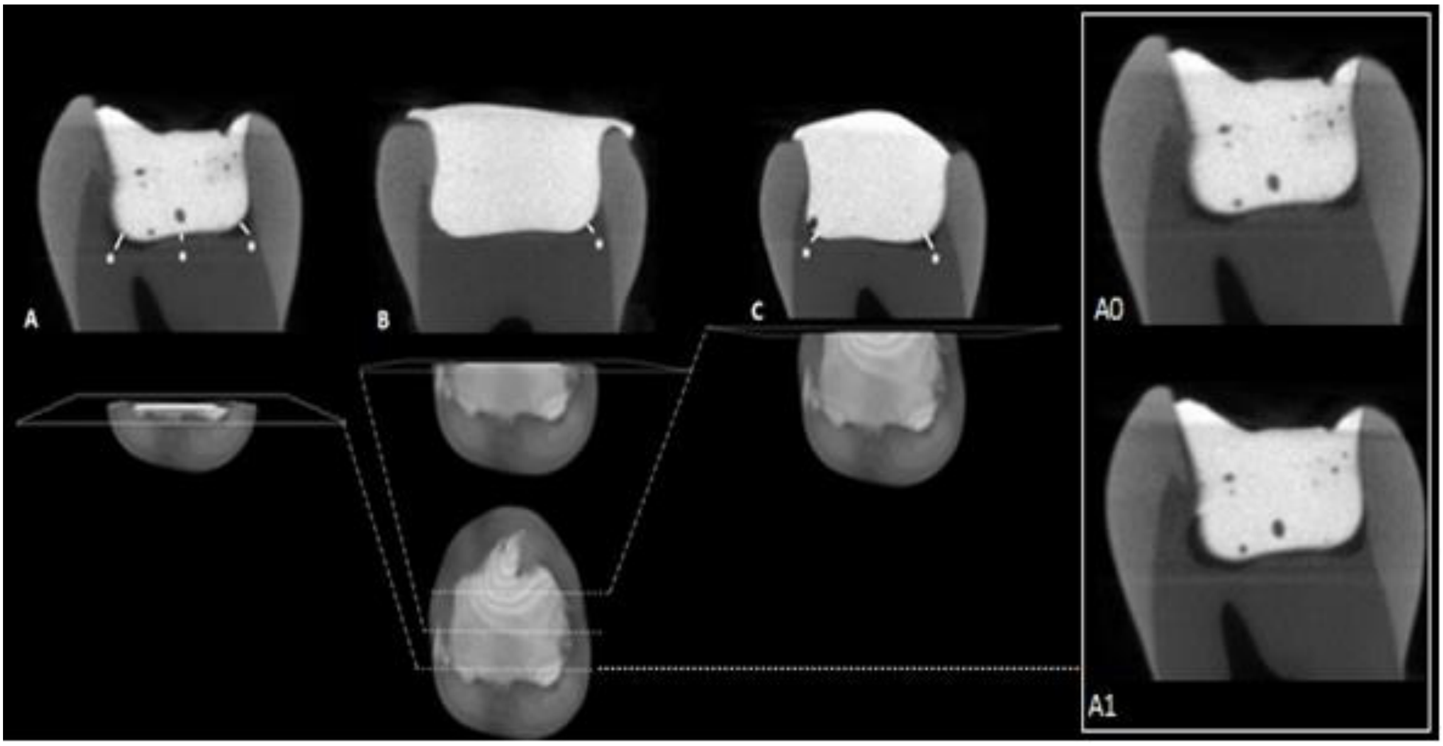

Figure 1. *Cavity filling with composite before curing (Tetric N Ceram Bulk Fill). (A): buccal third, (B) middle third, (C) lingual third. (*) presence of gaps in different sections in the same tooth before curing. A0 and A1: desadaptation of the composite before and after curing, respectively.*

Microtensile Bond Strength (µTBS)

After 1 week of storage in distilled water at 37 °C ± 1 °C, the teeth were transversely sectioned in the buccolingual and mesiodistal directions to obtain microspecimens with approximately 1 mm^2 thickness in serial cuts. The cross-sectional measurement was performed using a digital caliper (Mitutoyo Co., Tokyo, Japan). The specimens were fixed at their ends with ethyl cyanoacrylate-based glue (Atascadero, CA, USA) in a microtensile device coupled to a universal testing machine (INSTRON Equipment and Test systems Ltda., São José dos Pinhais, PR, Brazil). A speed of 1 mm/min was employed until the specimens fractured. The bond strength was expressed in megapascals (MPa), calculated by the ratio between the applied force (N) at the moment of fracture and the exposed area (A) (mm^2).

The type of failure was observed using a 50x magnification optical microscope (PanTec, Panambra Ind., and Technique AS, São Paulo, Brazil) and classified into 3 types: A (adhesive failure), C (cohesive failure in the composite or dentin), and M (mixed failure).

Statistical Analysis

The Shapiro-Wilk test indicated that the polymerization shrinkage values and bond strength did not present normal distributions. The data were analyzed using a Kruskal-Wallis test followed by a Dunn posthoc test for $p < 0.05$. The correlation between the bond strength and the volume shrinkage was obtained using Spearman's rank correlation coefficient ($p < 0.05$). The statistical software STATA 14 (College Station, TX, USA) was used for the data analysis.

RESULTS

Analysis of the polymerization shrinkage by μCT

The results for μCT were expressed in percentage volume of polymerization shrinkage (VS). For the volume of polymerization shrinkage, a statistically significant difference was observed between the Z350 XT (+) and Z350 XT (-) groups ($p = 0.03$) and the SF and Z350 XT (-) groups ($p = 0.012$) (Figure 2).

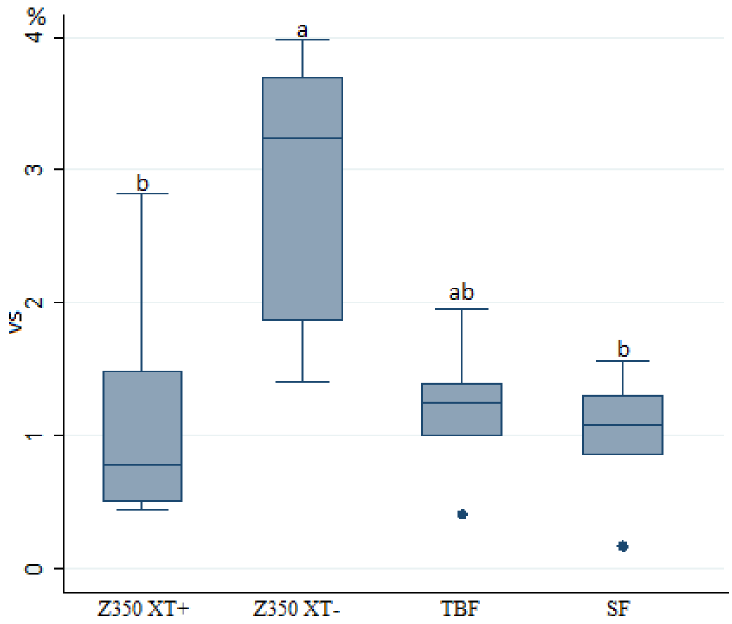

Figure 2. *Boxplot of the volumetric polymerization shrinkage in µCT. Groups with the same letter are not statistically significantly different (p > 0.05). VS: volumetric shrinkage; Z350 XT+: Filtek Z350 positive control; Z350 XT-: Filtek Z350 negative control; TBF: Tetric N-Ceram Bulk Fill; SF: SonicFill.*

Analysis of Bond Strength by Means of Microtensile Testing

The microtensile test showed a statistically significant difference in the bond strength of the studied groups ($p < 0.001$). The Z350 XT (+) group differed statistically significantly from all of the other groups in bond strength ($p < 0.001$). Also, the SF group differed statistically from the Z350 XT (-) group ($p = 0.049$). (Table 2).

Fewer failures were observed in the Z530 XT+ group. A predominance of adhesive and mixed faults was observed in all of the evaluated groups (Table 2). The values of µTBS ranged from 0 MPa (pre-test failure—Ptf) to 68.3 MPa. There was a statistically significant difference for the µTBS values when the pre-test failures with zero value ($p < 0.001$) were considered, but this statistical difference was not observed when these specimens were excluded from the analysis ($p = 0.50$).

Table 2: *µTBS results*

Experimental groups	**µTBS -ptf** (MPa) mean/SD	**ptf/n**	**µTBS +ptf** (MPa) mean/SD	**Failure Analysis** (%)
Z350XT(+)	30.9/9.0^{aA}	19/67	19.8/4.4^{aB}	A: 54 M: 30 C: 16

Z350XT(–)	24.6/14.5[aA]	64/67	1.62/3.97[cB]	A: 33 M: 67
TBF	30.9/13.6[aA]	56/71	7/7.96[bcB]	A: 26.7 M: 60
				A: 26.7 M: 60 C: 13.3
SF	34/16.1[aA]	52/72	8.6/12.0[bB]	A: 40 M: 40 C: 20

Z350 XT(+): Filtek Z350 XT (positive control). Z350 XT(-): Filtek Z350 XT (negative control). TBF: Tetric N-Ceram Bulk Fill; SF: SonicFill. SD: standard deviation; "-ptf" = without considering pre-test failures; "+ptf" = 0 MPa considering pre-test failures; n = number of specimens; A: adhesive; M: mixed; C: cohesive. Means followed by different letters differ from each other, columns to lowercase (Kruskal Wallis, p<0.01), rows to uppercase (Mann Whitney, p<0.05).

Correlation Between Shrinkage Volume and Bond Strength

A statistically significant correlation was not observed between bond strength and shrinkage volume (p = 0.21), with a weak negative association (r = -0.239) (Figure 3). Regarding the relationship between the bond strength and the number of specimens, the number of specimens obtained in the samples had a significant association with the bond strength in the group that considered the pre-test failures (p < 0.001). Each specimen had an increase of 2.37 MPa in bond strength.

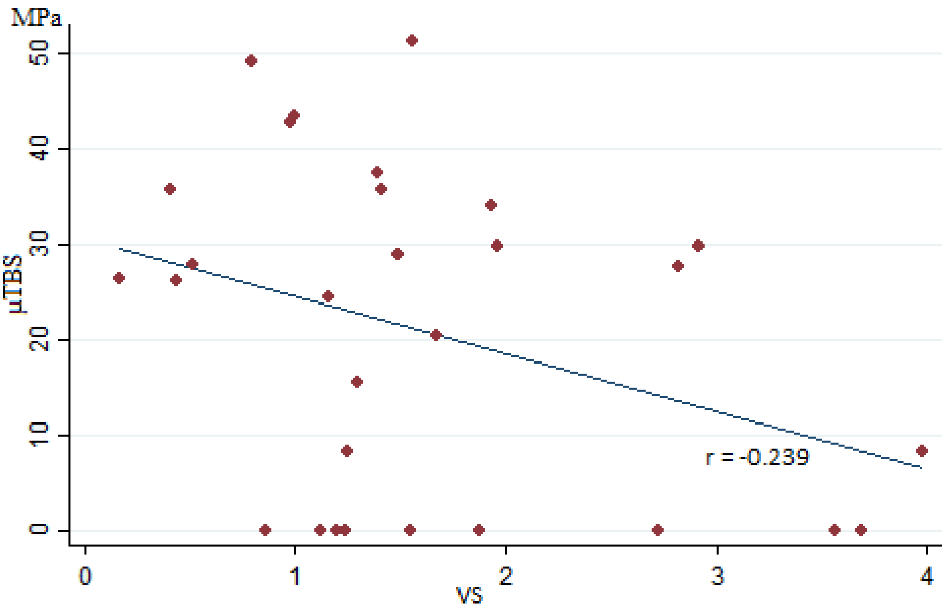

Figure 3. *Linear correlation between microtensile bond strength and volumetric shrinkage. VS: volumetric shrinkage. TBS: tensile bond strength.*

DISCUSSION

The desired appeal of simplifying procedures in clinical practice and the accelerated development of the single insertion of bulk fill composites confront the use of the already established incremental technique in conventional composites. The results of this study verify that the incremental insertion technique [Z350 XT (+)] and the sonic bulk fill technique (SF) presented lower volumes of PS and higher bond strength than the single insertion of conventional composite [Z350 XT (-)], but no correlation was found between these two factors. Thus, hypotheses I and II were rejected because there were significant differences in the polymerization shrinkage and bond strength of the bulk fill and conventional composite types. And the hypothesis III was not rejected because there was any correlation between the volume of PS and bond strength.

The determination of the VS was verified by means of μCT, a method recently shown in the literature as being efficient,[8] because it allows for better distribution of the dimension of the spaces along the walls in a 3D visualization. The μCT can be used effectively to characterize the volume of composites before and after the photopolymerization, thus allowing the determination of the shrinkages,[34,43] with the advantage of not destroying or promoting the induction of forces over the test body.[8] In addition, the evaluation by μCT can produce quantitative analyses of the PS—compared to the conventional methods, which are qualitative or semi-quantitative[43]—which are fundamental for determining the volume of shrinkage.

The microtensile test is widely used to verify the bond interface behavior of different materials and substrates.[5,30] The analysis of small areas of bond allows for a more uniform distribution of stress by obtaining a more homogeneous region with a smaller number of defects.[30,31]

The results of this study demonstrate a statistically significant difference for the %VS after the photopolymerization. The SF group presented lower shrinkage, followed by the Z350 XT (+), TBF, and Z350 XT (-) groups (Figure 2). Conventional nanoparticulate composites, such as the one used in this study, were introduced for their improved mechanical properties and reduced the shrinkages,[9] which explains the low observed shrinkage for the Z350 XT (+) group. The highest VS value being found for the Z350 XT (-) group is explained by the modification of the recommended technique (1.5 mm). For this material, the manufacturer does not indicate single insertions (4 mm), since insufficient polymerization in deeper portions of the material can compromise its physical properties.[3] The composites of this study, except for Z350 XT (-), presented low PS, which can be explained by the monomer system that comprises the materials (Table 1). According to the literature,[2] the magnitude of the composite's shrinkage is controlled by the material's composition. An increase in the loaded content may, until a certain point, reduce the polymerization shrinkage by decreasing the monomer content in relation to the charge/monomer.[6]

In this study, the bulk fill composites presented low shrinkage and no statistically significant differences between them ($p > 0.05$). The single insertion in bulk fill composite is allowed by modifications in the organic matrix that lead to a bond being formed to the tooth quickly, when compared to a conventional composite.[14] Currently, few studies have evaluated the polymerization shrinkage in class I cavities with high C-factor using µCT. These composites have been further studied in class II cavities with a lower C-factor.[7,17,24,28]

One of the explanations for the low shrinkage in the SF group may be the sonic insertion, which facilitates the material flow (up to 87%) during the filling,[1] allowing its use in deep cavities with a single increment. The low shrinkage observed in the TBF group may be due to the changes in the organic matrix. According to the manufacturer, TBF increases the polymerization depth through the insertion of a new initiator (Ivocerin), which, along with the camphorquinone/amine present in the composites, allow suitable polymerization, even in higher increments.[19] Moreover, the presence of prepolymerized particles of TBF can decrease the polymerization shrinkage.[23] Some factors may have contributed to the studied bulk fill composites presenting a low PS. To corroborate, Han and Park[17] did not observe a difference in the polymerization shrinkage between SF and TBF. However, when marginal integrity was evaluated, better results were observed for SF composite when compared to TBF.[28]

This study's results verify a statistically significant difference of bulk fill and conventional composites ($p < .001$) using µTBS. The evaluation of the restorations in deep cavities and with a high C-factor (C = 4.2) was intended to verify the behavior of these materials after a possible shrinkage stress in the walls of the cavity and its impact on the bond strength. Generally, the bond strength is lower in deep cavity preparations when compared to the insertion of the material on flat surfaces.[4] Other studies have shown that bond strength is greatly impacted by the cavity configuration factor[37,42] and by different types of bulk fill composites.[38]

The Z350 XT conventional composite with insertion using the incremental technique (+) showed higher bond strength values when compared to the bulk insertion (-) and bulk fill composites ($p < 0.001$) (Table 2). Likewise, Colak et al.[10] found higher bond strength in a conventional composite that was incrementally inserted than in bulk fill composites TBF and SF. Still, the bulk fill composites presented higher bond strength than the Z350 XT (-) single insertion (negative control), corroborating with the literature.[24,39] When the incremental and bulk fill techniques were compared, regardless of composites, better results were found for the incremental technique.[24]

In the μTBS study, one of the important factors to be evaluated is the number of pre-test failures. A value of 0 MPa was assigned for pre-test failures because doing so is a widely accepted method in the literature.[38] The results of this study showed that there was a statistically significant difference between the groups when the values of the pretested lost specimens were considered to be 0, (Table 2). Likewise, Van Ende et al.[38] observed higher values for the bond strength of the Surefil SDR composite, which did not present pre-test failures among the specimens submitted to microtensile testing in Class I cavities. Differently, other bulk fill and conventional composites showed low bond strength values, because they had more pre-test failures. Different results can be obtained when not considering pre-test failures, such as a study[32] that found greater results for the bond strength of a BFC when compared to conventional composites.

The μCT methodology used in this study was fundamental to analyzing, in 3D, that pre-test failures can be associated not only with the cutting technique and specimens' preparation but also with bad adaptation of materials during the insertion/condensation of the composites in the cavity before polymerization. This demonstrates the high variability in bond strength results and emphasizes the need for further investigation.[38]

The correlation analysis between the PS in μCT and μTBS in bulk fill composites for posterior class I restorations with a high C-factor was not significant, although there were differences in the isolated PS values and bond strength of the studied composites (Figure 3). Guo et al.[16] observed that in the composites, the bond strength during the polymerization time follows a similar tendency to stress/shrinkage tension, with the bond strength decreasing as the composite's thickness increases due to reduced irradiance at the tooth/composite interface. Importantly, in addition to the PS, the type of adhesive system[20,27,39] and other factors may influence the bond strength.[37]

The presence of gaps and cracks in the dentin wall of the cavity before photopolymerization (Figure 1) raises doubts about whether pre-test failures are exclusively caused by polymerization shrinkage. Sagsoz et al.[32] observed satisfactory results of μTBS for bulk fill composites; however, studies that evaluate the bond strength without observing the marginal adaptation before and after photopolymerization may not be reliable. The preparation of the specimens to evaluate the μTBS, as well as the preparation to observe the tooth/composite interface in scanning electronic microscopy[26], can generate stresses and cracks in the test body. Differently, the images obtained by µCT can be used to analyze the same sample several times without deteriorating or changing test body´s structure.[26]

The weak correlation between the volume of the shrinkage and the bond strength (r = −0.2399) confirms the premise that the composites contract towards the walls that are adhered,[41] and not—as was believed before—that the composites contracted in a direction toward the light.[25] Thus, the interface may rupture if the shrinkage stress,[12] between 13 and 17 MPa, is higher than the bond strength between the composite and the adhesive system, resulting in cracks.[12] The obtained results verify that the bond strength, when not considering the pre-test failures, ranged from 24.6 to 34.0 MPa, higher than the values indicated by Davisson et al. [12]; that is, the non-polymerization shrinkage was able to interfere with the bond strength.

CONCLUSION

Within the limits of this study, it was concluded that the bulk fill composites type present a polymerization shrinkage similar to that of the conventional nanoparticulate composite inserted using the incremental technique, and the single insertion of the conventional composite leads to higher shrinkages values. The bond strength was higher for the composite Z350 XT(+), which presented a lower number of pre-test failures when compared to bulk fill composites. No correlation was observed between the volume of polymerization shrinkage and the bond strength of the class I restorations with high C-factor when bulk fill and conventional composites were used.

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CLINICAL RELEVANCE

Bulk fill composites present low polymerization shrinkage, similar to conventional composites using the incremental technique, while simplifying the clinical steps. A conventional composite presented higher bond strength when compared to bulk fill composites in class I restoration with high C-factor.

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3. CAPÍTULO II - submetido à Revista Brazilian Oral Research

Correlation between polymerization shrinkage, gap formation, and void in bulk fill composites using micro-CT

ABSTRACT

This in vitro study aimed to evaluate the volume of polymerization shrinkage (VS), gap (VG), and void (VV) using computerized microtomography (μCT) in bulk fill resin composites (BFR) and conventional class I restorations as well as the correlation between these factors. Class I cavities (4x5x4 mm), C-factor= 4.2, were performed on human caries-free third molars that were randomized and divided into 5 groups (n = 6): Z350 XT (+): incremental insertion; Z350 XT (-): single insertion (SI); TBF [(Tetric N Ceram Bulk Fill: SI and manual filling (MF)]; SFM (Sonic Fill: SI/MF); and SFU (SonicFill: SI and Sonic filling). The teeth were scanned and analyzed in μCT at two times: T0- after the filling of the cavity with resin and T1- after the polymerization for VG and VV, and for VS (T1-T0). There was statistically significant difference in the VS in μCT for the groups Z350 XT (+) and Z350 XT (-) and between SF and Z350 (-) as well as a difference between VV for Z350 XT (+) and BFR (Kruskal-Wallis,p <0.05), and no difference in the VG between the conventional and BFR (p>0.05). There was a positive correlation between VS *versus* VG and between VS *versus* VV (Spearman, p <0.05). It was concluded that the BFR presented VS and gap formation similar to the incrementally inserted conventional resin composites. There is a positive correlation between polymerization shrinkage and gap formation and void. The final gap formation is related to the initial gap and not to the polymerization shrinkage.

Key words: Polymerization; X-ray Microtomography; Composite Resins; Imaging, three-dimensional

INTRODUCTION

The resin composites have been the focus of many studies in order to improve the performance of restorations, mainly by the existence of shrinkage and stress due to the material polymerization.[1] There is great evidence that the stress resulting from the polymerization shrinkage in the resin composites may lead to deleterious effects such as marginal infiltration, cusp deflection, dental cracking, reduction in bond strength, low mechanical properties, and gap formation. The control of these effects is of fundamental importance and causes these phenomena to be considered clinically relevant.[2] Major efforts have been made toward the advancement and development of materials and techniques that approach new polymerization strategies, and they control their effect on the tooth/restoration interface.[1,3]

During the polymerization of the resin composites, double and simple connections of carbon monomers form polymer chains with covalent connections, resulting in 1.5 to 5% of volumetric shrinkage.[4] When the polymerization shrinkage overcomes the adhesive force, its leads to the formation of marginal cracks, being that the incidence of these cracks may range from 1.67 to 5.68% of the total restoration volume.[5] One of the factors that exerts great influence in the polymerization shrinkage is the factor of cavitary configuration (C-factor). Higher C-factor cavities, such as class I cavities,[6] may lead to lower internal adaptation of the material[7] and higher stress in the tooth interface/restoration,[8] which may lead to greater gap formation.[9] However, these failures can be minimized with the incremental insertion of the resin composites, which promotes the decrease of the C-factor.[10] This technique has been recommended in order to decrease the polymerization shrinkage and a possible gap formation between the material and the tooth.[11,12]

In recent years, bulk fill resin composites are being proposed to decrease the polymerization shrinkage and gap formation in the pulp wall,[13] inserted in a single thick layer (4-5 mm).[14,15] They seem to present low polymerization shrinkage and percentage of voids[1,6] besides presenting great clinical acceptance[17,18] by simplifying the restorative process.[14] Another factor is that this sonically inserted resin composite is pointed out in the literature[19] as a coadjuvant for the reduction of the size and the number of spaces/voids in the material.

Despite the great number of studies related to polymerization shrinkage, the presence of voids and spaces inside the restoration is a negative aspect and has been widely ignored in the literature.[19] Voids and spaces may accelerate the process of material deterioration, generating marginal infiltration and discoloration, higher waste, and lower inflection strength.[20] These spaces between the monomers may be incorporated during the manufacturing process or during resin composite insertion handling.[21,22] Thus, it is recommended to reduce the handling of the material because this manipulation may influence the incorporation of air in the matrix forming voids and lead to a lower longevity.[22]

The polymerization shrinkage and/or gap formation have been evaluated in the literature by means of different destructible tests,[23-25] making it impossible to perform a more detailed analysis of the resin composite body before and after the polymerization. The computerized microtomography (μCT) has been used to quantify and evaluate the polymerization shrinkage,[26] in order to examine the tooth/restoration interface, as well as other changes in the material. This type of analysis spares the need for cuts and stresses to the tooth, unlike other methods, such as scanning electron microscopy (SEM).[23] The μCT is a safe and non-destructible method that may analyze the material in 3D,[27] although little used to quantify spaces and voids in the restorative material.[19]

Few studies have shown the behavior of bulk fill resin composites inserted manually and sonically in relation to the polymerization shrinkage, gap, and void formation in the wide restorations class I types. Thus, this in vitro study aimed to correlate the polymerization shrinkage, gap, and void in cavities with a high C-factor restored with conventional composites and bulk fill, by means of analysis in μCT. The null hypotheses tested are that: (1) there is no difference in the volume of polymerization shrinkage (VS), gap (VG), and void (VV) between bulk fill and conventional resin composites; and (2) there is no correlation between polymerization shrinkage, gap, and void in bulk fill and conventional resin composites.

METHODOLOGY

This study was submitted to the Research Ethics Committee and approved under the number 1708531.

The data analysis was performed in the statistical software STATA 14 (College Station, TX, USA). The calculation to determine the sample size, by the inter-group variance (147.2) and the intra-group variance (104.8), suggested four specimens per group with the statistical power of 80% and an alpha of 5%.[29]

Sample preparation

Thirty human caries-free third molars were cleaned and stored in thymol at 0.5%. Subsequently, the prophylaxis and storage of the teeth were carried out in distilled water at 37 ± 1° C (24 h).

Standard class I cavities (4x5x4 mm) with a high C-factor (C=4.2) were made with diamond tips No. 1090 and 1014 (KG SORENSEN, Cotia, SP, Brazil) at high rotation under refrigeration. The live internal angles were rounded with tip 1014[25] in order to facilitate the material adaptation and the force dissipation. The prepared teeth were randomized and allocated in 5 experimental groups (n=6), according to the resin composites and insertion technique used; Z350 XT (+): Filtek Z350XT resin composite; Z350 XT (-): Filtek Z350XT; TBF: Tetric N-Ceram Bulk Fill resin composite; SFM: SonicFill; and SFU: SonicFill. The restorative materials and their respective insertion/filling information and techniques are described in Table 1.

Restorative procedure

After preparation of the samples, the cavities were conditioned with phosphoric acid at 37% (FGM, Joinville, SC, Brazil) for 30s in the enamel and 15s in the dentin. The excess of water was removed with absorbent paper, followed by the application of Adper Single Bond 2 adhesive system (3M, ESPE, ST Paul, MN, USA) according to the manufacturer's directions and then photopolymerized for 10s with the LED polymerization apparatus of high power (Bluephase, Ivoclar Vivadent AG, Austria). The resin composite insertion in each of the groups was performed according to Table 1, and the condensation was performed with a cosmedent SP2 spatula (Cosmedent, Chicago, IL, USA). The restorations were photopolymerized for 40s with 1200 mW/cm².[28] The same operator performed all the restorative procedures. The specimens were then stored in distilled water and kept in an oven at 37°±1°C for 24h.

Evaluation in computerized microtomography (µCT)

A computerized microtomography (SkyScan 1176, Kontich, Antwerp, Belgium) was used to analyze the restored cavities. Each tooth was scanned in two times: T0- after the insertion of the resin composite and T1- after the final polymerization. The microtomography acted under the operating condition for the power device (90 Kvp, 275 microamperes) with a resolution of 17.48 µm (filter Cu =0.1 mm). The average of the total number of slices was 250, with an average reading time close to 28 minutes. The images were standardized in the DataViewer® software and the analyses were performed in CTAnalyser®. All the calculations were performed on the volume of interest (VOI) obtained from the region of interest (ROI) centered on the delimitations of restorative material.

The volumetric analyses aimed to quantify the resin composite volume at two different moments. The initial reading (T0) was considered "reference," and the final reading (T1) was considered a "target" in order to align the images geometrically. From these measures, the volume of the polymerization shrinkage, gap, and void was calculated in percentage through analysis of the anatomical structure of the restoration.[27]

Table 1 Technique and materials used in the study.

Code	Material/Manufacturer	Composition	Batch No.	Insertion	Technical
Z350 XT(+)	Filtek Z350 XT/ 3M ESPE, St. Paul, MN, EUA	Non-agglomerated silica nanoparticles (20nm), non-agglomerated zirconia (4 to 11nm). 78.5 wt% and 63.3 vol%. Matrix: Bis-GMA UDMA, TEGDMA, PEGDMA and bis-EMA	387672	3 increments (≅1.3 mm each)	**IT/MF**
Z350 XT(-)	Filtek Z350 XT/ 3M ESPE, St. Paul, MN, EUA	Non-agglomerated silica nanoparticles (20nm), non-agglomerated zirconia (4 to 11nm). 78.5 wt% and 63.3 vol%. Matrix: Bis-GMA UDMA, TEGDMA, PEGDMA and bis-EMA	387672	Single increment (4 mm)	**BFT/ MF**
TBF	Tetric N-Ceram Bulk Fill/ Ivoclar Vivadent, Schaan, Liechtenstein, GE	Barium aluminium silicate glass with two different mean particle sizes, an isofiller. Ytterbium fluoride and spherical mixed oxide), ivocerin initiator, 79-81 wt%, 61 vol% and 17vol% “Isofillers”. Matrix: BisGMA, BisEMA, UDMA	U03089	Single increment (4 mm)	**BFT/ MF**
SFM	SonicFill/ Kerr, Orange, CA, EUA	Barium glass, silicon dioxide (5-10%), oxide, chemicals (10-30%), MPS (10- 30%), silicon dioxide, EBPDMA (1- 5%), bisphenol A bis (2-hydroxy-3- methacryloxypropyl) ether (1-5%), and TEGDMA (1-5%) (Filler 83.5 wt%)	5560135	Single increment (4 mm)	**BFT/ MF**
SFU	SonicFill/ Kerr, Orange, CA, EUA	Barium glass, silicon dioxide (5-10%), oxide, chemicals (10-30%), MPS (10- 30%), silicon dioxide, EBPDMA (1- 5%), bisphenol A bis (2-hydroxy-3- methacryloxypropyl) ether (1-5%), and TEGDMA (1-5%) (Filler 83.5 wt%)	5560135	Single increment (4 mm)	**BFT/ SF**
	Adper Single bond 2	Bis-GMA, HEMA, dimetthacrylates, photoiniciator, methacrylate functional copolymer of polyacrylic and polyitaconic acids, 10% by weight of 5 nanometer-diameter spherical silica particles, water, ethanol	N677700	**Apply two consecutive coats of adhesive to the tooth surface with gentle agitation for 15 seconds; gently air thin; light cure for 10 seconds**	

Bis-EMA: Bisphenol-A polyethylene glycol diether dimethacrylate; Bis-GMA: Bisphenol-A diglycidyl ether dimethacrylate; EBPDMA: Ethoxylated Bisphenol-A-dimethacrylate; TEGDMA: Triethylene glycol dimethacrylate; PEGDMA: polyethylene glycol dimethacrylate; UDMA: Urethane dimethacrylate, MPS: 3-(trimethoxysilyl) propyl methacrylate; HEMA: 2-Hydroxyethyl methacrylate. Incremental Technique (IT); Bulk Fill Technique (BFT); Manual Filling (MF); Sonic filling (SF).

Statistical analysis

The Shapiro-Wilk test has indicated that of the three evaluated outcomes, the polymerization shrinkage and void values did not present a normal distribution. These data were analyzed using Kruskal-Wallis, followed by Dunn posthoc ($p < 0.05$). ANOVA was used for the gap volume, followed by Tukey ($p < 0.05$). The correlation between the volume of shrinkage, void, and gap was obtained by the Spearman rank correlation coefficient. In all analyses, the significance level adopted was $p < 0.05$.

RESULTS

The results of the µCT analysis were expressed as a percentage of volumetric polymerization shrinkage, gap, and void.

Volume of polymerization shrinkage (VS)

Unlike the evaluation of the volume of gap (VG) and void (VV), for the analysis of the volumetric polymerization shrinkage (VS), the values of the resin composite volume difference in each time were considered (T1-T0) because this result represents the shrinkage of the studied material. Thus, a statistically significant difference was observed between the polymerization shrinkage volume of the Z350 XT (+) and Z350 XT (-) (p = 0.03) groups as well as SFU and Z350 XT (-) (p = 0.01) (Table 2).

Table 2 Volume of polymerization shrinkage (VS), gap (VG) and void (VV) using µCT.

Experimental groups	VS (%)	VG		VV	
	mean/SD*	VG0 (%) mean/SD*	VG1(%) mean/SD*	VV0 (%) mean/SD*	VV1(%) mean/SD*
Z350 XT+	$1.21/0.99^{b}$	$1.95/0.90^{a}$	$2.19/0.85^{a}$	$9.04/3.74^{a}$	$9.87/4.77^{a}$
Z350 XT–	$2.91/1.05^{a}$	$1.75/0.50^{a}$	$1.97/0.48^{a}$	$12.01/2.43^{ab}$	$13.35/3.28^{ab}$
TBF	$1.21/0.50^{ab}$	$1.80/0.67^{a}$	$2.30/0.75^{a}$	$18.23/2.27^{c}$	$19.27/2.39^{b}$
SFM	$1.69/0.59^{ab}$	$2.54/0,70^{a}$	$2.95/0.93^{a}$	$15.45/3.47^{bc}$	$16.56/3.66^{bc}$
SFU	$1.01/0.48^{b}$	$2.14/0.93^{a}$	$2.58/0.81^{a}$	$16.09/2.74^{bc}$	$16.75/2.88^{bc}$

"0": after the insertion of the composite resin; "1": after the final polymerization; SD: standard deviation. * Means with same superscript are not statistically different from each other ($p<0.05$).

Volume of gap (VG)

The gap volume was measured in nine different points of the pulp wall to the restoration (Figure 1), where the sum of these points determined the total volume of gap per tooth at two different moments: VG0 and VG1. The analysis of the gap and void volume on two different instances was intended to show the presence of gap after the resin composite insertion and after the polymerization of the material and that the analysis of the difference between the final and initial times only shows the size of the gap increase and not its real volume before and after the polymerization.

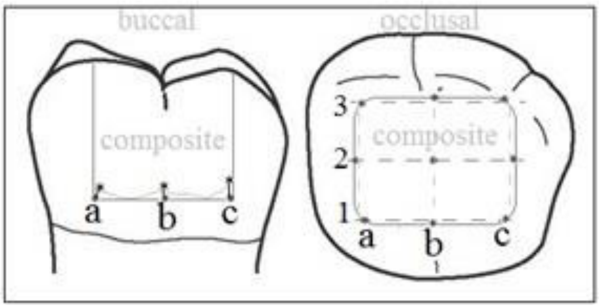

Figure 1 Analysis of gaps at resin/tooth interface.
* Point of gap measurement by region.

The presence of gap in the tooth/restoration interface in all the groups was observed before (VG0) and after (VG1) the photopolymerization (Table and Figure 2); however, there was no significant statistical difference for the presence of gap between groups on the two occasions: before (Kruskal-Wallis, p=0.43) and after the photopolymerization (p=0.64) (Table 2).

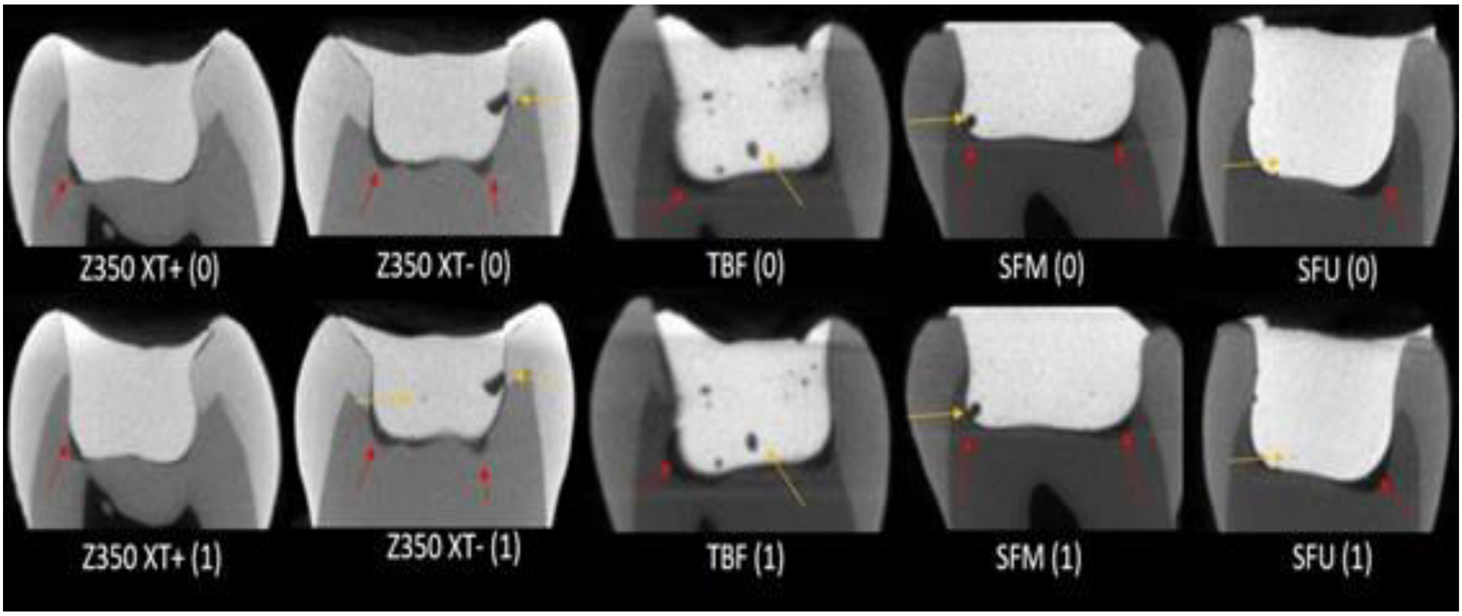

Figure 2 Presence of gaps and voids in the composite resins before (0) and after (1) evidenced by the arrows in
Z350 XT+: Filtek Z350 XT; Z350 XT-: Filtek Z350 XT; TBF: Tetric N-Ceram Bulk Fill; SFM: SonicFill (ma
filling); SFU: Sonic
Fill (sonic filling).

Volume of void (VV)

The void volume was considered for all the spaces, and voids and porosity observed in the body of the material before the photopolymerization (VV0) and after polymerization (VV1b). The analysis for the VV was made similar to VG and showed a statistically significant difference between the void volume before (VV0) photopolymerization for the resin composite Z350 XT (+) and TBF (p <0.001), Z350 XT (+) and SFM (p=0.008), Z 350 XT (+) and SFU (p=0.003), and Z350 XT (-) and TBF (p=0.01). In the same way, the difference between the resin composites was observed after the final photopolymerization (VV1) for Z350 XT (+) and TBF (p=0.001), Z350 XT and SFM (p=0.02), and Z 350 XT (+) (p=0.01).

Correlation between the volume of shrinkage, gap, and void

A moderate positive and statistically significant correlation (p=0.003, r=0538) was observed between the volume of shrinkage (% VS) and the volume of the final gap (%VG1) (Figure 3A) as well as a weak positive correlation between the volume of the polymerization shrinkage (% VS) and the volume of the final void (%VV1) (Figure 3B, p=0.009, r=0.476).

The linear regression analysis showed an association between the final gap volume (VG1) and the initial gap volume (VG0) in the pulp wall of the tooth/restoration interface (p<0.001).

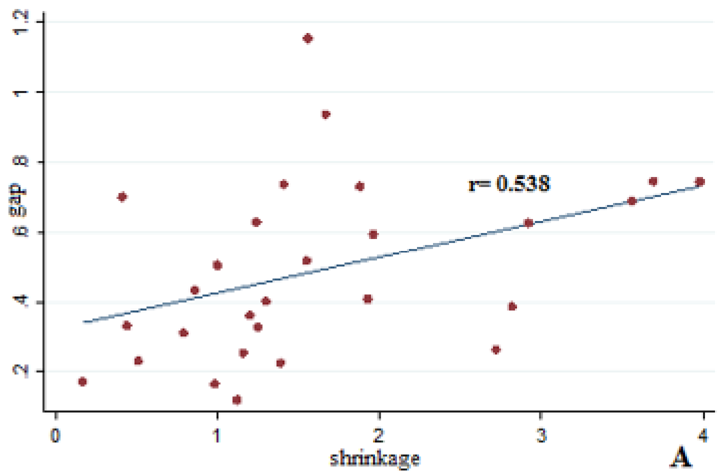

Figure 3A Correlation between the volume of polymerization shrinkage and final gap in the pulp wall of resin/tooth interface (p = 0.003).

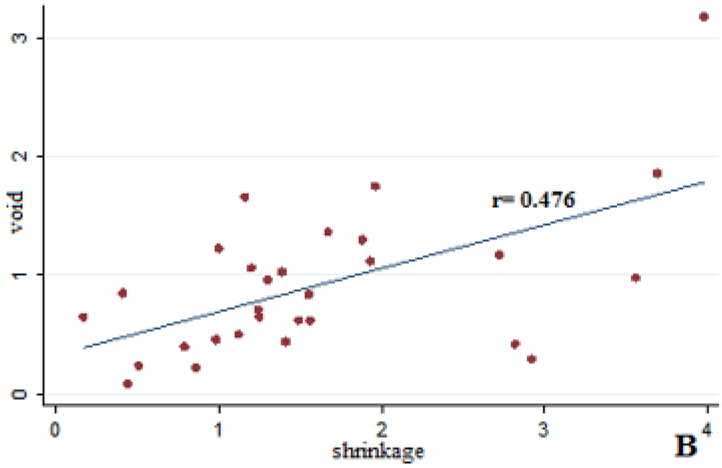

Figure 3B Correlation between the volume of polymerization shrinkage and final void (p = 0.009).

DISCUSSION

The results of the present study have demonstrated that, regardless of the insertion and filling technique, all the resin composites presented polymerization shrinkage, gap, and void. The first null hypothesis was partially rejected because it was observed that there was a difference between the volume of polymerization shrinkage for Z350 XT (+) and Z350 XT (-) and SFU and Z350 XT (-) and a void for Z350 XT (+) and the bulk fill resin composites; however, no difference was found for gap volume (VG) between the bulk fill and conventional resin composites. The second null hypothesis was rejected because the analysis showed a significant positive correlation between the polymerization shrinkage *versus* gap (p=0.003) and void (p=0.009) in the studied resin composites.

In the last years, the μCT has become an important tool to analyze the polymerization shrinkage,[26] gap formation,[27] and void.[19] The evaluation through μCT can produce quantitative analyses of the polymerization shrinkage when compared to conventional methods, which are qualitative or semi-quantitative.[30] Also, this type of analysis does not cause stress, deterioration, cracking formation, or destruction of the stick, thus allowing for the sample to be analyzed in 3D before and after treatment without interfering in the experiment, unlike scanning electron microscopy (SEM)[23] and other methods with two-dimensional analyses.

The volume of polymerization shrinkage of the materials studied in this study ranged from 1.01 to 2.91%, being considered an acceptable value in the literature (Ferracane, 2005). Statistically significant difference was observed between the groups Z350 XT (+) and Z350 XT (-) (p=0.03) as well as SFU and Z350 XT (-) (p=0.01). The low values of shrinkage observed in the studied resin composites may be explained by the increase in the inorganic load[31] because it is known that resin composites with a lower fill load percentage may have a shrinkage greater than those with a high percentage. The existence of high molecular weight monomers also influences the degree of resin composite shrinkage during the polymerization.[32]

A higher polymerization shrinkage was observed in Z350 XT (-), and this can be explained by the application of the single increment resin composite (4mm), unlike that from the incremental insert recommended by the manufacturer (Table 1). Low shrinkage values were observed for SFU (1.01%), and one hypothesis that may explain this behavior refers to the sonic insertion since the sonic energy released in the vibration facilitates the flow and decreases the viscosity to 87%.[33] However, in this study, the sonic insertion technique of SonicFill (SFU) did not show a better performance in relation to manual insertion of this same resin composite (SFM) or to that of Tetric N-Ceram (TBF) for the shrinkage volume, nor did it differ in the incremental insertion of Z350 XT ($p > 0.05$). In corroboration, Benetti et al.[34] also did not observe the difference in the polymerization shrinkage between SFU and TBF, but class II cavities were analyzed through a linear variable differential transformer (LVDT). Already, Orlowski et al.[35] observed better results for a SFU resin composite compared to TBF; however, their work verified the marginal sealing of cavities and not the polymerization shrinkage.

All the cavities presented a gap in the interface between the resin composite and the pulp wall (Figure 2), being that the final gap volume ranged from 1.97 to 2.95% between the groups. There was no difference between the initial gap volume (VG0) between bulk fill and conventional resin composites as well as for the comparison of the final gap volume (VG1) ($p > 0.05$). In this study, the sonic technique (SFU) did not influence the lower formation of a gap, because the bulk fill resin composites (SFM and TBF) that were inserted manually did not differ statistically from the resin composite inserted with sonic. In addition, Benetti et al.,[34] when using an LVDT and electron microscope, did not observe the difference when comparing the polymerization shrinkage, gap formation, and polymerization depth in the SFU and TBF resin composites in class II cavities. However, Kapoor, Bahuguna, and Anand[13] found better adaptability and a lower gap formation in the pulp wall when bulk fill resin composites were used in relation to a conventional resin composite, but the analysis was verified by SEM, which may generate doubts about the formation of the final gap found.[23]

The understanding of gap formation is a complex phenomenon and from the interaction of several factors.[34] The polymerization shrinkage is not the only factor involved in gap formation around the cavity edges because some other factors, such as cusp deflection,[36] type of cavity,[7,9,34] and insertion technique,[13] may also generate a gap. However, observing the presence of a gap in SEM or through other destructible tests generates an uncertainty about the period in which the same was formed, before and/or after the photopolymerization, because the preparation of the sample can cause stress and deterioration on the tooth/restoration interface, causing doubt about whether the gap occurred before or after the photopolymerization or if the failure was caused by the insertion technique and if different areas, mainly near the angles, were not properly selected in the cuts. Therefore, the analysis in μCT has become an accurate, safe, and non-destructible method to evaluate these materials in 3D.[23,27]

Such bulk fill as conventional resin composites presented voids in the body of the material (Table 2), in that a smaller void volume for the conventional resin composite was found ($p < 0.05$). Among them, bulk fill resin composites did not differ statistically ($p > 0.05$). It is reported in the literature that the handling of resin composites should be minimized to avoid the air incorporation by the operator.[22] Restoring cavities, especially deep ones, with increments of 2 mm thickness, may be delayed, and this implies a risk of incorporation of air bubbles during the incremental technique.[37] However, in the present study, the insertion/condensation of the conventional resin composite by the incremental technique resulted in reduction of the number of voids in the body of the material when compared to the sonic and manual technique of bulk fill resin composites ($p < 0.05$); one hypothesis is that the spatulation could reduce the number of bubbles or other defects present in the matrix during the material manufacturing process.

The results of the present study indicate a moderate positive correlation between polymerization shrinkage and the gap formation ($p=0.003$, $r=0.538$). Corroborating these results, a strong positive correlation was found in the literature,[34] although the polymerization shrinkage was not analyzed through LVDT for the polymerization shrinkage, and the gap was evaluated with visible scale in the microscope objective. This positive correlation, as found in the present study and in the literature,[34] demonstrates that there is an association between the polymerization shrinkage and the final gap. However, the presence of the final gap in the restoration seems to be associated more with insertion/filling of the material in the cavity (initial gap) than that with the polymerization shrinkage. This fact has not been previously described in the literature, and the linear regression model performed in the present study explained 89% of the final gap variation, being that the initial gap was the main related factor ($p < 0.001$). For each increase of 1mm³ in the initial gap, the final gap increased in 0.95mm³ maintaining the volume of void and polymerization shrinkage constants.

The correlation between void volume and polymerization shrinkage was significant and positive (Figure 3B). The regression analysis showed that 94.5% of the void volume increased; that is, the final void volume, was due to the polymerization shrinkage ($p < 0.001$). One hypothesis raised to explain these data is that molecular rearrangement of monomers in a smaller space[38] and the internally generated polymerization shrinkage forces[39] would increase the gap/void that would be near this region. However, it is suggested that further in vitro studies be conducted in order to determine the association of the insertion/filling technique with the void volume in the material.

Even with the development of new polymeric materials, the polymerization shrinkage is an unavoidable phenomenon because it is an inherent property of resin composites.[40] The positive correlations observed for the polymerization shrinkage show that this phenomenon may interfere in other factors and that non-destructible tests such as μCT can help to better understand these factors as the final gap formation in the restoration.

CONCLUSION

The use of bulk fill resin composites of both sonic and manual inserts in wide cavities shows polymerization shrinkage and gap formation similar to the conventional nanoparticulate resin composite when inserted in an incremental way. Also, there is a positive correlation between the polymerization shrinkage versus the formation of gaps and voids in the composite resin composites and the final gap formation is related to the initial gap and not to the polymerization shrinkage. It seems sensible to state that the final gap is formed mainly by the initial gap generated during the insertion/filling of the material into the cavity.

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4. CONSIDERAÇÕES FINAIS

O avanço de novas tecnologias como a microtomografia computadorizada, considerada um teste não destrutível, tem permitido uma análise satisfatória dos materiais utilizados na odontologia restauradora. Essa análise não destrutível permite que outros testes também possam ser realizados no mesmo material, proporcionando correlações com outros testes, como a microtração. Ao avaliar a resistência de união em microtração, palitos perdidos pré-teste devem ser considerados para possibilitar um resultado mais fidedigno. A análise em µCT, no presente trabalho, permitiu verificar a presença de contração de polimerização, gaps e poros em todas as resinas, independentemente do tipo convencional ou do tipo bulk fill. Ainda, a formação de gap foi melhor avaliada por meio desse teste, já que foi observado não só a presença do gap final, como é relatado na literatura, mas também um gap inicial na interface dente/restauração.

Apesar dos estudos de novos materiais poliméricos, a contração de polimerização é um fenômeno inevitável, pois é propriedade inerente das resinas compostas (47). O desenvolvimento das resinas bulk fill tem contribuído para o controle das propriedades indesejáveis, como menor contração de polimerização e os seus efeitos deletérios, além de possibilitar menor tempo clínico pela simplificação de passos (14) e uma considerável longevidade da restauração (15). Todavia, mais estudos laboratoriais e novos estudos clínicos devem ser realizados para avaliar o comportamento dessas resinas compostas e fomentar a sua utilização.

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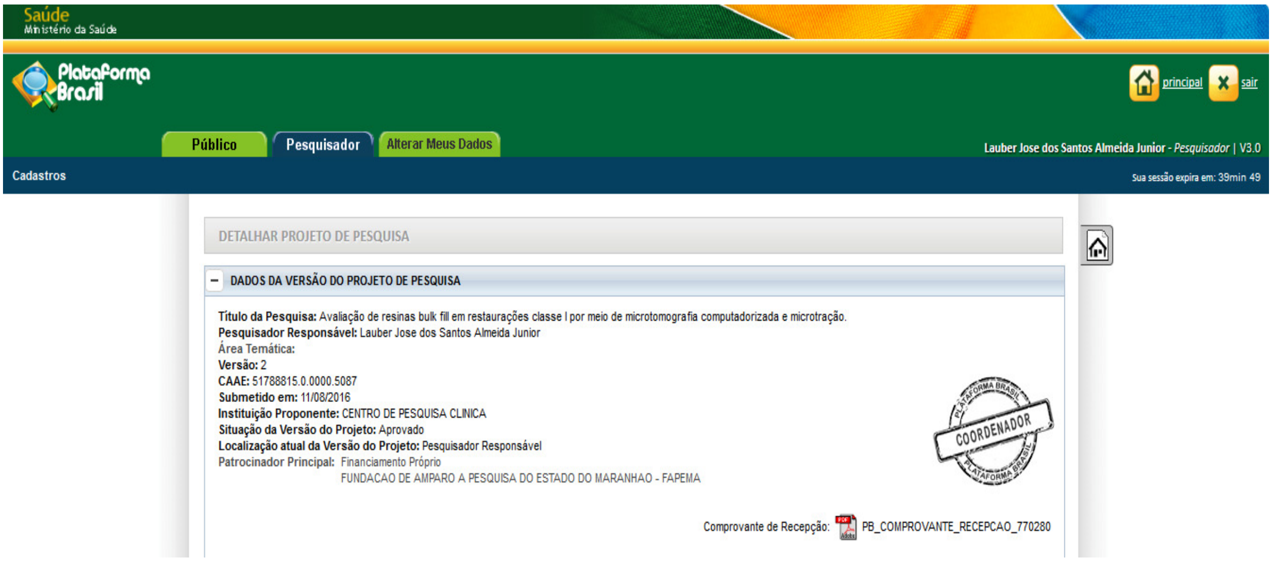
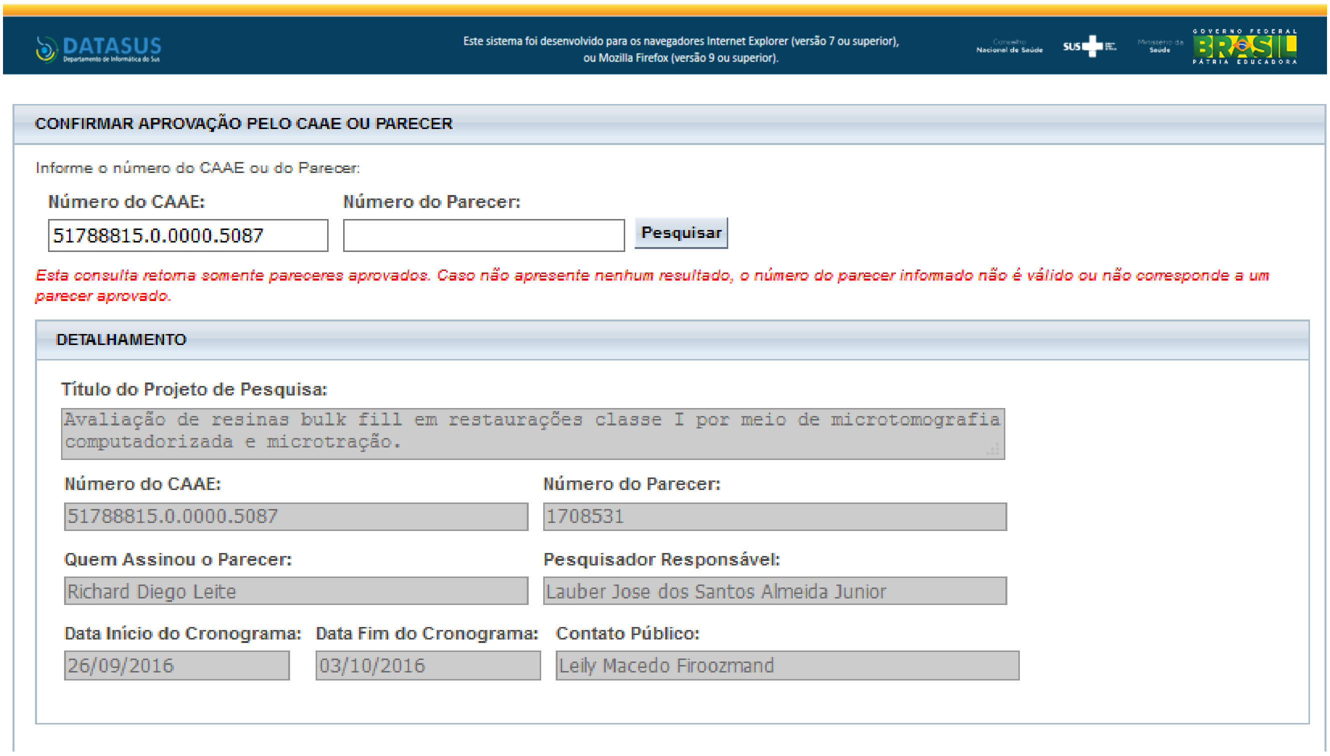
ANEXOS

ANEXO A – Aprovação do Comitê de Ética em Pesquisa (CEP/CONEP).

Link: http://aplicacao.saude.gov.br/plataformabrasil/login.jsf





PARECER CONSUBSTANCIADO DO CEP

DADOS DA EMENDA

Título da Pesquisa: Avaliação de resinas bulk fill em restaurações classe I por meio de microtomografia computadorizada e microtração. **Pesquisador:** Lauber Jose dos Santos Almeida Junior **Área Temática:**

Versão: 2

CAAE: 51788815.0.0000.5087

Instituição Proponente:CENTRO DE PESQUISA CLINICA

Patrocinador Principal: Financiamento Próprio
FUNDACAO DE AMPARO A PESQUISA DO ESTADO DO MARANHAO FAPEMA

DADOS DO PARECER Número do Parecer:

1.708.531

Apresentação do Projeto:

Recentemente um novo grupo de resinas compostas denominadas Bulk Fill (BF), foram lançadas no mercado, com o objetivo de serem utilizadas em um único incremento com espessura igual ou maior que 4 mm, otimizando o tempo de atendimento clínico e minimizando o stress de contração de polimerização. Porém, poucos resultados clínicos e laboratoriais indicam que as resinas compostas de baixa contração de polimerização apresentem vantagens significativas em sua indicação que encorajem os clínicos a implementarem essa técnica. Assim, o objetivo deste estudo é avaliar a contração de polimerização por meio de microtomografia computadorizada e a resistência de união por teste de microtração, restaurações realizadas em extensas cavidades do tipo classe I com diferentes técnicas de inserção de resinas bulk fill: fluída, fluída ativada sonicamente e condensáveis, após envelhecimento in vitro. Serão confeccionadas cavidades classe I (5 mm de

profundidade,x 4 mm de comprimento x 4 mm de largura) em terceiros molares hígidos extraídos por finalidade terapêutica. Os dentes serão alocados em 5 subgrupos (n=10) e restaurados segundo as recomendações dos fabricantes, distribuídos em G1 (BF fluída), G2 (BF condensável, G3 (BF fluída ativada sonicamente), G4(resina convencional
nanoparticulada) e G5(resina convencional nanoparticulada - técnica incremental). Os dentes serão

seccionados para obtenção de palitos com aproximadamente 1 mm² de espessura em cortes seriados e submetidos a teste de microtração para análise da resistência adesiva e, também, para análise do tipo de falha adesiva. Após o teste, a resistência de união será analisada estatisticamente. Também os dentes serão avaliados por meio de microtomógrafo a contração de polimerização por subtração antes e após a fotopolimerização.

Objetivo da Pesquisa:

Objetivo Primário:

Avaliar a contração de polimerização, por meior de microtomografia computadorizada e a resistência de união, por meio do teste de microtração, em extensas cavidades do tipo classe I com diferentes técnicas de inserção de resinas Bulk fill; flow, flow ativada sonicamente e condensáveis, após envelhecimento “in vitro”. Objetivo Secundário:
Avaliar a resistência de união de resinas bulk fill flow, condensáveis e flow ativada sonicamente em restaurações classe I comparadas a resinas convencionais; Comparar a resistência de união de resinas bulk fill de baixa e média viscosidade;Comparar diferentes técnicas de inserção de resinas bulk fill flow, condensáveis e flow ativada sonicamente em cavidades classe I comparadas a resinas convencionais;Verificar os tipos de
falhas ocorridas na interface dente/restauração. Correlacionar a resitência de união com a presença de bolhas, falhas e gaps na restuaração e com a contração de polimerização.

Avaliação dos Riscos e Benefícios:

Riscos:

Não há risco, pois não serão realizados procedimentos em pacientes. Os dentes utilizados serão coletados de banco de dentes.

Benefícios:

Espera-se com a pesquisa proporcionar a diminuição no tempo clínico durante o atendimento ao paciente que envolvem restaurações semelhantes ao do estudo proposto.

Comentários e Considerações sobre a Pesquisa:

A pesquisa esta bem elaborada com todos os elementos necessários ao seu pleno desenvolvimento.

Considerações sobre os Termos de apresentação obrigatória:

Todos os termos de apresentação obrigatórios foram entregues e estão de acordo com a resolução 466/12 do CNS.

Recomendações:

Não existem recomendações.

Conclusões ou Pendências e Lista de Inadequações:

Não existem pendências.

Considerações Finais a critério do CEP:

Este parecer foi elaborado baseado nos documentos abaixo relacionados:

Tipo Documento	Arquivo	Postagem	Autor	Situação
Informações Básicas do Projeto	PB_INFORMAÇÕES_BÁSICAS_770280_E1.pdf	11/08/2016 22:20:12		Aceito
Outros	PROJETO_PLATAFORMA_BRASIL_BULK_FILL_VERSAO_2.pdf	11/08/2016 22:18:54	Lauber Jose dos	Aceito

			Santos Almeida Junior	
Outros	PROJETO_PLATAFORMA_BRASIL_BULK_FILL_VERSAO_2.docx	11/08/2016 22:18:28	Lauber Jose dos Santos Almeida Junior	Aceito
Outros	CORRECOES_PARCER_PENDENTE_PLATAFORMA_BRASIL.docx	11/08/2016 22:17:46	Lauber Jose dos Santos Almeida Junior	Aceito
Declaração de Instituição e Infraestrutura	CARTA_DE_ACEITE_UNESP.pdf	30/11/2015 12:53:45	Lauber Jose dos Santos Almeida Junior	Aceito
TCLE / Termos de Assentimento / Justificativa de Ausência	TERMO_DE_CONSENTIMENTO_LIVRE_E_ESCLARECIDO.pdf	30/11/2015 12:52:46	Lauber Jose dos Santos Almeida Junior	Aceito
Projeto Detalhado / Brochura Investigador	PROJETO_PLATAFORMA_BRASIL_BULK_FILL.docx	02/09/2015 10:18:01	Lauber Jose dos Santos Almeida Junior	Aceito
TCLE / Termos de Assentimento / Justificativa de Ausência	TERMO_DE_CONSENTIMENTO_LIVRE_E_ESCLARECIDO.docx	02/09/2015 10:17:31	Lauber Jose dos Santos Almeida Junior	Aceito
Folha de Rosto	FOLHA_DE_ROSTO_PLATAFORMA_BRASIL.pdf	25/08/2015 21:14:50	Lauber Jose dos Santos Almeida Junior	Aceito

Situação do Parecer:

Aprovado

Necessita Apreciação da CONEP:

Não

SAO LUIS, 01 de Setembro de 2016

Assinado por:

Richard Diego Leite

(Coordenador)

ANEXO B – Proof de envio à revista The Journal of Adhesive Dentistry

Manuscripts in review

JADD-2017-18 - (3125)

Title: Correlation Between Polymerization Shrinkage and Bond Strength in Class I Restorations With Bulk Fill Composites Using Micro-CT
Type: Original Article
Keywords: polymerization; x-ray microtomography; composite resins; tensile strength
Authors: Lauber Almeida Junior (corresp), Estevam Lula, Karla Penha, Vinicius Correia, Fernando Magalhães, Darlon Martins, Leily Firoozmand

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Review overview

Manuscript ID: JADD-2017-18 - (3125)
Status: In review
Days in review:

ANEXO C – Comprovante de revisão do artigo 1

Print View—654374

Papercheck editors correct grammatical errors that writers of all levels may overlook, including punctuation, verb tense, spelling, and sentence structure. The document body and thesis statement are proofread, ensuring effective communication of the written concept from the writer to the reader. Clients requiring academic editing can request editors to follow any of the standard writing styles: MLA, APA, ASA, AMA, Chicago, CSE, Turabian, or AP.

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- **Editing Style:** Executive
- **English Type:** US English
- **PaperTomb Charge:**
- **SMS Charge:**
- **Non-Standard Style Charge:**
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Final Charges:

ANEXO D - Diretrizes para publicação de trabalhos na The Journal of Adhesive Dentistry

GUIA PARA SUBMISSÃO "The Journal of Adhesive Dentistry".

https://jad.quintessenz.de/jad/downloads/authorguidelines_jad.pdf

The Journal of Adhesive Dentistry GUIDELINES FOR AUTHORS

The Journal of Adhesive Dentistry is a bi-monthly journal that publishes scientifically sound articles of interest to practitioners and researchers in the field of adhesion to hard and soft dental tissues. The Journal publishes several types of peer-reviewed original articles:

1. Clinical and basic science research reports – based on original research in adhesive dentistry and related topics.

2. Reviews topics – on topics related to adhesive dentistry

3. Short communications – of original research in adhesive dentistry and related topics. Max. 4 printed pages, including figures and references (max. characters 18,000). High priority will be given to the review of these papers to speed publication.

4a. Invited focus articles – presenting a position or hypothesis on a basic science or clinical subject of relevant related topics. These articles are not intended for the presentation of original results, and the authors of the articles are selected by the Editorial Board.

4b. Invited commentaries – critiquing a focus article by addressing the strong and weak points of the focus article. These are selected by the Editorial Board in consultation with the focus article author, and the focus article and the commentaries on it are published in sequence in the same issue of the Journal.

5. Invited guest editorials – may periodically be solicited by the Editorial Board.

6. Proceedings of symposia, workshops, or conferences – covering topics of relevance to adhesive dentistry and related topics.

7. Letters to the Editor – may be submitted to the editorin-chief; these should normally be no more than 500 words in length.

SUBMISSION INSTRUCTIONS

Submission of manuscripts in order of preference:

1. Submission via online submission service(www.manuscriptmanager.com/jadd). Manuscript texts should be uploaded as PC-word files with tables and figures preferably embedded within

the PC-word document. A broad range of file formats are acceptable. No paper version required but high resolution photographs or illustrations should be sent to the editorial office (see below). Online submissions are automatically uploaded into the editorial office's reviewer assignment schedule and are therefore processed immediately upon upload.

2. Submission via e-mail as a PC-word document (wintonowycz@quintessenz.de). Illustrations can be attached in any format that can be opened using Adobe Photoshop, (TIF, GIF, JPG, PSD, EPS etc.) or as Microsoft PowerPoint Documents (ppt). No paper version required but high resolution photographs or illustrations should be sent to the editorial office.

3. One paper copy of the manuscript plus a floppy diskette or CD-ROM (mandatory) containing a PC-word file of the manuscript text, tables and legends. Figures should be included on the disk if possible in any format that can to be opened using Adobe Photoshop, (TIf, GIf, JPG, PSD, EPS etc.) or as a Microsoft PowerPoint Document (ppt)

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versions of your tables and illustrations upon resubmission will assure a faster publication time if the manuscript is accepted.

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MANUSCRIPT PREPARATION

- The Journal will follow as much as possible the recommendations of the International Committee of Medical Journal Editors (Vancouver Group) in regard to preparation of manuscripts and authorship (Uniform requirements for manuscripts submitted to biomedical journals. Ann Intern Med 1997;126: 36-47).

- Title page. The first page should include the title of the article (descriptive but as concise as possible) and the name, degrees, job title, professional affiliation, contribution to the paper (e.g., idea, hypothesis, experimental design, performed the experiments in partial fulfillment of requirements for a degree, wrote the manuscript, proofread the manuscript, performed a certain test, consulted on and performed statistical evaluation, contributed substantially to discussion, etc.) and full address of all authors. Phone, fax, and e-mail address must also be provided for the corresponding author, who will be assumed to be the first listed author unless otherwise noted. If the paper was presented before an organized group, the name of the organization, location, and date should be included.

- 3-8 keywords.

- Structured abstract. Include a maximum 250-word structured abstract (with headings Purpose, Materials and Methods, Results, Conclusion).

- Introduction. Summarize the rationale and purpose of the study, giving only pertinent references. Clearly state the working hypothesis.

- Materials and Methods. Present materials and methods in sufficient detail to allow confirmation of the observations. Published methods should be referenced and discussed only briefly, unless modifications have been made. Indicate the statistical methods used, if applicable.

- Results. Present results in a logical sequence in the text, tables, and illustrations. Do not repeat in the text all the data in the tables or illustrations; emphasize only important observations.

- Discussion. Emphasize the new and important aspects of the study and the conclusions that follow from them. Do not repeat in detail data or other material given in the Introduction or Results section. Relate observations to other relevant studies and point out the implications of the findings and their limitations.

- Acknowledgments. Acknowledge persons who have made substantive contributions to the study. Specify grant or other financial support, citing the name of the supporting organization and grant number.
- Abbreviations. The full term for which an abbreviation stands should precede its first use in the text unless it is a standard unit of measurement.
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- Clinical Relevance. Please include a very brief (2 sentences or 3 lines) clinical relevance statement.

REFERENCES

- All references must be cited in the text, according to the alphabetical and numerical reference list.
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Journal reference style:

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Book reference style:

1. Hannam AG, Langenbach GEJ, Peck CC. Computer simulations of jaw biomechanics. In: McNeill C (ed). Science and Practice of Occlusion. Chicago: Quintessence, 1997:187-194.

ILLUSTRATIONS

- All illustrations must be numbered and cited in the text in order of appearance.
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Line drawings–Figures, charts, and graphs should be professionally drawn and lettered large enough to be read after reduction. Good-quality computer-generated laser prints are acceptable (no photocopies); also provide electronic files (eps, ai) if possible. Lines within graphs should be of a single weight unless special emphasis is needed.

Legends–Figure legends should be grouped on a separate sheet and typed double-spaced.

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85

The Journal of Adhesive Dentistry

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ANEXO E – Proof de envio à revista Brazilian Oral Research

Brazilian Oral Research

1 SUBMITTED MANUSCRIPTS

Status	Id	Title	Created	Submitted
ADM: Leitão, Cristina • Awaiting Admin Processing	BOR-2017-0325	Correlation between polymerization shrinkage, gap formation, and void in bulk fill composites using micro-CT View Submission	08-May-2017	08-May-2017

ANEXO F – Comprovante de revisão do artigo 2

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ANEXO G – Diretrizes para publicação de trabalhos na Brazilian Oral Research

Brazilian Oral Research

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INSTRUCTIONS TO AUTHORS

Mission, scope, and submission policy

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BOR invites the submission of original and review manuscripts and papers in the following typology: Original Research (complete manuscript or Short Communication), Critical Review of Literature, Systematic Review (and Meta-Analysis) and Letters to the Editor. All submissions must be exclusive to.

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Presentation of the manuscript

The manuscript text should be written in English and provided in a digital file compatible with "Microsoft Word" (in DOC, DOCX, or RTF format).

All figures (including those in layouts/combinations) must be provided in individual and separate files, according to recommendations described under the specific topic. Photographs, micrographs, and radiographs should be provided in TIFF format, according to the recommendations described under the specific topic.

Charts, drawings, layouts, and other vector illustrations must be provided in a PDF format individually in separate files, according to the recommendations described under the specific topic.

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- Informative and concise title, limited to a maximum of 110 characters, including spaces.
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Main Text

Introduction: This should present the relevance of the study, and its connection with other published works in the same line of research or field, identifying its limitations and possible biases. The objective of the study should be concisely presented at the end of this section.

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- EMBL: http://www.ebi.ac.uk/embl/Submission/index.html
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Results: These should be presented in the same order as the experiment was performed, as described under the "Methodology" section. The most significant results should be described. Text, tables, and figures should not be repetitive. Statistically relevant results should be presented with enclosed corresponding p values.

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Discussion: This must discuss the study results in relation to the work hypothesis and relevant literature. It should describe the similarities and differences of the study in relation to similar studies found in literature, and provide explanations for the possible differences found. It must also identify the study's limitations and make suggestions for future research.

Conclusions: These must be presented in a concise manner and be strictly based on the results obtained in the research. Detailing of results, including numerical values, etc., must not be repeated.

Acknowledgments: Contributions by colleagues (technical assistance, critical comments, etc.) must be given, and any bond between authors and companies must be revealed. This section must describe the research funding source(s), including the corresponding process numbers.

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- Results
- Discussion
- Conclusion
- Acknowledgments
- Tables
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- Results
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- Conclusion
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- References: a maximum of 12 references
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- Results
- Discussion
- Conclusion
- Acknowledgments
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- Review update
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EXAMPLES OF REFERENCES

Journals

Goracci C, Tavares AU, Fabianelli A, Monticelli F, Raffaelli O, Cardoso PC, et al. The adhesion between fiber posts and root canal walls: comparison between microtensile and push-out bond strength measurements. Eur J Oral Sci. 2004 Aug;112(4):353-61.

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Usunoff KG, Itzev DE, Rolfs A, Schmitt O, Wree A. Nitric oxide synthase-containing neurons in the amygdaloid nuclear complex of the rat. Anat Embryol (Berl). 2006 Oct 27. Epub ahead of print. doi: 10.1007/s00429-006-0134-9

Walsh B, Steiner A, Pickering RM, Ward-Basu J. Economic evaluation of nurse led intermediate care versus standard care for post-acute medical patients: cost minimisation analysis of data from a randomised controlled trial. BMJ. 2005 Mar 26;330(7493):699. Epub 2005 Mar 9.

Papers with Title and Text in Languages Other Than English

Li YJ, He X, Liu LN, Lan YY, Wang AM, Wang YL. [Studies on chemical constituents in herb of Polygonum orientale]. Zhongguo Ahong Yao Za Zhi. 2005 Mar;30(6):444-6. Chinese.

Supplements or Special Editions

Pucca Junior GA, Lucena EHG, Cawahisa PT. Financing national policy on oral health in Brazil in the context of the Unified Health System. Braz Oral Res. 2010 Aug;24 Spec Iss 1:26-32.

Online Journals

Barata RB, Ribeiro MCSA, De Sordi M. Desigualdades sociais e homicídios na cidade de São Paulo, 1998. Rev Bras Epidemiol. 2008;11(1):3-13 [cited 2008 Feb 23]. Available from: http://www.scielosp.org/pdf/rbepid/v11n1/01.pdf.

Books

Stedman TL. Stedman's medical dictionary: a vocabulary of medicine and its allied sciences, with pronunciations and derivations. 20th ed. Baltimore: Williams & Wilkins; 1961. 259 p.

Books Online

Foley KM, Gelband H, editors. Improving palliative care for cancer [monograph on the Internet]. Washington: National Academy Press; 2001 [cited 2002 Jul 9]. Available from: http://www.nap.edu/books/0309074029/html/.

Websites

Cancer-Pain.org [homepage on the Internet]. New York: Association of Cancer Online Resources, Inc.; c2000 [cited 2002 Jul 9]. Available from: http://www.cancer-pain.org/.

Instituto Brasileiro de Geografia e Estatística [homepage]. Brasília (DF): Instituto Brasileiro de Geografia e Estatística; 2010 [cited 2010 Nov 27]. Available from: http://www.ibge.gov.br/home/default.php.

World Health Organization [homepage]. Geneva: World Health Organization; 2011 [cited 2011 Jan 17]. Available from: http://www.who.int/en/