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Maria Tereza Hordones Ribeiro

Efeito do pré-aquecimento realizado com Caps warmer e Dispenser nas propriedades mecânicas, contração pós-gel e tensão de contração de resinas compostas bulk fill

Effect of preheating performed with caps warmer and dispenser on mechanical properties, post-gel shrinkage and shrinkage stress of bulk fill materials

Dissertação apresentada à Faculdade de Odontologia da Universidade Federal de Uberlândia como requisito parcial para obtenção do título deMestre em Odontologia na Área de Clínica Odontológica Integrada.

Uberlândia, 2021

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Orientador: Profº. Drº. Carlos José Soares

Banca examinadora:
Profº. Drº. Carlos José Soares
Profº. Drº. Paulo César de Freitas Santos Filho
Profº. Drº. Fabrício Mezzomo Collares

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Carlos José Soares Professor(a) do Magistério Superior

Paulo César de Freitas Santos Filho Professor(a) do Magistério Superior

Fabricio Mezzomo Collares Usuário Externo

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SUMÁRIO

RESUMO

Avaliar o efeito do pré-aquecimento de diferentes resinas compostas bulk fill em diferentes temperaturas e métodos de aquecimento nas propriedades mecânicas expressas por resistência à flexão, módulo de elasticidade, grau de conversão, dureza Knoop, contração pós-gel e tensão de contração. Três resinas compostas bulk fill de consistência regular disponíveis em cápsulas (FO - Filtek One Bulk Fill, 3M-ESPE; VC - VisCalor Bulk, VOCO; XF - x-tra Fil, VOCO) foram pré-aquecidas usando VisCalor Dispenser (VOCO) a 65ºC e Caps Warmer (VOCO) a 37ºC, 54ºC e 68ºC. Todos os materiais foram ativados por luz usando uma LCU multi espectos (VALO Cordless, Ultradent) por 20 segundos. A contração pós-gel (Shr, %) foi calculada usando um extensômetro. A resistência à flexão (FS, MPa), módulo de elasticidade (E, MPa) foram calculados pelo teste de flexão de 3 pontos. O grau de conversão (DC, %) usando FTIR e dureza Knoop (KNH, N / mm^2) foram testados nas superfícies superiores. A resistência à tração diametral (DTS) e a resistência à compressão (CS) foram determinadas (n = 10). As tensões de contração foram analisadas usando a análise tridimensional de elementos finitos. Os dados foram analisados por ANOVA de dois fatores e testes de Tukey (α = 0,05). Os diferentes processos de aquecimento e temperaturas não influenciaram nas propriedades mecânicas testadas. Os valores de FS não foram influenciados pelos métodos de aquecimento, temperatura e tipo de resina composta testada. FO apresentou os menores valores de E, DC e KNH, VC apresentou valores intermediários e XF apresentou os maiores valores. Os valores de DTS e CS não foram influenciados pelos métodos de aquecimento, temperaturas e tipo de resina composta testada. O processo de aquecimento a 65ºC realizado com um dispensador e a 68ºC e 54ºC realizado com tampas mais quentes resultou em menos tensão de encolhimento do que o pré-aquecimento a 37ºC usando tampas mais quentes, independentemente do composto de resina bulk fill. O método VisCalor possui as tensões mais baixas. O novo dispensador de aquecimento testado demonstrou eficiência na produção de aquecimento de resinas compostas, sem influenciar as propriedades mecânicas. O aquecimento de compósitos de resina de enchimento viscosos a granel em temperaturas mais elevadas (54ºC, 65ºC e 68ºC) reduziu a tensão de retração em comparação ao aquecimento a 37ºC.

PALAVRAS-CHAVE: pré-aquecimento, resinas compostas, grau de conversão, dureza, resistência flexural, contração pós-gel, tensão de contração.

ABSTRACT

To evaluate the effect of the preheating of different bulk fill composites at different temperatures and heating methods on the mechanical properties expressed by flexural strength, modulus of elasticity, degree of conversion, Knoop hardness, post-gel contraction and contraction stress. Three bulk fill composite resins of regular consistency available in capsules (FO - Filtek One Bulk Fill, 3M-ESPE; VC - VisCalor Bulk, VOCO; XF - x-tra Fil, VOCO) were preheated using VisCalor Dispenser (VOCO) a 65ºC and Caps Warmer (VOCO) at 37ºC, 54ºC and 68ºC. All materials were activated by light using a multipeak LCU (VALO Cordless, Ultradent) for 20 seconds. Post-gel contraction (Shr, %) was calculated using an extensometer. Flexural strength (FS, MPa), modulus of elasticity (E, MPa) were calculated by the 3-point flexion test. The degree of conversion (DC, %) using FTIR and Knoop hardness (KNH, N / mm^2) were tested on the upper surfaces. The diametrical tensile strength (DTS) and the compressive strength (CS) were determined (n = 10). The contraction stresses were analyzed using three-dimensional finite element analysis. The data were analyzed by two-way ANOVA and Tukey's tests (α = 0.05). The different heating processes and temperatures did not influence the tested mechanical properties. The FS values were not influenced by the heating methods, temperature and type of composite resin tested. FO presented the lowest values of E, DC and KNH, VC presented intermediate values and XF presented the highest values. The DTS and CS values were not influenced by the heating methods, temperatures and type of composite resin tested. The heating process at 65oC performed with a dispenser and at 68ºC and 54ºC performed with warmer covers resulted in less shrinkage tension than preheating at 37ºC using warmer covers, regardless of the bulk-fill resin compound. The VisCalor method has the lowest stresses. The new tested heating dispenser demonstrated efficiency in producing heating of composite resins, without influencing the mechanical properties. The heating of bulk viscous filler resin composites at higher temperatures (54 ºC, 65ºC and 68ºC) reduced the shrinkage tension compared to heating at 37ºC.

KEYWORDS: preheating, resin composite, degree of conversion, microhardness, flexural strength, post-gel shrinkage, shrinkage stress.

1. INTRODUÇÃO E REFERÊNCIAL TEÓRICO

As restaurações de resina composta são amplamente utilizadas como materiais restauradores diretos em odontologia, pois apresentam bom desempenho clínico e longevidade comprovada. (da Rosa Rodolpho *et al.*, 2006; Heintze *et al*., 2012) Na última década, as resinas compostas *bulk fill* chegaram ao mercado mundial como um novo conceito restaurador. (Ilie *et al.*, 2014; Al Sunbul *et al.*, 2016; Engelhardt *et al.*, 2016) Como principal vantagem, a técnica permite que incrementos de até 4-5 mm sejam inseridos na cavidade. (Oliveira *et al.,* 2016) Além disso, estudos laboratoriais mostram desempenho semelhante ou melhor de resinas compostas *bulk fill* em comparação com resinas compostas convencionais em termos de tensão de contração, deflexão de cúspide, fraturas marginais, grau de conversão, resistência à flexão e à fratura. (Cidreira *et al.*, 2019) As resinas compostas *bulk fill* têm características otimizadas e estão ganhando popularidade para restaurar dentes posteriores. (Abdulrazzaq *et al.*, 2015)

As resinas compostas *bulk fill* de consistência regular por serem inseridos em grandes incrementos podem gerar bolhas no momento da inserção e comprometer a adaptação do material à cavidade. A adaptabilidade também pode interferir no selamento marginal, fator essencial para a longevidade de restauração em dente posterior. O comprometimento da adaptação marginal da restauração pode levar ao desenvolvimento de cáries secundárias, resultando em falha da restauração e comprometimento da saúde pulpar. (Demarco *et al*., 2012) O pré-aquecimento de resinas compostas antes da inserção tem sido utilizado para melhorar a adaptação ao preparo cavitário, uma vez que o aquecimento reduz a viscosidade permitindo melhor acomodação. (Alshali *et al.*, 2015)

Recentemente, estudos mostraram que o pré-aquecimento de resinas compostas *bulk fill* pode melhorar as propriedades mecânicas, como aumentar o grau de conversão e reduzir a tensão de contração, (Tauböck *et al.*, 2015) melhorar a microdureza, (Dionysopoulos *et al.*, 2016) e pode até aumentar a resistência à fratura. (Abdulmajeed *et al.*, 2020) O pré-aquecimento também torna a resina composta *bulk fill* pasta mais fluida e mais fácil de manipular, sem

comprometer suas propriedades mecânicas superiores quando comparado com o material fluido. (Alshali *et al.*, 2015; Metalwala *et al.*, 2018)

Atualmente, existe no mercado dispositivos desenvolvidos para aquecer cápsulas de resina composta, como Caps Warmer, que operam na faixa de temperatura entre 37-68 ºC. (Daronch *et al.*, 2007; Nada *et al.*, 2011; Deb *et al.*, 2011; Tauböck *et al.*, 2015; Yang *et al.*, 2019) Para agilizar o procedimento restaurador, o novo Dispenser de pré-aquecimento (VOCO, Alemanha) foi criado para aquecer cápsulas de resina composta a temperatura de 64 ºC em menor período de tempo. O dispositivo Dispenser é capaz de aquecer as novas cápsulas VisCalor® em 30 segundos e outras cápsulas de resina composta em 70 segundos, dando ao operador tempo para trabalhar enquanto mantém as cápsulas aquecidas. Com a chegada de diferentes dispositivos no mercado, a necessidade de estudos que avaliem o desempenho do aquecimento de resina composta se torna oportuno.

Portanto, o objetivo deste estudo foi avaliar o efeito do pré-aquecimento de resinas compostas bulk fill de consistência regular em diferentes temperaturas e métodos de aquecimento nas propriedades mecânicasexpressas por resistência à flexão, módulo de elasticidade, dureza Knoop, retração pós-gel e tensão de retração.

2. CAPÍTULO 1

ARTIGO 1

Effect of preheating performed with caps warms and dispenser on mechanical properties, post-gel shrinkage and shrinkage stress of bulk fill resin composites

***Artigo a ser enviado para o periódico Dental Materials**

Effect of preheating performed with caps warmer and dispenser on mechanical properties, post-gel shrinkage and shrinkage stress of bulk fill materials

Maria Tereza Hordones Ribeiro[a], Gabriel Felipe Bragança[b], Laís Rani Sales Oliveira[c], Stella Sueli Lourenço Braga[d], Helena Letícia Quirino de Oliveira[e], Richard Bengt Price[f], Carlos José Soares[g]

[a] DDS, MSc student, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Uberlândia, Minas Gerais, Brazil. Email: mt_hribeiro@hotmail.com

[b] DDS, MSc, PhD student, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Uberlândia, Minas Gerais, Brazil. Email: gabriel.braganca@hotmail.com

[c] DDS, MSc, PhD student, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Uberlândia, Minas Gerais, Brazil. Email: laisrani@hotmail.com

[d] DDS, MSc, PhD student, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Uberlândia, Minas Gerais, Brazil. Email: stellabraga2@hotmail.com

[e] DDS, MSc student, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Uberlândia, Minas Gerais, Brazil. Email: helena_leticia@hotmail.com

[f] DDS, MSc, PhD, Professor Department of Dental Clinical Sciences, Dalhousie University, Halifax, Nova Scotia, Canada. Email: rbprice@dal.ca

[g] DDS, MSc, PhD, Professor and chair, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Uberlândia, Minas Gerais, Brazil. Email: carlosjsoares@ufu.br

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Corresponding author:

Dr. Carlos José Soares

Federal University of Uberlândia, School of Dentistry

Avenida Pará, 1720, Bloco 4L, Anexo A, Sala 42, Campus Umuarama

Uberlândia - Minas Gerais – Brazil - CEP 38400-902

Phone: +55 34 3225 8119 / Cell Phone: +55 34 999713472, E-mail: carlosjsoares@ufu.br

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Highlights

1. The mechanical properties of the resin composites are not affected by heating procedures;
2. Heating of bulk fill resin composites promote decrease in the shrinkage stress;
3. Dispenser can offer shorter clinical working time and heat resin composites efficiently;
4. A new preheating resin composite, VisCalor, showed a good performance on the tested mechanical properties and shrinkage stress.

Effect of preheating performed with caps warmer and dispenser on mechanical properties, post-gel shrinkage and shrinkage stress of bulk fill materials

Abstract

Objectives. To evaluate the effect of preheating different bulk fill resin composites of different temperatures and heating methods on the mechanical properties expressed by flexural strength, elastic modulus, Knoop hardness, post-gel shrinkage, and shrinkage stress.

Methods. Three bulk-fill resin composites of regular viscosity available in capsules (FO - Filtek One Bulk Fill, 3M-ESPE; VC - VisCalor Bulk, VOCO; XF - x-tra Fil, VOCO) were preheated using VisCalor Dispenser (VOCO) at 65ºC and Caps Warmer (VOCO) at 37ºC, 54ºC and 68ºC. All materials were light activated using a multi-peak LCU (VALO Cordless, Ultradent) for 20 seconds. Post-gel shrinkage (Shr, %) was calculated using strain-gauge. Flexural strength (FS, MPa), elastic modulus (E, MPa) were calculated by 3-bending flexural test. Degree of conversion (DC, %) using FTIR and Knoop hardness (KNH, N/mm^2) were tested on top surfaces. Diametral tensile strength (DTS) and compressive strength (CS) were determined (n=10). Shrinkage stress were analized using three-dimensional finite element analysis. The data was analyzed using Two-way ANOVA and Tukey's tests ($\alpha = 0.05$).

Results. The different heating processes have not influenced on tested mechanical properties. The FS values were not influenced by heating methods, temperatures and type of the resin composites tested. FO presented the lowest E, DC and KNH values, VC showed intermediate values and XF exhibited the highest values. The DTS and CS values were not influenced by heating methods, temperatures and type of resins composite tested. The heating process at 65ºC performed with a dispenser and at 68ºC and 54ºC performed with caps warmer resulted in less shrinkage stress than the preheating at 37ºC using caps warmer, regardless of the bulk-fill resin composite. VisCalor method present the lowest stresses.

Significance. The new heating dispenser tested demonstrated efficiency to produce heating of resin composites, without influencing the mechanical properties. Heating bulk

fill resin composites of regular viscosity at higher temperatures (54 °C, 65 °C and 68 °C) reduced the shrinkage stress when compared to heating at 37 °C.

Keywords: preheating, resin composite, degree of conversion, microhardness, flexural strength, post-gel shrinkage, shrinkage stress.

INTRODUCTION

Resin composite restorations are widely used as direct restorative materials in dentistry, as they have good clinical performance and proven longevity. [1,2] In the last decade, bulk fill resin composites arrived on the market bringing a new restorative concept. [3, 4, 5] As a main advantage, the technique allows increments up to 4-5mm. [6] They also can be classified according to consistency in flowable (low viscosity) or high viscosity resin composites. [7] In addition, laboratory studies have shown similar or better performance of bulk fill resin composites compared to traditional resin composites in terms of shrinkage stress, cusp deflection, marginal crack, degree of conversion, flexural and fracture resistances. [8, 9, 10] Bulk fill resin composites have optimized characteristics and are gaining popularity for restoring posterior teeth. [11]

The bulk fill resin composites of regular consistency for being used in large increments can generate bubbles at the time of insertion, compromising the adaptation of the material to the cavity. [12] The adaptability can also interfere on the marginal sealing, that is an essential factor for the longevity of a posterior restoration. [13] The impairment of the marginal adaptation of a restoration can lead to the development of secondary caries, resulting in failure of the restorative procedure and compromised pulp health. [14] The preheating of resin composites before insertion into the cavity has been used to improve the adaptation to the cavity preparation, since the heating reduces the fluidity allowing better accommodation. [13, 15] Furthermore, preheating also makes the regular consistency bulk fill resin composite more fluid and easier to manipulate, without compromising its mechanical properties when compared to a flowable material. [15, 16] Recent studies have shown that preheating bulk-fill resin composites can improve mechanical properties, such as the degree of conversion and reducing the shrinkage stress, [17] despite improving microhardness [18], and may even result on superior fracture resistance. [20]

Some devices available on the market intended to heat resin composite capsules, such as caps warmers, which have shown operational ranges at a temperature of 37-68^{o}C. [17, 21-24] To speed up the restorative procedure, a new preheating dispenser (VOCO, Germany) was created in order to heat resin composite capsules to a

temperature of 65°C in a shorter period of time. The heating dispenser device is able to heat the new VisCalor® composite capsules in 30 seconds and other resin composite capsules in 70 seconds, giving the operator time to work while keeping the capsules warmed. On the best of the authors' knowledge, there is no study evaluating the effect of different heating methods including the new heating dispenser mentioned, on the mechanical properties of regular viscosity bulk fill resin composites and also on the shrinkage stress reduction.

Therefore, the aim of this study was to evaluate the effect of preheating different regular viscosity bulk fill resin composites at distinct temperatures and heating methods on the mechanical properties expressed by flexural strength, elastic modulus, Knoop hardness, post-gel shrinkage, diametral tensile strength, compressive strength and shrinkage stress. The null hypotheses of this study were: 1) the preheating performed with caps warmer and dispenser would not influence the mechanical properties of different regular viscosity bulk fill resin composites; 2) the post-gel shrinkage and shrinkage stress would not influenced by bulk fill resin composite types and preheating methods.

MATERIALS AND METHODS

Study design

Three regular viscosity bulk-fill resin composites (FO - Filtek One Bulk Fill, 3M-ESPE; VC - VisCalor Bulk, VOCO; XF - X-Tra Fil/Caps, VOCO) were tested in this study. The information of the tested resin composites is summarized in Table 1. Two devices were used to preheat the resin composites: heat dispenser (VisCalor Dispenser, VOCO) and a capsule warmer with multiple temperatures (Caps Warmer, VOCO). The materials were preheated as follows: Heating Dispenser at 65°C; Caps Warmer at 37 °C, 54 °C and 68 °C. All materials were light activated using a multi-peak light-curing unit (LCU) (VALO Cordless; Ultradent, South Jordan, UT, USA) for 20 seconds. Post-gel shrinkage (Shr) was calculated using strain-gauge test. Flexural strength (FS, MPa), elastic modulus (E, MPa) were calculated by 3-bending flexural test. Degree of conversion (DC, %) was calculated using FTIR and Knoop hardness (KNH, N/mm^2) were tested on top surfaces of the

specimens. Shrinkage stress was calculated by using a 3D finite element analysis of a Class II resin composite restoration in a molar tooth.

Post-gel Shrinkage (Shr)

Post-gel shrinkage was determined using the strain-gauge. [25] For each group 10 specimens were tested. Immediately after preheating the material with different methods the resin composite was inserted into a mold (1.5 mm x 2.0 mm x 2 mm) on top of a biaxial strain gauge (CEA-06-032WT-120, Excel Sensores, Taboão da Serra, SP, Brazil), which measured the shrinkage strains in two directions (perpendicular and parallel). A strain conditioner (ADS0500IPg, Lynx, São Paulo, SP, Brazil) converts changes in electrical resistance on the strain gauge into changes in voltage through a quarter-bridge circuit with an internal reference resistance. The strain values measured along the two axes were calculated, since the material properties were homogeneous and isotropic on a macro scale. All materials were inserted by the same operator and activated by light with a multi-peak LCU (VALO Cordless, Ultradent, South Jordan, UT, USA) with 1297.9 mW/cm^2 checked on MARC resin calibrator (Blue-Light Analytics, Halifax, NS, Canada). All tests were performed in a dark room with yellow light. Deformation values were collected for ten minutes after the light activation to monitor the real-time measurement of the shrinkage strain. The average shrinkage strain was expressed in %, which represented the linear shrinkage, was converted into a volumetric percentage multiplying by 3 and 100%. [9]

Flexural Strength (FS) and Young's Elastic Modulus (E)

The flexural strength of all groups was determined by a three-bending flexural test performed in accordance with ISO 4049 Standard [26]. According to the methodology described above, ten specimens made for each group were tested after being stored for 24h in a dry oven at 37^{o}C.

The specimens (n = 10) were prepared in stainless steel molds (25 x 2 x 2 mm) in a dark room with yellow light. To minimize the presence of bubbles and obtain a smooth surface, the mold was placed on a glass plate and a polyester strip was positioned between the glass plate and the mold. Each material was kept for 15 minutes when

heated in the Caps Warmer. When heated in the dispenser, VC was kept for 30 seconds and the other resin composites for 70 seconds. Then, the resin composite was inserted into the mold using a condenser for better adapt to the material, then another polyester strip was placed and a second glass plate was used to press the material in order to force the excess resin composite out. The maximum time between the removal of the material in the heating unit and the photoactivation was 40s. [13] The specimens were light activated for 20 seconds in 3 light exposition covering the entire sample extension whit a multi-peak LCU (VALO Cordless).

The test was performed using the three-point bending set-up using a universal testing machine (Instron ElectroPulsTM E3000, Instron, High Wycombe, UK) and assembled using the software (Bluehill Universal, Instron Training Center, Norwood, MA, USA) with a crosshead speed of 0.5 mm / min. Flexural strength was determined according to the following formula:

$$\alpha = 3FL/2wt;$$

where F is the maximum force applied (N); L is the distance between the support beams (mm); *w* is the width of the specimen (mm); and *t* is the thickness of the specimen (mm). The modulus of elasticity (MPa) was determined using flexural deflectometer (W-E401-J, E-Series Deflectometer, Instron, Norwood, MA, USA). The deflectometer tip was positioned at the center of sample base during the test to measure the deflexion during loading application. The modulus of elasticity (MPa) was determined to the following formula:

$$E = FL^3/ 4BH^3d$$

where F is the maximum load (N); L is the length of the specimen (mm); B is the width of the specimen (mm); H is the height of the specimen (mm) and d is the deflection (mm) corresponding to the load F. [27]

Degree of conversion (DC)

The degree of conversion (DC) was assessed within 24 hours. They were stored dry at 37°C protected from light. DC as evaluated using Fourier transform infrared spectroscopy (FTIR, Vertex 70, Bruker Optik GmbH, Ettlingen, Germany) with attenuated total reflectance, medium infrared (MIR) sampling and deuterated triglycine sulfate (Bruker Optics) detecting elements. The spectra were obtained between the stretching

vibrations of the C = C bonds of aromatic internal pattern (1608 cm^{-1}) and the stretching vibrations of the C = C aliphatic bonds (1638 cm^{-1}), with a resolution of 4 cm^{-1} and average of 32 scans. All analyzes were performed under controlled conditions of temperature (25 ± 1°C) and humidity (60 ± 5%). The DC was calculated from the aliphatic (1638 cm^{-1}) and aromatic (1608 cm^{-1}) proportions of the cured (C) and uncured (U) resin composite. FO resin composite does not contain Bis-GMA or Bis-EMA, so the 1450 cm^{-1} peak was used as an alternative internal standard. [28] The formula used to calculate the degree of conversion was:

$$DC\ (\%) = (1\text{-}C / U) \times 100.$$

Knoop Hardness (KNH)

After measuring the degree of conversion, specimens from each group were used for KNH analysis of bulk fill resin composites on the upper surfaces. The surfaces were polished with metallographic diamond pastes 6, 3, 1 and 0.25 µm (Arotec, São Paulo, SP, Brazil). [29] The Knoop indentation values were determined with a microhardness tester (FM700; FutureTech Corp., Kawasaki, Japan) applying a load of 50 g for 15 s. Five indentations were made in the middle of each surface with an interval of 1 mm between them to obtain an average value.

Diametral tensile strength (DST) and Compressive strength (CS)

Diametral tensile strength and compressive strength tests (n = 10) of each resin composite were performed. The resin composite was placed into a cylindrical Teflon mold for the compressive strength test (6 mm height, 3 mm diameter) or the diametral tensile strength test (2 mm height, 4 mm diameter). The specimens for the compressive test made with bulk fill resin composites were polymerized with 4.0 mm for the first increment and 2.0 mm for second increment. Afterwards, the specimens were stored in distilled water for 24 h at 37 °C. The specimens were submitted to compressive strength and diametral tensile testing in a universal testing machine (DL2000, EMIC, São José dos Pinhais, PR, Brazil) at a crosshead speed of 0.5 mm/min until failure occurred. Compressive strength values (MPa) were calculated by dividing the fracture load (F) by the cross-sectional area and converted into MPa. Diametral tensile strength values (MPa) were calculated using the equation:

$$DTS = 2F/¶dt,$$

where d is the specimen diameter, and t is the height of the specimen.

Three-Dimensional Finite Element Analysis (3D-FEA)

The 3D-FEA was performed simulating a 4 mm depth MOD cavity preparation and the bulk fill technique used in this *in vitro* study. The 3D model was created from an intact and healthy human second molar using micro-CT (Skyscan 1272, Bruker, Belgium) and an interactive medical imaging software (Mimics 16.0; Materialize, Leuven, Belgium). The segmentation of dental structures and restorative materials was performed based on the image density threshold. All simulated structures were included in the solid model. Each mask resulting from dental structures and restorative materials was then converted into a 3D file (triangulated stereolithography [STL], bilinear and interplanar interpolation algorithm). An advanced STL design and mesh software (3-Matic 8.0, Materialize) was used to prepare the tooth sample as an STL file. After processing, the different parts were merged into a single STL file called an assembly. The final assembly was then redone using the 3-matic REMESH option.

The STL models were imported into MSC.Patran 2010r2 (MSC Software, Santa Ana, CA, USA) and combined with tetrahedral elements, which is element number 134 in the finite element software (MSC.Marc / Mentat, MSC Software). Therefore, the volumetric meshes of all model components were generated based on the optimized STL surfaces. [30] The volumetric mesh was imported into an FEA software package (MSC.Marc / MSC.Mentat). The properties applied to dental structures, such as the values of the elastic modulus and the Poisson's ratio, were taken from the literature (Table 2). [31]

Structural analysis was performed and all materials were considered linear, isotropic and homogeneous (Tables 3). [32-36] The Poisson’s ratio was chosen to be the same for all resin composites at 0.24. [6] Stress distributions were analyzed using modified von Mises equivalent stresses. The equivalent stress used was based on the well-known von Mises formulation, modified to consider the difference between compressive strength and tensile strength for the materials calculated experimentally (Table 4). [32,33]

Statistical Analysis

The post-gel shrinkage, flexural strength, elastic modulus, degree of conversion, Knoop hardness, diametral tensile strength and compressive strength data were tested for normal distribution (Shapiro–Wilk) and equality of variances (Levene's test), followed by parametric statistical tests. Two-way analysis of variance (ANOVA – 3 x 4) was performed for each mechanical property considering 3 resin composites and 4 heating processes. Multiple comparisons were made using Tukey's test. Pearson correlation was calculated for all mechanical properties calculated. All tests employed $\alpha = 0.05$ significance level and all analyses were carried out with the statistical package (Sigma Plot version 13.1, Systat Software Inc, San Jose, CA, USA). The modified von Mises stresses values were analyzed qualitatively.

RESULTS

The post-gel shrinkage values and standard deviations of 3 resin composites and 4 heating processes are shown in Figure 1. Two-way ANOVA showed no significant differences for the resin composites ($P = 0.733$), for the interaction between resin composites and heating processes ($P = 0.569$), however significant differences was found for heating processes ($P < 0.001$). The heating process at 65°C performed with Dispenser and at 68°C and 54°C using caps warmer resulted in lower Shr values than 37°C caps warmer heating process, irrespective of bulk fill resin composites (Fig. 1).

The mean flexural strength values and standard deviations for the 3 resin composites and 4 heating processes are shown in Table 5. Two-way ANOVA showed no significant differences between the resin composites ($P = 0.116$), between the heating processes ($P = 0.564$), and the interaction between resin composites and heating processes factors ($P = 0.200$).

The mean elastic modulus values and standard deviations for the 3 resin composites and 4 heating processes are shown in Table 5. Two-way ANOVA showed significant difference between the resin composites ($P < 0.001$), however no significant differences between the heating process ($P = 284$) and the interaction between resin composites and heating processes factors were detected ($P = 992$). Tukey's test showed that FO had the lowest E values, the VC had intermediate values and XF had the highest values.

The mean degree of conversion values and standard deviations for the 3 resin composites and 4 heating processes are shown in Table 6. Two-way ANOVA showed significant differences between the resin composites ($P < 0.001$); however no significant differences between the heating processes ($P = 0.994$) and the interaction between resin composites and heating processes factors were detected (P = 0134). Tukey's test showed that FO had the lowest DC values, the VC had intermediate values and XF had the highest values.

The mean Knoop hardness values and standard deviations for the 3 resin composites and 4 heating processes are shown in Table 6. Two-way ANOVA showed significant differences between the resin composites ($P < 0.001$); however no significant differences between the heating processes ($P = 0.307$) and the interaction between resin composites and heating processes factors were detected (P = 0.087). Tukey's test showed that FO had the lowest KHN values, the VC had intermediate values and XF had the highest values.

The mean compressive strength, diametral tensile strength values, the standard deviations and the stress differential effect for the 3 resin composites and 4 heating processes are shown in Table 4. Two-way ANOVA showed no significant differences between the resin composites (P = 0.967), between the heating processes (P = 0.878) and the interaction between resin composites and heating processes (P = 0.769) for compressive strength data. For diametral tensile strength data, two-way ANOVA also showed no significant differences between the resin composites (P = 0.986), between heating processes ($P = 0.955$) and the interaction between resin composites and heating processes (P = 0.889) for diametral tensile strength data. The stress differential effect values demonstrated that the resin composites inserted using different heating methods presented between 2.9 at 4.2 more resistant for compression than tension stress.

Correlation plots between mechanical properties of resin composites preheated with different methods are shown in Fig. 2. Degree of conversion had higher direct correlation with elastic modulus (R=0.80) and Knoop hardness (R=0.78). The elastic modulus had also higher direct correlation with Knoop hardness (R=0.73). Lower correlation was found between flexural strength and Knoop hardness (R=0.08), elastic modulus (R=0.01), and degree conversion (R=0.10).

Shrinkage stress distributions during restoration (modified von Mises stress) are shown in Figs. 3, 4 and 5. The buccal cusps (Fig. 3) demonstrated higher stresses than lingual cusps (Fig. 4), irrespective of the bulk fill resin composite and heating process used. The heating process at 65°C performed with dispenser and 68 °C and 54 °C caps warmer resulted in lower stress than 37°C caps warmer heating process, irrespective of bulk fill resin composite (Figs. 3, 4 and 5). The XF had higher modified von Mises stresses in the both cusps and intaglio cavity surface than FO (Fig. 5). VC showed the lowest modified von Mises stresses.

DISCUSSION

This study evaluated whether different heating processes and temperatures would impair the mechanical properties of regular viscosity bulk fill resin composites. The new preheating dispenser performed similarly to the caps warmer with no influence on the tested mechanical properties; therefore, the first null hypothesis was accepted. However, the resin composite type demonstrated significant effect on E, DC, KHN, Shr, DTS, CS values, less for FS values. The heating process at 65°C performed with Dispenser and also at 68°C and 54°C using caps warmer, resulted in less post-gel shrinkage and shrinkage stress than preheating at 37°C using caps warmer, irrespective of the tested resin composite; therefore, the second hypothesis was rejected.

One of the main causes of failures on dental restorations is related to fractures. [37] The resin composite clinical longevity may be influenced by patients, different materials and technical factors. [38] The mechanical properties of resin composites, such as the ones tested in the present investigation (E, DC, KHN, Shr, DTS, CS and FS) cannot predict the clinical performance of these materials, mainly when they are severely affected by clinical procedures. [9] Despite, testing dental materials in a laboratory represents the ideal condition and may have imitated clinically relevance, but provides valid results for ideally characterize the materials. [39] For better understanding the clinical effect of procedures, such as heating process on resin composites, the association of different methodologies can be helpful to explain and orientate about the real effectivity of this approach. [6, 7, 20, 39]

Flexural strength is an important mechanical property for brittle materials. [39, 40] When the resin composite is subjected to masticatory forces, it must offer sufficient

flexural strength to properly resist to fracture. According to ISO 4049-2009, resin composites evaluated with this standard methodology must present a minimum flexural strength value of 80 MPa. [26] Therefore, all resin composites used with different heating processes tested in this study showed an adequate flexural strength. This mechanical property was not affected by the preheating procedures of different temperatures. Previous studies have shown similar results, in which they concluded that preheating had no effect on the flexural strength of resin composites. [20, 40] The high light energy delivered during the 3 light curing activation cycles used in this study may have eliminated eventual differences between some of the studied factors, mainly the ones related to the resin composites that had effect on other mechanical properties. These results also show the great importance of the light activation protocol used for posterior restorations, since the use of light sources covering all the restoration area can minimize inadequate polymerization and its negative effects on the mechanical properties. [41]

The elastic modulus and Knoop hardness are mechanical properties that can be associated with a better performance of resin composites used for posterior restorations. [9] The hardness reflects the ability of the material to withstand forces from chewing, while the modulus of elasticity is associated with the dissipation of stresses through dental tissues. [34] The results of this study show that the heating process had no interference on these properties. Actually, previous investigations showed that the pre-heating of some resin composites can improve the hardness of these materials. [42, 43] This study confirms that bulk fill resin composites with regular consistency have satisfactory elasticity modulus correlate with good hardness, and great elastic modulus also showed high direct correlation with Knoop hardness. These associated results can be related to good fracture resistance of resin composites. [7]

The filler content, the filler size and also the monomer integration with fillers are directly related to the physical and mechanical properties of the resin composites. [34] Studies have studies have shown that the filler volume and the filler load level of resin composites has a direct relationship with the modulus of elasticity. [44-48] Despite of no significant differences between the heating processes was observed, the present study showed that the resin composites tested showed significant differences on E and KHN values. The resin composite with higher filler volume exhibited the greatest flexural

strength and elastic modulus. The FO has the lowest filler content followed by VC and XT, which was the same sequence observed for the E and KHN values; FO had the lowest values, followed by VC and the by XF, with the highest E, KHN and DC values. This is explained by the filling content, since the resin composite with higher filler content resulted in a less deformable solid when gaining the material becomes rigid. [49]

Shrinkage stress can lead to cusp deformation after the resin composite polymerization process. [50] As a clinical consequence of the shrinkage stress, the appearance of enamel cracks, postoperative sensitivity and marginal debonding, that contributes to marginal staining, has been erroneously used as a criterion for replacement of direct and indirect composite restorations. [51] The deformation generated during the post-gel phase of the resin composite can be measured using the strain-gauge method. [52, 53] The filler volume increasing generally leads to a reduction in the volumetric relaxation during the polymerization. [34] The XF resin composite is a regular viscosity material that present higher and stiff filler content, and this aspect can explain the higher post-gel shrinkage values observed for this material. During the polymerization process of the bonded resin composite to the cavity, the shrinkage volume can generate stress when the material stiffness increases. [34] The shrinkage stress is related not only to post-gel shrinkage, but also to the elastic modulus. [54] The combination of the higher post-gel shrinkage with higher elastic modulus leads higher shrinkage stress. [7] This association was confirmed in this study, since XF showed higher modified von Mises stresses on the both cusps and also along the cavosurface margin as compared to than FO and VC.

Another important aspect observed in this study was that the higher heating temperatures (54, 65 and 68^{o}C), reduced shrinkage stress for all tested resin was observed. The heating process increases the flowability of the resin composite allowing longer deformation during polymerization process. The use of resin composite in capsules is helpful to reduce the time necessary to fill the cavity. This step may be more time consuming when the increments need to be removed from the syringe to be inserted into the cavity, a time that is sufficient for heating releasing the heat. [55] The new heating dispenser produced can improve the benefit of the heating process during the insertion of the resin composite inside the cavity. The heating process was maintained during all the insertion procedure, because the capsule was inserted and

maintained into the heating dispenser. When the capsule is positioned on the caps warmer, is necessary at least 10 to 20 seconds to remove it from the device and insert on the conventional dispenser to allow the resin composite to be placed into the cavity. The new preheating resin composite VisCalor, showed a good performance for all tested parameters. Sold in capsule format which facilitates fitting in heating devices and its applicator tip helps to take the material to the bottom of the cavity. The heating dispenser proved to be effective and easy to handle, in addition to performing well in the results obtained in this study. Another positive aspect related to the dispenser, is that this device shortens warm-up time of the composite, which improves the clinical time of the procedure.

The resin composites were not tested at room temperature, and this aspect can be considered a limitation of the study. However, the manufacturer recommends that the VC resin composite should be used only when heated, because its handling is very difficult under normal temperature conditions. The pressure necessary to extrude the resin composite make this material inapplicable at room temperature. Thus, the results of the study cannot be compared with results of other resin composites tested at room temperature. The specimens tested in this *in vitro* study were made under controlled conditions following ISO standard 4049. Therefore, the authors suggest that new *in vitro* studies that would simulate restorations made under clinical conditions and with different pre-heating temperatures.

Although the pre-heating of resin composites may not improve on the mechanical properties, it can contribute to the reduction of the polymerization shrinkage. It is a technique that can be chosen by the professional to perform on posterior restorations and even in the luting of indirect restorations. It is up to the clinician to select a material that offers a better association between mechanical properties and post-gel shrinkage to present better clinical performance. Combined with a careful selection of the material, the professional can also use the composite heating methods that may improve the clinical procedures.

CONCLUSION

Within the limitations of this *in vitro* study, the following conclusions can be drawn:

- The mechanical properties of the tested bulk fill resin composites were not affected by the heating procedures and temperatures;
- The mechanical properties of the bulk fill resin composites are not improved by heating;
- There are efficient devices in heating bulk fill resin composites, reducing the clinical time required for insertion into posterior cavities;
- The Voco Dispenser proved to be effective for heating bulk fill resin composites;
- Heating bulk fill resin composites decreased post-gel shrinkage and consequently polymerization shrinkage.

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Figures

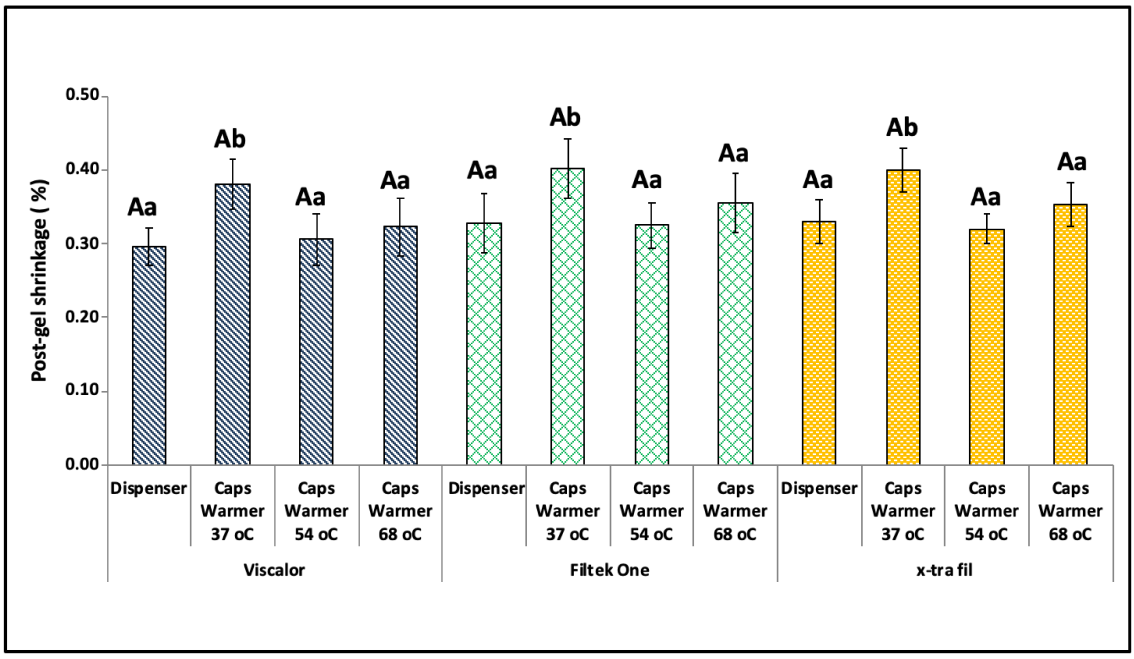

Figure 1. Mean and standard deviation values of volumetric post-gel shrinkage (%). Different uppercase letters indicate significant difference between the resin composites and lowercase letters indicate significant difference between heating methods ($P < 0.05$).

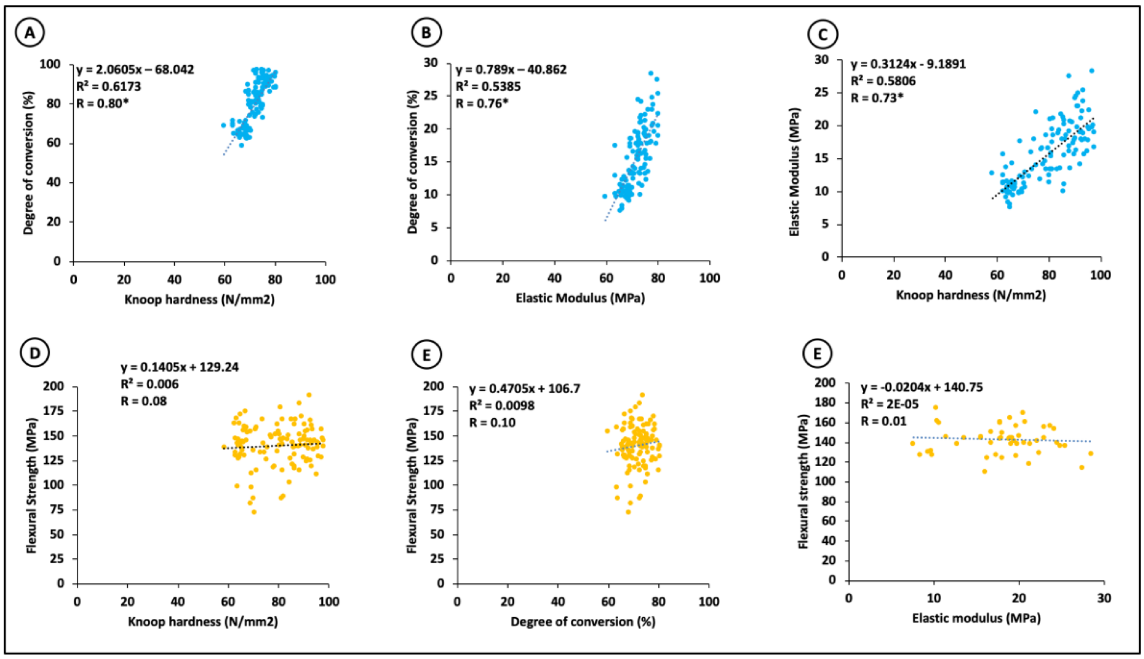

Figure 2. Pearson correlation between mechanical properties for all tested conditions. High correlations: A. DC (%) x KHN (N/mm^2); B. DC (%) x E (MPa); C. KHN (N/mm^2) x E (MPa); Correlation plots for all mechanical properties for resin composites and heating process association r- value demonstrated a high degree of correlation between the results of A. DC (%) x KHN (N/mm^2); B. DC (%) x E (MPa); C. KHN (N/mm^2) x E (MPa); and low degree of correlation between the results of. FS (MPa) x KHN (N/mm^2); B. DC (%) x FS (MPa); C. FS (MPa) x E (MPa).

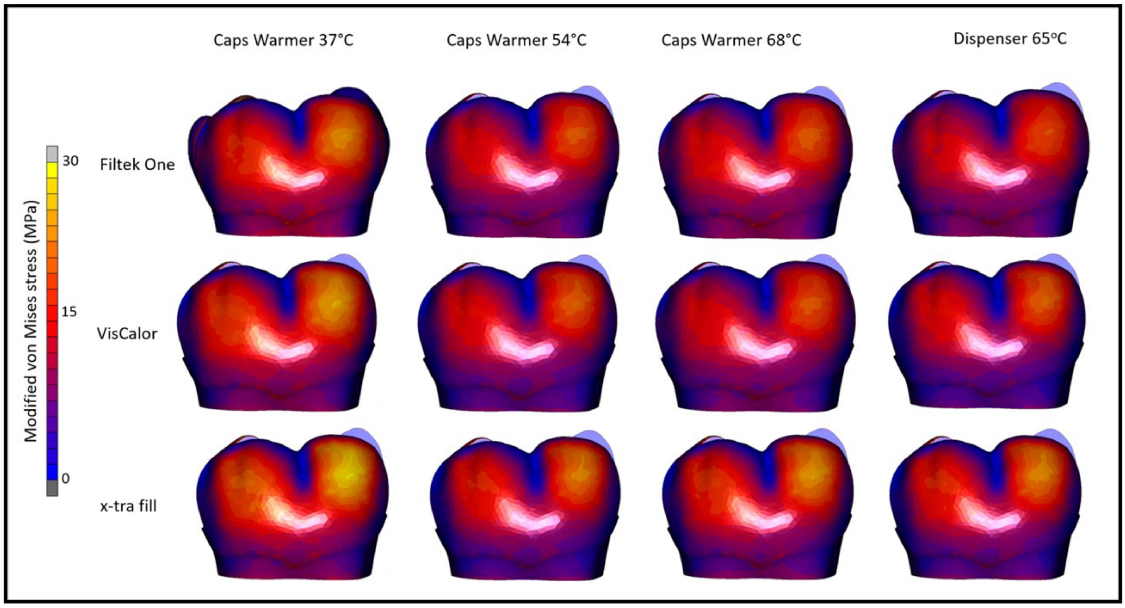

Figure 3. Modified von Mises stress distributions of buccal cusps for the different restorative filling materials and heating techniques after polymerization.

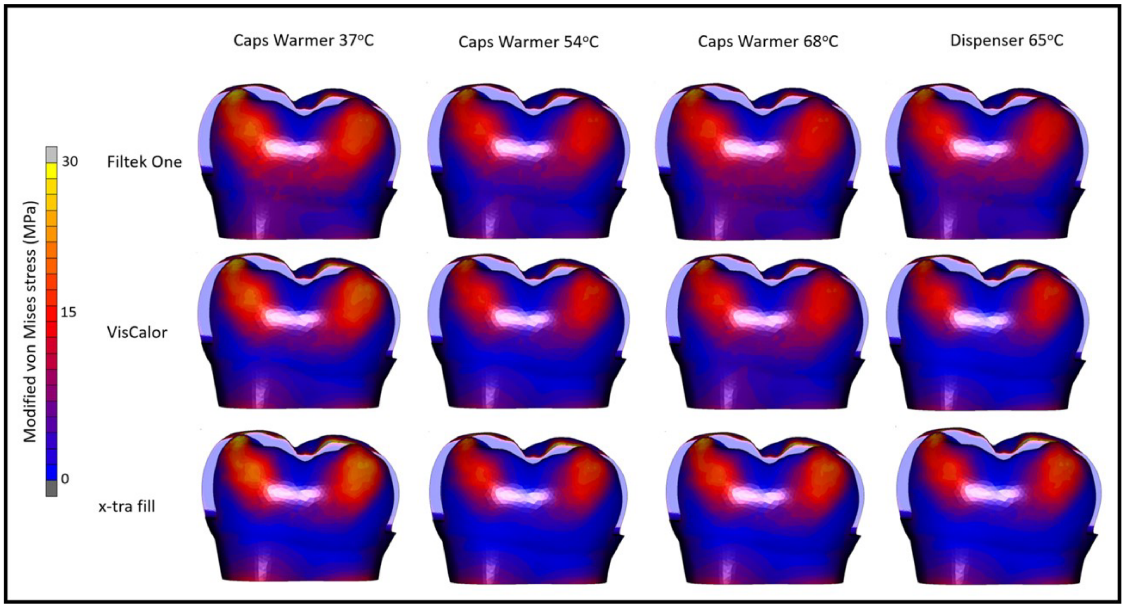

Figure 4. Modified von Mises stress distributions of lingual cusps for the different restorative filling materials and heating techniques after polymerization.

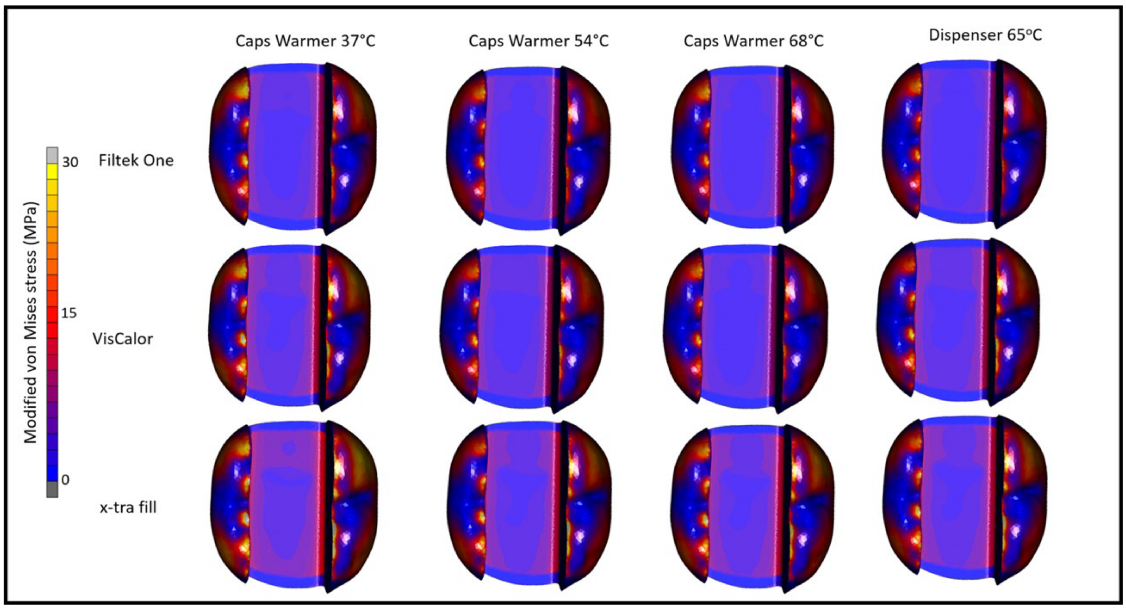

Figure 5. Modified von Mises stress distributions of cavosurface margin and on pulp floor for the different restorative filling materials and heating techniques after polymerization.

Tables

Table 1. Resin composites composition.

Material	Shade	Batch number	Organic Matrix (**)	Filler content	Filler % (wt)	Manufacturer
FO - Filtek One Bulk Fill	A2	1918325 1921308	AUDMA, diurethane- DMA, 1,12-dodecane- DMA	Silica, zirconia, ytterbium trifluoride	76.5	3M Oral Care, St Paul, MN, USA
VC - VisCalor	A2	2031100777	Bis-GMA, aliphatic dimethacrylate	Silicon dioxide	83	VOCO, Cuxhaven, Germany
XF - x-tra fil	A2	2030423	Bis-GMA, UDMA, TEGDMA	Silicon dioxide	86	VOCO, Cuxhaven, Germany

(**) Abbreviations: AUDMA, aromatic dimethacrylate; DMA, dimethacrylate; Bis-GMA: bisphenol-A glycol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

Table 2. Tissue properties

Structures	Elatic Modulus (Mpa)	Poisson Ratio	Tensile Strength (MPa)	Compressive Strength (Mpa)	References
Dentine	18.600	0.31	98.7	297.0	[31]
Enamel	84.100	0.20	10.3	384.0	[31]

Table 3. Materials properties

Resin composite/heating process		Elatic Modulus (Mpa)	Poisson Ratio (*)	Tensile Strength (MPa)	Compressive Strength (Mpa)
FO	**Caps warmer 37 ºC**	10925.5	0.24	52.8	183.2
	Caps warmer 54 ºC	12629.4	0.24	49.3	162.1
	Caps warmer 68 ºC	10022.0	0.24	51.3	121.6
	Dispenser 65ºC	10925.5	0.24	45.5	144.5
VC	**Caps warmer 37 ºC**	16031.6	0.24	52.8	183.2
	Caps warmer 54 ºC	17642.9	0.24	33.2	163.8
	Caps warmer 68 ºC	12815.0	0.24	46.5	208.7
	Dispenser 65ºC	21870.2	0.24	42.3	166.3
XF	**Caps warmer 37 ºC**	19847.8	0.24	43.3	203.4
	Caps warmer 54 ºC	23023.8	0.24	51.4	173.3
	Caps warmer 68 ºC	19088.9	0.24	43.9	153.2

Dispenser 65ºC	21870.2	0.24	47.2	197.7

*Data from Vianna *et al.,* 2018 [31].

Table 4. Mean (SD) of compressive strength (MPa), diametral tensile strength (MPa), and stress differential effect (SDE - Compressive strength/Tensile strength) values and standard deviation.

Groups	Compressive strength (MPa)			Diametral tensile strength (MPa)			Stress differential effect (SDE)		
	Filtek One	VisCalor	x-tra fil	Filtek One	VisCalor	x-tra fil	Filtek One	VisCalor	x-tra fil
Caps warmer 37ºC	183.2 (31.4) Aa	156.8 (24.5) Aa	219.8 (10.7) Aa	52.8 (3.2) Aa	41.7 (10.8) Aa	53.7 (3.2) Aa	3.5	3.8	4.1
Caps warmer 54ºC	162.1 (17.8) Aa	158.7 (13.9) Aa	173.3 (13.2) Aa	49.3 (4.3) Aa	40.9 (2.0) Aa	51.4 (4.3) Aa	3.3	4.1	3.4
Caps warmer 68ºC	141.0 (23.8) Aa	190.0 (35.1) Aa	197.0 (27.6) Aa	49.3 (4.3) Aa	52.2 (7.6) Aa	53.1 (3.6) Aa	2.9	3.6	3.7
Dispenser 65ºC	144.5 (12.8) Aa	166.3 (39.6) Aa	197.7 (26.3) Aa	45.5 (10.0) Aa	42.3 (8.8) Aa	47.2 (7.3) Aa	3.2	3.9	4.2

Different uppercase letters indicate significant difference between the resin composites; and lowercase letters indicate significant difference between heating methods ($P < 0.05$).

Table 5. Mean (SD) Flexural strength and elastic modulus values (MPa) and standard devitation.

Groups	Flexural Strength (MPa)			Elastic modulus (MPa)		
	Filtek One	**VisCalor**	**x-tra fil**	**Filtek One**	**VisCalor**	**x-tra fil**
Caps warmer 37 ºC	134.2 (21.5) Aa	129.6 (29.8) Aa	135.2 (16.4) Aa	11.4 (2.0) Ac	15.7 (2.9) Ab	19.7 (4.6) Aa
Caps warmer 54 ºC	137.3 (27.1) Aa	137.3 (14.7) Aa	141.2 (10.0) Aa	11.3 (1.5) Ac	16.3 (3.0) Ab	20.5 (3.9) Aa
Caps warmer 68 ºC	143.9 (16.6) Aa	145.7 (14.7) Aa	146.4 (13.0) Aa	11.0 (1.8) Ac	15.5 (3.6) Ab	19.1 (1.8) Aa
Dispenser 65ºC	128.8 (28.1) Aa	151.4 (24.5) Aa	147.4 (12.4) Aa	11.5 (2.5) Ac	15.8 (3.0) Ab	21.0 (2.4) Aa

Different uppercase letters indicate significant difference between the resin composites; and lowercase letters indicate significant difference between heating methods ($P < 0.05$).

Table 6. Mean (SD) of degree of conversion (%) and Knoop hardness (N/mm^2) values and standard deviation.

Groups	Degree conversion (%)			Knoop Hardness (N/mm^2)		
	Filtek One	**VisCalor**	**x-tra fil**	**Filtek One**	**VisCalor**	**x-tra fil**
Caps warmer 37 ºC	66.9 (2.9) Ac	72.0 (1.5) Ab	78.0 (2.1) Aa	66.1 (3.2) Ac	79.7 (3.5) Ab	92.9. (3.7) Aa
Caps warmer 54 ºC	67.9 (1.7) Ac	72.6 (1.7) Ab	74.9(3.0) Aa	66.0 (2.2) Ac	80.1 (3.0) Ab	90.7 (2.1) Aa
Caps warmer 68 ºC	66.8 (1.7) Ac	73.5 (1.2) Ab	74.6 (1.7) Aa	64.3 (2.3) Ac	79.0 (3.3) Ab	93.9 (2.3) Aa
Dispenser 65ºC	65.8 (2.1) Ac	71.1 (2.0) Ab	78.1 (1.8) Aa	66.6 (3.4) Ac	85.8 (3.1) Ab	90.0 (2.4) Aa

Different uppercase letters indicate significant difference between the resin composites; and lowercase letters indicate significant difference between heating methods ($P < 0.05$).

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ANEXOS

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