UNIVERSIDADE DE SÃO PAULO
FACULDADE DE ODONTOLOGIA DE BAURU

DANIELLA CRISTO SANTIN

Influence of volume and thickness on shrinkage stress and depth of cure of conventional and bulk fill composites

Influência do volume e da espessura no estresse de contração e profundidade de cura de compósitos convencionais e *bulk fill*

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Dedico este trabalho aos meus pais, Maria e Valdecir, pelo amor e dedicação em prol do meu crescimento pessoal e profissional.

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"Você nunca alcança o sucesso verdadeiro a menos que você goste do que está fazendo"

Breckenridge Carnegie

ABSTRACT

Influence of volume and thickness on shrinkage stress and depth of cure of conventional and bulk fill composites

Objective: The present study investigated, *in vitro,* the influence of volume on shrinkage stress and the influence of thickness on the depth of cure by microhardness of convencional and bulk fill composite resins.

Material and methods: Six commercial composite resins were selected: conventional (Filtek Z350 – Z350; Vittra APS – VAPS), high viscosity bulk fill (Filtek One Bulk Fill – ONE; Opus Bulk Fill APS – OBF) and low viscosity (Filtek Bulk Fill Flowable – BFF; Opus Bulk Fill Flow APS – OBFF). For the shrinkage stress analysis, specimens (n=5) were made varying the volume/C-factor (24 mm^3/0.75 and 48 mm^3/0.375). The contraction forces (N) generated from LED light-curing (30s; 1200mW/cm^2) were recorded for 300s in an UTM and the shrinkage stress was calculated (MPa). For microhardness, using two molds with 4mm diameters, 2 and 4mm thicknesses (n=5), the specimens were made and light-cured for 30s. After 24h, Knoop microhardness measures (KHN) on the top and bottom surfaces were obtained and the ratio of bottom/top microhardness was calculated determining the depth of cure. After confirming normality (Shapiro-Wilk), all data were evaluated by two-way ANOVA, followed by Tukey's test ($p \leq 0.05$).

Results: The increase of composite resin volume resulted in higher shrinkage stress for the Z350, VAPS and ONE, regardless of the C-factor employed ($p \leq 0.05$). The Z350, VAPS, BFF and OBF showed a decrease in microhardness (depth of cure) when the thickness was increased ($p \leq 0.05$). Overall groups showed adequate polymerization (bottom/top microhardness >80%) at 2mm depth, except for the OBFF, while only the ONE presented adequate polymerization at 4mm depth.

Conclusion: The volume and thickness of the increment influenced the stress and the depth of cure, respectively. The bulk fill ONE composite resin showed lower tension maintaining cure efficiency independent of volume compared to the other tested materials.

Keywords: Composite resins. Dental stress analysis. Hardness. Polymerization.

RESUMO

Influência do volume e da espessura no estresse de contração e profundidade de cura de compósitos convencionais e *bulk fill*

Objetivo: O presente estudo investigou, *in vitro,* a influência do volume no estresse de contração e a influência da espessura na profundidade de cura por microdureza de resinas compostas convencionais e *bulk fill*.

Materiais e métodos: Foram selecionadas seis resinas compostas comerciais: convencional (Filtek Z350 – Z350, Vittra APS - VAPS), *bulk fill* de alta viscosidade (Filtek One Bulk Fill - ONE, Opus Bulk Fill APS - OBF) e baixa viscosidade (Filtek Bulk Fill Flowable - BFF, Opus Bulk Fill Flow APS – OBFF). Para a análise do estresse de contração, espécimes (n=5) foram confeccionados variando o volume/fator C (24 mm^3/0,75 e 48 mm^3/0,375). As forças de contração (N) geradas a partir da fotoativação (30s, 1200mW/cm^2) foram registradas durante 300s em UTM e o estresse de contração (MPa) foi calculado. Para microdureza, utilizando dois moldes com 4mm de diâmetro, 2 e 4mm de espessura (n=5) espécimes foram preparados e polimerizados por 30s. Após 24h, leituras de microdureza Knoop (KHN) nas superfícies superior e inferior dos espécimes foram obtidas e a razão percentual de microdureza inferior/superior foi calculada determinando a profundidade de cura. Após confirmação da normalidade (Shapiro-Wilk), a análise dos dados foi realizada utilizando ANOVA a dois critérios e Tukey ($p \leq 0.05$).

Resultados: O aumento do volume de resina composta resultou em um maior estresse de contração para Z350, VAPS e ONE, independentemente do fator C empregado ($p \leq 0,05$). As resinas compostas Z350, VAPS, BFF e OBF apresentaram diminuição na microdureza (profundidade de cura) quando a espessura foi aumentada ($p \leq 0,05$). Com exceção da OBFF, todos os grupos mostraram adequada polimerização (microdureza base/topo >80%) a 2mm de profundidade, enquanto somente a ONE apresentou polimerização adequada a 4mm de profundidade.

Conclusão: O volume e a espessura do incremento influenciaram o estresse e a profundidade de cura, respectivamente. A resina composta *bulk fill* ONE mostrou menor tensão mantendo eficiência de cura independente do volume em comparação aos outros materiais testados.

Palavras-chave: Análise do estresse dentário. Dureza. Polimerização. Resinas compostas.

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LIST DE ABBREVIATIONS AND ACRONYMS

APS	Advanced Polymerization System
ONE	Filtek One Bulk Fill
OBF	Opus Bulk Fill APS
BFF	Filtek Bulk Fill Flowable
OBFF	Opus Bulk Fill Flow APS
Z350	Filtek Z350
VAPS	Vittra APS
UTM	Universal Testing Machine
%KHN	Relative Microhardness
CANs	Covalent Adaptable Networks
UDMA	Urethane Dimethacrylate
AUDMA	Aromatic Urethane Dimethacrylate
AFM	Additional Fragmentation Monomer
Bis-GMA	Bisphenol-A Glycidyl Methacrylate
Bis-EMA	Ethoxylated Bisphenol-A-dimethacrylate
TEGDMA	Triethylene Glycol Dimethacrylate

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1 INTRODUCTION

1 INTRODUCTION

Despite the evolution of composite resins and satisfactory clinical performance, drawbacks related to material contraction and the tensions generated by it during polymerization kinetics, are limitations that may compromise the restorations longevity.[1-4] Polymerization stress has been associated with a series of clinical complications[4]. When the contraction forces overcome the bond strength of the composite resin to the dental substrate, internal cracks develop and, from of these gaps the dentin fluid may circulate, degrading the adhesive interface.[5] Adverse effects such as hypersensitivity, misadaptation and secondary caries are possible to occur.[4-6]

Studies suggest that a composite resin shrinkage standard and its respective tensions are influenced by many factors such as chemical composition,[1,7,8] polymerization kinetics,[4,8] volume of increment,[9] elastic modulus[8,10] and cavity configuration factor (C-factor).[9,11] Feilzer *et al*.[12] (1987), observed that the C-factor influences the shrinkage stress of chemically activated composite resins, since the increase of the C-factor (ratio of the the adhesion area to the free area) determined higher rates of stress rates in these materials. However, photoactivated composite resins do not seem to follow this theory[9]. For these materials the volume prevails, since the polymerization reaction happens rapidly[8,9]. Thus, the researchers main focus has been to minimize the tensions generated on the adhesive interface in order to contribute to the clinical success rates.

Among the strategies used for this purpose, the incremental technique is described in the literature as the most used in dental practice.[4,13,14] This technique consists of placing the composite resin in the maximum of 2 mm thickness increments, followed by light-curing. Some authors claim that this technique can incorporate air bubbles and increase clinical time.[6,13,15] On the other hand, the incremental insertion of the composite resin causes lesser cusp deflection due to the reduction of the final volumetric contraction of the restorative material.[2,7,16] In addition, the technique allows greater stress relief since there are more free areas between the composite resin layers for the distribution of contraction forces.[7]

Nevertheless, with the purpose of decreasing chair time and minimizing dimensional shrinkage, bulk fill composite resins were launched in the market.[13,15,17,18,19] The aim is to insert the material in a single increment of 4 to 5 mm of thickness, because they present modifications in its formulation, addition of monomers with high molecular weight, modulators of polymerization, photoinitiators with higher reactivity, increase of translucency and changes in load content and/or organic matrix.[10,13,18,20] However, one of the greatest concerns with the use of a bulk fill technique would be the decline of mechanical properties due to decrease of conversion degree and depth of cure.[10,14,18,21,22]

Recently, a new technology, Advanced Polymerization System (APS), was developed and applied in conventional resin and bulk fill composites.[23] With APS technology, the amount of canphorquinone was reduced and replaced with other photoinitiator mixtures, to increase the polymerization capacity and degree of conversion of the material.[23] Another highlight is the absence of Bisphenol-A (BPA), compound of exogenous and toxic action capable of generating adverse biological effects.[24,25] In the absence of the Bis-GMA monomer, lower polymerization contraction is expected because Bis-GMA is highly viscous and when incorporated in the resin matrix, it requires the addition of diluents, which contributes to an increase in composite resin shrinkage.[8]

Many studies analyzed the polymerization stress and hardness of different bulk fill composite resins.[5,14,17-20] However, according to the authors knowledge, no data was published about the influence of APS technology in these properties. Considering that the incremental technique is the most employed until now, the insertion of bulk fill composite resins in reduced volumes could provide lower contraction and tension of polymerization, allowing greater photopolymerization efficiency and optimizing the clinical performance of the restorations. Therefore, the aim of the present study was to investigate the influence of the volume and C-factor on shrinkage stress and thickness on depth of cure by microhardness of bulk fill composite resins with and without APS technology, compared with conventional composite resins. The null hypothesis were: i) The volume or C-factor does not influence the polymerization shrinkage stress of the composite resins evaluated; ii) The depth of cure is not compromised by the variation of the composite resin increment thickness.

2 ARTICLE

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ABSTRACT

The present study investigated, *in vitro,* the influence of volume on shrinkage stress and the influence of thickness on the depth of cure by microhardness of convencional and bulk fill composite resins. Six commercial composite resins were selected: conventional (Filtek Z350 – Z350; Vittra APS – VAPS), high viscosity bulk fill (Filtek One Bulk Fill – ONE; Opus Bulk Fill APS – OBF) and low viscosity (Filtek Bulk Fill Flowable – BFF; Opus Bulk Fill Flow APS – OBFF). For the shrinkage stress analysis, specimens (n=5) were made varying the volume/C-factor (24 mm^3/0.75 and 48 mm^3/0.375). The contraction forces (N) generated from LED light-curing (30s; 1200mW/cm^2) were recorded for 300s in an UTM and the shrinkage stress was calculated (MPa). For microhardness, using two molds with 4mm diameters, 2 and 4mm thicknesses (n=5), the specimens were made and light-cured for 30s. After 24h, Knoop microhardness measures (KHN) on the top and bottom surfaces were obtained and the ratio of bottom/top microhardness was calculated determining the depth of cure. After confirming normality (Shapiro-Wilk), all data were evaluated by two-way ANOVA, followed by the Tukey's test ($p \leq 0.05$). The increase of composite resin volume resulted in higher shrinkage stress for the Z350, VAPS and ONE, regardless of the C-factor employed ($p \leq 0.05$). The Z350, VAPS, BFF and OBF showed a decrease in microhardness (depth of cure) when the thickness was increased ($p \leq 0.05$). Overall groups showed adequate polymerization (bottom/top microhardness >80%) at 2mm depth, except for the OBFF, while only the ONE presented adequate polymerization at 4mm depth. In conclusion, the volume and thickness of the increment influenced the stress and the depth of cure, respectively. The bulk fill ONE composite resin showed lower tension maintaining cure efficiency independent of volume compared to the other tested materials.

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INTRODUCTION

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MATERIALS AND METHODS

1. Experimental design

Shrinkage stress was analyzed through two factors: composite resin at 6 levels and increment volume at 2 levels. For depth of cure by microhardness, composite resins at 6 levels and thickness at 2 levels were the factors in study. Commercial composite resins with and without APS technology were selected. Bulk fill composite resins of high and low viscosity were compared to conventional composite resins, totaling 12 groups (n=5) for each analysis. The quantitative response variables were the shrinkage stress (MPa) and microhardness (ratio bottom/top microhardness, KHN). The restorative materials used in the present study are listed in Table 1.

Table 1: Manufacture's information about the composite resin materials used in the study.

Material	Code	Manufacture	Lot N^0	Composition	Color
Filtek Z350	Z350	3M ESPE (St. Paul, MN, USA)	688062	Bis-GMA, UDMA, TEGDMA, PEGDMA, Bis-EMA. Silica, zirconia and zirconia/silica cluster fillers (82 wt%)	EA2
Vittra APS	VAPS	FGM (Joinville, SC, BR)	181017	UDMA, TEGDMA, APS photoinitiator composition, co-initiators. Zirconia and silica particles (72-82 wt%)	EA2
Filtek One Bulk Fill	ONE	3M ESPE (St. Paul, MN, USA)	930814	AFM, AUDMA, UDMA, 1-12-dodecano-DMA. Silica, zirconia, zirconia/silica cluster and ytterbium trifluoride fillers (76.5 wt%)	A2
Opus Bulk Fill APS	OBF	FGM (Joinville, SC, BR)	290517	UDMA, photoiniciator and co-initiator. Silanized silica dioxide (79 wt%)	A2
Filtek Bulk Fill Flowable	BFF	3M ESPE (St. Paul, MN, USA)	N865244	Bis-GMA, UDMA, Bis-EMA, Procrylat. Ytterbium trifluoride, zirconia /silica fillers (64.5 wt%)	A2
Opus Bulk Fill Flow APS	OBFF	FGM (Joinville, SC, BR)	290517	UDMA, camphorquinone, co-initiator. Silanized silica dioxide (68 wt%)	A2

Bis-GMA: Bisphenol A glycidyl methacrylate; UDMA: urethane dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; PEGDMA: Poly ethylene glycol dimethacrylate; Bis-EMA: ethoxylated bisphenol-A-dimethacrylate; AFM: additional fragmentation monomer; AUDMA: aromatic urethane dimethacrylate.

2. Shrinkage stress

The shrinkage stress was measured from load cell deformation, similar to the methodology described by Mondelli *et al*.[9] (2016). The experimental arrangement consisted of two rectangular steel bases (6 mm x 2 mm) arranged parallel to one another. The upper base contained a movable arm connected to a load cell of 50 KgF, whereas the lower base was attached to an Universal Testing Machine (UTM - Instron 3342, Instron Co., Canton, USA) (Figure 1).

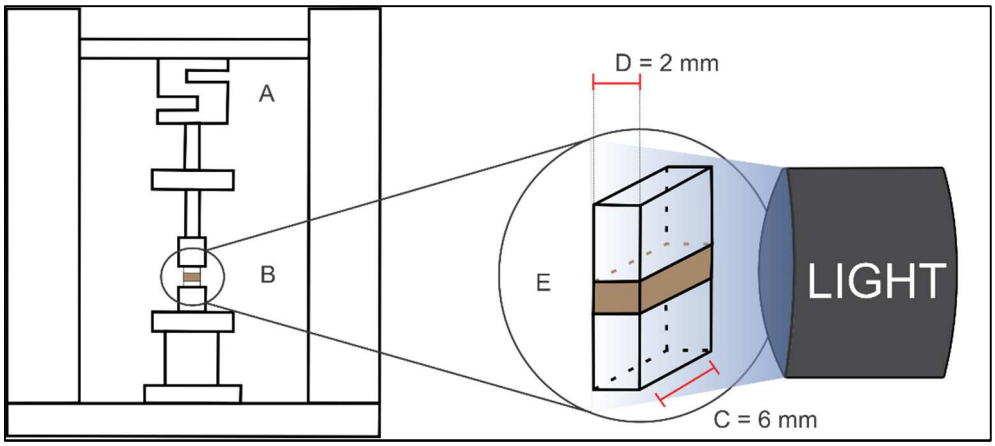

Figure 1: Equipment scheme used during the test. **A.** Load cell; **B.** Steel bases; **C.** Steel bases length; **D.** Steel base widths; **E.** Adjustable space between steel bases to insert composite resin (Adapted from Mondelli *et al*., 2016).

In order to guarantee the micromechanical retention of the composite resin to the metal bases, the internal surface of the metal bases was treated with aluminum oxide jets. Next, the composite resin was inserted in a single increment between the two steel bases. The increment was light-cured for 30s according to the manufacturer's recommendations with LED light-curing unit (Poly Wireless Kavo, Kavo, Joinville, Brazil). In order to allow the correct transmission of light through the specimen, the light source was positioned on the larger side of these bases (Figure 1). Furthermore, the irradiance (1200 mw/cm^2) was previously checked using a radiometer device (Radiometer RD-7, ECEL, Ribeirão Preto, Brazil).

During the polymerization reaction, the contraction forces (N) generated from shrinkage of the composite resin were recorded from the start of light-curing until reaching 300 s.[26,27,28] The measurements were obtained at room temperature (25^o C) by a single operator. At the end, a graph of force (N) x time (s) was assembled to present the results of the forces generated by the evaluated materials at intervals of 20, 65, 120 and 300 s by UTM software. The shrinkage stress (MPa) was calculated from the values of contraction force (N) at 300 s divided by the area of transversal section (mm^2) of the metallic bases.

The distance between the metallic bases determined the resin thickness to be inserted, varying 2 and 4mm (n=5). In this case, two geometries with different volumes and two C-factors were tested.

2.1 Determination of the composite resin volume

In order to establish the volume of composite resin to be contracted and subjected to shrinkage stress, the length was multiplied by the width of the base and composite resin thickness (or distance between the bases) (Figure 1), as shown below:

Calculation of the volume for increment with 2mm thickness:

$$v_2 = 6 \text{ X } 2 \text{ X } 2 \rightarrow v = 24\ mm^3$$

Calculation of the volume for increment with 4mm thickness:

$$v_4 = 6 \text{ X } 2 \text{ X } 4 \rightarrow v = 48\ mm^3$$

2.2 Determination of the cavity configuration factor

The C-factor was calculated from the ratio between the adhered and free surfaces of the restorative material[12]. The bonded surface consists of the sum of the base faces that were in contact with the composite resin (2 CD - Figure 1) and the free surface by the sum of the sides of the composite resin increment free from the contact with the metal bases (2 CE + 2 DE - Figure 1), as exemplified below:

Calculation of C-factor for increment with 2mm thickness:

$$\frac{2\text{X}(CD)}{2\text{X}(CE)+2\text{X}(DE)} = \frac{2\text{X}(2\text{X}6)}{2\text{X}(6\text{X}2)+2\text{X}(2\text{X}2)} = \frac{2\text{X}12}{24+8} = \frac{24}{32} = 0.75$$

Calculation of C-factor for increment with 4mm thickness:

$$\frac{2\text{X}(CD)}{2\text{X}(CE)+2\text{X}(DE)} = \frac{2\text{X}(2\text{X}6)}{2\text{X}(6\text{X}4)+2\text{X}(2\text{X}4)} = \frac{2\text{X}12}{48+16} = \frac{24}{64} = 0.375$$

3. Depth of cure by Knoop microhardness

In order to test two depth/thicknesses, specimens were prepared (n=5) for each composite resin described in Table 1 with an opaque poly-acrylic mold (Dentsply Caulk) of 4 mm diameter and 2 or 4 mm thicknesses. To allow for regular indentations, the surfaces of the specimens were made under a glass plate with Mylar strip.[29] After the composite resin filling, the set was covered by another Mylar strip to avoid inhibition by oxygen. Next, the excess of composite resin was removed with a glass slide. The composite resin was light-cured through

the polyester strip for 30 s with the same LED light-curing unit. The specimens were stored dry in the dark for 24 hours.

The Knoop microhardness was conducted by a single operator using a MicroMet 6040-Buehler (Buehler LTD, Lake Bluff, IL, USA) microdurometer. According to ISO 4545-1, a diamond indenter with a predetermined load of 50 KgF was used for 10 s.[29] In each specimen, three indentations (with 100µm distance between the points) along the top and bottom surfaces were performed. The mean of the three surface readings was calculated and the surface microhardness in percentage (%KHN) was obtained by the bottom/top ratios.[30] For effective curing, a minimum of 80% of bottom/top microhardness was established.

4. Statistical analysis

Statistical analysis was accomplished after verification of the normality and homogeneity of the data by the Shapiro-Wilk and Levene tests, respectively. All data were evaluated by two-way ANOVA, followed by the Tukey's test. For the analyses, 5% was set as the significance level ($p<0.05$) (Statistica Version 13, StatSoft Inc., Tulsa, USA).

RESULTS

All data presented normal distribution. Figure 2 shows the time-dependent development of the shrinkage forces (N) generated during the composite resin polymerization. Analyzing the graphic, the 12 groups performed the same standard during development of the forces. During light-curing (30 s) the contraction forces increased quickly and after LED light removal, the contraction forces remained at lower intensity until the end of the test (300 s) (Figure 2).

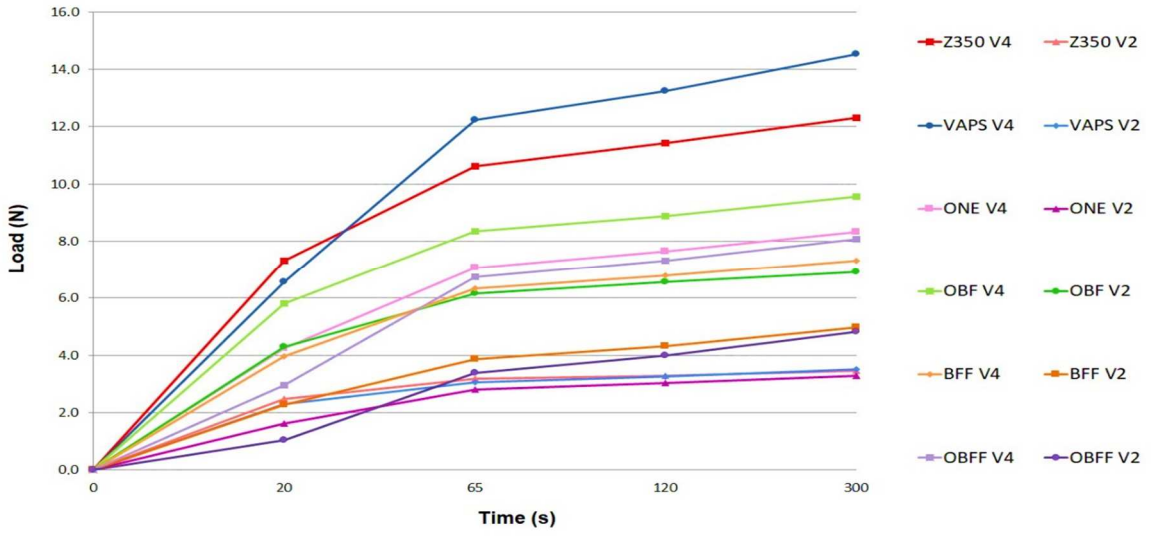

Figure 2: Curve of the forces (N) generated during the composite resin polymerization contraction in relation to time (s), according to the volume and C-factor variations.

Table 2 presents the means and standard deviations of shrinkage stress at the final period of observation (300 s). Composite resin and volume showed significant differences when evaluated individually ($p=0.000175$; $p<0.001$) and simultaneously ($p<0.001$).The values ranged from 0.270±0.04 to 1.210±0.25 MPa. The results demonstrated that the higher volume determined the highest shrinkage stress in all groups evaluated. The conventional composite resins with and without APS technology (VAPS and Z350) and the high viscosity bulk fill composite resin (ONE) developed stress significantly lower when inserted in a lower volume (24 mm^3) ($p<0.05$). The level of stress generated in the low viscosity bulk fill (OBFF and BFF) and high viscosity bulk fill with APS technology (OBF) was similar in both tested volumes and C-factors ($p>0.05$).

Table 2: Mean values and standard deviation of the shrinkage stress (MPa) at 300 s of composite resins tested in different volumes and C-factors (n=5).

Material	**24 mm^3 / C-factor 0.75**	**48 mm^3 / C-factor 0.375**
Z350	0.288 ± 0.05^{f}	1.026 ± 0.21^{ab}
VAPS	0.293 ± 0.14^{cf}	1.210 ± 0.25^{a}
ONE	0.270 ± 0.04^{f}	0.730 ± 0.14^{d}
OBF	0.586 ± 0.11^{cde}	0.796 ± 0.09^{bd}
BFF	0.414 ± 0.07^{cef}	0.610 ± 0.14^{de}
OBFF	0.401 ± 0.07^{cef}	0.667 ± 0.09^{de}

The superscript letters indicate comparisons among all tested groups (Interaction between study factors: Volume and Material). Different superscript letters shows significant differences between groups (Two way ANOVA and Tukey test, $p<0.05$).

Regarding depth of cure, the results indicated significant differences for thickness ($p<0.001$), composite resin ($p<0.001$) and for interaction between these two factors ($p<0.001$). All groups showed lower microhardness values at the bottom surface in comparison to the top (Table 3). Overall, the specimens with smaller thickness (2 mm) achieved higher bottom microhardness values compared to higher thickness specimens (4 mm). The ONE composite resin exhibited the higher ratio in percentage value of bottom/top microhardness (95.20%±2.58%) with 2 mm thickness, while the VAPS material achieved the lowest value (40.80%±1.30%) with 4 mm thickness. For conventional composite resins with and without APS technology (VAPS and Z350), low viscosity bulk fill (BFF) and high viscosity (OBF), the difference of thickness was statistically significant ($p<0.05$) since increments with higher depth determined reduced microhardness values (% KHN). No differences were found in percentage value of bottom/top microhardness for the ONE and OBFF materials when varying the increment thickness ($p>0.05$).

Table 3: Mean values and standard deviation of Knoop microhardness (KHN) at the top, bottom and depth of cure (bottom/top microhardness ratio, %) of composites for different thicknesses (n=5).

Thickness	Material	Top microhardness (KHN)	Bottom microhardness (KHN)	Bottom/Top microhardness ratio (%)
2mm	Z350	60.67 ± 1.91	55.69 ± 2.35	91.40 ± 1.94^{bc}
	VAPS	57.60 ± 2.93	48.60 ± 3.95	83.60 ± 3.36^{bd}
	ONE	53.03 ± 2.29	50.71 ± 2.11	95.20 ± 2.58^{c}
	OBF	54.27 ± 0.52	51.01 ± 1.31	92.80 ± 1.78^{bc}
	BFF	34.51 ± 0.87	31.61 ± 0.89	91.20 ± 4.38^{bc}
	OBFF	29.02 ± 2.41	21.58 ± 1.83	74.00 ± 4.35^{ad}
4mm	Z350	60.77 ± 2.16	44.61 ± 3.20	73.00 ± 7.17^{ad}
	VAPS	56.95 ± 1.79	23.65 ± 0.73	40.80 ± 1.30^{e}
	ONE	48.21 ± 3.14	45.80 ± 2.96	94.40 ± 3.36^{bc}
	OBF	53.89 ± 0.84	41.28 ± 3.19	76.40 ± 5.17^{ad}
	BFF	34.62 ± 5.25	24.79 ± 4.18	71.80 ± 9.88^{a}
	OBFF	29.32 ± 2.94	19.23 ± 1.03	65.60 ± 8.14^{a}

The superscript letters indicate comparisons among all tested groups (Interaction between study factors: Thickness and Material). Different superscript letters shows significant difference between groups (Two way ANOVA and Tukey test, $p<0.05$).

DISCUSSION

The polymerization kinetics of composites based on methacrylic monomers consists of free monomers approaching formation of strongly bonded polymeric structures, resulting, unavoidably in the dimensional shrinkage of the material (1-6%).[5,8,31,32] Associated with this process, internal tensions are produced in the dental cavity in order to restrict the composite resin contraction.[32] As a result, a variety of clinical defects may appoint the replacement of the restorations.[1,4,5,6,32]

In an attempt to minimize the stress generated by evaluated resinous materials, our study hypothesized that the insersion of increments with lower volumes could decrease the forces generated by bulk fill composite resins's polymerization shrinkage. For the Z350, VAPS and ONE materials, the volume was significantly relevant, but the C-factor did not have any influence, rejecting the first null hypothesis. As expected, in agreement with previous

studies,[18,28,33] in the present study, the conventional composite resins showed the highest stress values when inserted in a thickness of 4 mm (48 mm^3). It is important to highlight that although the manufacturers recommend their use in increments of at most 2 mm thickness, the configuration allowed to compare the behavior of these materials with bulk fill composite resins.

With respect to the limitations of the study, the shrinkage stress is not considered a property of the materials, thus, to assess this, an indirect method recommended by Academy of Dental Materials was used which is based on the load cell deformation connected in a UTM.[34] In addition, because the specimens were kept between metal bases, a lateral light-curing method was adopted instead of through the composite resin. Although the stress can be evaluated for several hours, the recording time of the contraction forces was adjusted to 300 s. According to previous observations, 5 min is sufficient time to assess the standard of polymerization stress development,[19,22,26,27,28,34] because most of the shrinkage occurs in the first 60 s after the light-curing.[26,35]

The magnitude of stress is influenced by many factors such as curing reaction, composition of the material, elastic modulus and degree of conversion.[1,4,8-11] The impact of C-factor has been controversial because some authors assume that the amount of light-curing composite resin available for cure (volume) is not taken into account.[8,9,32] The data found in the present research may be explained following this argument. Higher levels of tension were obtained for increments of higher volume with a C-factor correspondingly low (0.375), not supporting the theory described by Feilzer *et al*.[12] (1987). More monomers were available for formation of cross-links, resulting in a higher shrinkage and subsequently higher polymerization stress.[8,9,16]

In general, all composite resins showed lower shrinkage stress when inserted in lower volume (24 mm^3) and higher C-factor (0.75). Whit the increase of volume (48 mm^3) and decrease of C-factor (0.375), higher shrinkage stresses were induced, regardless of their physicochemical characteristics. Among the four bulk fill composite resins tested, the ONE group presented significant differences in shrinkage stress with the variation of the increment volume and the lowest levels of stress were found for the lower volume (24 mm^3). These data seems to confirm the manufacturers statement that modifications in the composite chemistry contribute to shrinkage stress relief. These findings also allows to suggest that although the material is indicated to restore large cavities up to 5 mm in depth, clinically, the insertion of increments with reduced volumes could generate less shrinkage stress, optimizing the clinical performance of the material.

As mentioned in the literature, the resinous organic matrix formulation performs an important role in the development and intensity of polymerization stress.[1,4,7,8] Besides the use of monomers with high molecular weight that provide low shrinkage,[4] the polymer reversible reorganization through the presence of covalent adaptable networks (CANs) has been described as alternative to reduce shrinkage stress.[36] The low tension observed in the bulk fill composite resin ONE can be explained by the formulation to be based on UDMA and AUDMA (Table 1) monomers. Associated with this, according to Shah *et al*.[35] (2017), the presence of an additional fragmentation monomer (AFM) allows the formation of CANs that contribute, consequently, for the shrinkage stress relief. While the polymeric network is being formed, the monomer interacts dynamically breaking the covalent bonds of the network, determining the release of the accumulated stress.[36]

The bulk fill flow composite resins were less influenced by the volume and C-factor, showing no statistical differences.The volume was not significantly associated to the maximum value of stress obtained in the flowable materials. The BFF and OBFF resins showed the lowest stress rates for the higher volume tested (48 mm^3), with the other groups. This finding is in agreement with previous studies, which can be attributed to the low elasticity modulus achieved by the least amount of inorganic filler, presence of monomers that exhibit high molecular weight and reduced viscosity (UDMA, Procrylat).[18,32] The viscoelastic behavior of the material is an important factor in the shrinkage stress relief.[4,20,32]

The present study also investigated if the APS technology could contribute to decrease of the shrinkage stress. It was assumed that because the materials are free of viscous monomers (Bis-GMA and Bis-EMA), less diluents such as TEGDMA would be added to the composite resin formulation, resulting in lower stress because diluent monomers tend to provide higher contraction rates due to their high reactivity and low molecular weight[8] and, consequently, highest levels of stress may occur in these conditions. However, the findings of the present research do not agree with this statement, since the values of shrinkage (Figure 1) and stress (Table 2) obtained were equivalent for the conventional composite resins. Future researches could be perfomed to evaluate other properties of this materials with the same technology, complementaing our study.

In order to analyze the depth of cure of the evaluated materials, the Knoop microhardness test was done, because there is an adequate correlation with the degree of cure.[10,21,34] The effect of the resin increment thickness in the microhardness was also investigated. The present study demonstrated that the thickness variation significantly compromise the efficiency of cure of the Z350, VAPS, BFF and OBF composite resins, rejecting the second null hypothesis. These results corroborate the findings of previous

researches, which showed a decrease in microhardness values with the increase of the composite resin increment thickness.[30,37]

It was reported that ideally the materials should exhibit a minimum percentage of 80% of bottom/top microhardness to present a good clinical performance.[38] In the present study, only one (ONE) of the four bulk fill composite resins evaluated showed enough polymerization whit the 4 mm thickness. Similar results were found by Yap *et al*.[39] (2016). Thus, the use of bulk fill composite resins with 4 mm thickness should be questioned according to results obtained in the current study.

The difference in the microhardness values of the materials tested probably is related to the composition. As mentioned in the literature, the concentration and size of the filler particles may change the light refractive indices, impairing its penetration into the deeper layers of the material.[30,37] The type of photoinitiator present in the formulations shoud be considered, because the incompatibility with the wavelength issued by the light-curing unit can limit the polymerization reaction.[4,10]

The APS technology materials showed low values of microhardness. These results may be the reason for the difficulty in measuring the test indentation, more frequently for the bottom surface analysis. Another reason is that the photoinitiators used to replace part of the camphorquinone of the composition have little absorption in the wavelength range emitted by the light-curing unit used in our research (420 - 480 nm). This data is relevant and highlights the knowledge that the professional should have regarding the composition of the materials used and the wavelength of the light-curing unit. However, the data must be interpreted with caution, more researches are necessary to investigate the technology's purpose in the materials performance.

On the other hand, some studies found satisfactory curing depth in bulk fill composite resins, mainly those of flowable viscosity.[22,33,39] Although the literature states that increasing translucency in low viscosity bulk fill composite resins favors light dissipation through the material, and the higher organic matrix content containing low molecular weight monomers allows for high crosslinking density,[22,40] the present study has been unable to demonstrate adequate polymerization in these composite resins. Parameters used in the method, besides factors related to the confection and storage of specimens, may have contributed to discrepancies in the findings.[39]

The present research showed that composite resin ONE, even being a high viscosity material, was able to accumulate less tension. Contrarily to flowable composite resins, higher opacity did not affect the light transmission through the material since adequate polymerization levels (bottom/top microhardness higher than 80%) were found in the two-thicknesses tested. Therefore, for this material, the low stress levels can not be attributed to subpolymerization.

Probably, the differential of the composite resin is that, while the shrinkage forces relief occurs, the monomers rearrange to form new bonds.[36]

CONCLUSION

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

1. The insertion of the materials in great volume increments produced higher shrinkage stress, even in the composites bulk fill.
2. The increase of the resin thickness reduced the depth of cure.
3. Conventional resins showed better performance in increments of 2 mm thickness and 24 mm^3 of volume.
4. Despite the four investigated bulk fill resins, only the ONE group showed lower stress rates and bottom/top microhardness higher than 80% when inserted in 4mm.
5. Composites with APS technology did not demonstrate superior performance to composites without this technology.

ACKNOWLEDGMENT

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3 DISCUSSION

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4 FINAL CONSIDERATIONS

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Within the limitations of the present *in vitro* study, it was concluded that greater volume of increments produced higher shrinkage stress mainly in conventional composite resins. The increase of the thickness reduced the depth of cure of the evaluated materials. Only the ONE group showed enough polymerization on 4mm thickness and lower stress rates. Composites with APS technology did not demonstrate superior performance to composites without this technology.

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APPENDIX

APÊNCIDE A - DECLARAÇÃO DE USO EXCLUSIVO DE ARTIGO EM DISSERTAÇÃO/TESE

DECLARATION OF EXCLUSIVE USE OF THE ARTICLE IN DISSERTATION/THESIS

We hereby declare that we are aware of the article (Influence of volume and thickness on shrinkage stress and depth of cure of conventional and bulk fill composites) will be included in (Dissertation/Thesis) of the student (Daniella Cristo Santin) and may not be used in other works of Graduate Programs at the Bauru School of Dentistry, University of São Paulo.

Bauru, april 23rd, 2019.

Daniella Cristo Santin _____

Author Signature

Rafael Francisco Lia Mondelli _____

Author Signature