Abstract
The study analyzed the influence of temperature on the depth of cure of bulk-fill composite resins. Three discs (ISO 4049/2000) from each group were made, and four restorative materials were investigated: Opus Bulk-Fill Flow, Opus Bulk-Fill APS, Filtek™ One Bulk-Fill, and Filtek™ Bulk-Fill Flow. They were light-cured (Gnatus ± 1,200 mW/cm²) for 20 seconds, varying the temperature (23°C and 5°C) and simulating the use of the product both at room temperature and under refrigeration. The materials were inserted in aluminum matrices with 10 mm in depth and 4 mm in diameter. A clear film strip and a glass microscope slide were positioned at the top and bottom surfaces. The material was condensed and light-cured with the tip of the light source in close contact, for 20 seconds on the top surface. Immediately after irradiation, the samples were removed from the mold, and the uncured part was removed with a plastic spatula. The measurement was performed with a micrometer ± 0.1 mm, and the value was divided by 2. The data were analyzed with two-way ANOVA at a significance of 0.05. There was a statistical difference in the temperature between the results obtained in Opus Bulk-Fill APS (p <0.001) and Filtek™ Bulk-Fill Flow (p = 0.018) resins. For the temperature of 5°C, Filtek™ Bulk-Fill Flow showed a statistical difference compared to the other resins, while Opus Bulk-Fill Flow and Opus Bulk-Fill APS did not. For the temperature of 23°C, Filtek™ Bulk-Fill Flow maintained the statistical difference from the others, but Filtek™ One Bulk-Fill and Opus Bulk-Fill APS did not present statistical differences. Storing the Filtek™ Bulk-Fill Flow composite resin in the refrigerator caused a greater depth of cure than the other resins, and the depth of cure decreased at room temperature. As for Opus Bulk-Fill APS, the depth of cure decreased proportionally to the temperature decrease. The temperature may affect the depth of cure of some composite resins.

Keywords: Composite resins. Depth of cure. Temperature.
factor, the most common composite resins indicate adding the material in small increments of up to 2 mm and light-cure them, repeating the process until filling the cavity (Altan et al. 2018).

Restorations made with the increment method have been a standard for restorations to this day (Ilie et al. 2015). This method is based on inserting small resin layers in the cavity to be restored, which provides a more natural result. However, the time and handling of the resin can represent a weakness of this technique. The decrease in material volume during light-curing is also a major clinical disadvantage for composite resin restorations (Vinagre et al. 2016). Shrinkage can cause marginal cracks, cavity floor detachment, and movement of the tooth structure, such as cusp deflection, leading to the risk of infiltration, enamel and dentin cracks, and cusp fractures (Heintze et al. 2015).

Dentists increasingly seek materials to facilitate and accelerate dental care, preventing or at least minimizing the risk of contamination, infiltration, and debonding, among others, which may compromise the success of procedures. These materials facilitate professional performance and provide more comfort to the patient because the procedures can be performed in a shorter time than usual, resulting in less time-consuming and more profitable treatments, and ultimately increasing outcome expectations (Altan et al. 2018). However, for a long and satisfactory outcome, other factors may interfere, such as the condition of tissues in the cavity to be inserted, the adhesive system used, the correct use of the restorative material following the manufacturer's instructions, patient cooperation, and operator ability to perform the procedure.

Besides conventional high- and low-viscosity resins applied in incremental layers of up to 2 mm, a new technique has been used to facilitate and simplify restorative procedures performed in stages. It is called the bulk-fill technique, performed with bulk-fill resins (Altan et al. 2018). This type of resin was developed due to the need for incremental layers, as they allow inserting the material in deep cavities (4-5mm) (Maciel et al. 2017). This material is used inside the cavities, and according to the manufacturers, areas of high masticatory effort require a layer of conventional resin to make the restoration more resistant (Guo et al. 2016; Yasa et al. 2017).

An ideal material has been sought to replace all properties of the tooth structure to reach the maximum capacity of restorative materials, considering something that can be essential to the final result: the storage of such material in the dental office (Souza et al. 2011).

Thus, several studies are being developed, in an attempt to understand the interference of material temperature at the time of the restorative procedure on the final result and treatment longevity. Thus, many researchers understood that increasing the temperature during restorative material storage before the procedure facilitates polymerization, producing more satisfactory and lasting results. Some manufacturers indicate storage in cold room temperatures (Souza et al. 2011).

This study aims to evaluate whether the flowable and paste bulk-fill composite resins will not show differences in depths of cure, varying the temperature during storage time. Thus, the null hypothesis of the following study is that flowable and paste composite resins will not show differences in depth of cure at different temperatures.

2. Material and Methods

Composite resins

Four restorative materials were investigated: 1) Paste bulk-fill composite resins: Filtek™ One Bulk-Fill (F1, 3M-ESPE, St Paul, MN, USA) and OPUS Bulk-Fill APS (O1, FGM, Joinville, SC, Brazil); 2) Flowable bulk-fill composite resins: Filtek™ Bulk-Fill Flow (F2, 3M-ESPE, St Paul, MN, USA) and OPUS Bulk-Fill Flow (O2, FGM, Joinville, SC, Brazil). Table 1 describes the composition of the materials tested.

Storage temperature

The composite resins were initially used at room temperature (23 ± 1°C) and later submitted to storage for 24 hours at low temperatures (5°C ± 1°C).
Table 1. Composition of composite resins.

<table>
<thead>
<tr>
<th>INITIALS</th>
<th>NAME</th>
<th>LOT</th>
<th>MANUFACTURER</th>
<th>COMPOSITION</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>Filtek™ One Bulk Fill A2</td>
<td>3M-ESPE (St Paul, MN, USA)</td>
<td>3M-ESPE (St Paul, MN, USA)</td>
<td>As inorganic filler particles are a combination of non-agglomerated / non-aggregated 20 nm silica particles, non-agglomerated / non-aggregated zirconia particles of 4 to 11 nm, zirconia / silica nanoagglomerates (composed of silica particles 20 nm and zirconia particles from 4 to 11 nm) and ytterbium trifluoroetho particles in agglomerated particles of 100 nm. The inorganic content is about 76.5% by weight (58.5% by volume). A 3M ™ Filtek ™ One Resin Bulk Fill with AFM (monomer for dynamic relief of polymerization contraction stresses), AUDMA, UDMA and 1, 12-dodecanedMA. Filtek One Resin Bulk Fill is applied without prior use or use of methacrylate-based dentin adhesive systems, such as those manufactured by 3M, which permanently unites restoration to dental structure. A Filtek One Resin Bulk Fill is packed in traditional seringas and single-dose capsules (unitized).*</td>
</tr>
<tr>
<td>O1</td>
<td>OPUS Bulk Fill A2</td>
<td>290819</td>
<td>FGM (Joinville, SC, Brazil)</td>
<td>Urethane dimethacrylate monomers, stabilizers, photoinitiating composition (APS) and co-initiators; inorganic filler of silanized silicon dioxide (silica), stabilizers and pigments.*</td>
</tr>
<tr>
<td>F2</td>
<td>Filtek™ Bulk Fill Flow A2</td>
<td>3M-ESPE (St Paul, MN, USA)</td>
<td>3M-ESPE (St Paul, MN, USA)</td>
<td>Treated silanized ceramic; diurethane dimethacrylate (UDMA); substituted dimethacrylate; bifesnol A polyethylene glycol diether dimethacrylate (BISEMA); ytterbium fluoride; bisphenol A di- (2-hydrox-ipropoxy) dimethacrylate (BISGMA); benzotriazole; triethylene glycol dimethacrylate (TEGDMA) and ethyl 4-dimethylaminobenzoate.*</td>
</tr>
<tr>
<td>O2</td>
<td>OPUS Bulk Fill Flow A2</td>
<td>051120</td>
<td>FGM (Joinville, SC, Brazil)</td>
<td>Urethane dimethacrylate monomers, stabilizers, camphorquinone and co-initiator; inorganic fillers of silanized silicon dioxide (silica), stabilizers and pigments.*</td>
</tr>
</tbody>
</table>

* Composition reported by manufacturer.

Depth of cure

Three discs of each material (F1, O1, F2, and O2) were prepared with a Gnatus light source, following the ISO 4049 standard. The materials were inserted in aluminum matrices of 10 mm in depth and 4 mm in diameter. A clear film strip and a glass microscope slide were positioned at both ends (Figures 1A to E).

The material was condensed and light-cured with the tip of the light source in close contact, for 20 seconds on the top surface. Immediately after irradiation, the samples were removed from the mold, and the uncured material was scraped with a plastic spatula. The cylinder height of the polymerized material was measured with a micrometer with a precision of ± 0.1 mm and divided by two (Figures 2A to F) (Lima et al. 2018).

Statistical analysis

The data will be analyzed with two-way ANOVA and Tukey’s test with a significant difference test (α=0.05). The homogeneity of variances will be analyzed with the Levene test. Normality tests will be analyzed with the Shapiro-Wilk test and equality of variances, followed by parametric statistical tests.

3. Results

There was a statistical difference in the temperature among the results obtained from the Opus Bulk-Fill APS (p<0.001) and Filtek™ Bulk-Fill Flow (p=0.018) resins. The samples preserved at 5°C and Filtek™ Bulk-Fill Flow showed statistical differences from the other groups, but Opus Bulk-Fill Flow and Opus Bulk-Fill APS did not show statistically significant
Effect of low temperature and room temperature on the depth of cure of bulk-fill composite resins

differences. As for the room temperature at 23°C, Filtek™ Bulk-Fill Flow maintained the statistical difference from the others, but Filtek™ One Bulk-Fill and OpusBulk-Fill APS did not (Table 2).

Figure 1. Materials used: A - Glass plate; B - Aluminum matrix; C - Polyester strip. D - Internal measure of the matrix; E - Height of the Matrix.

Figure 2. Sample preparation: A and B- adaptation of the resin in the matrix for light curing; C - resin cured. D - Uncured material remaining in the polyester strip; E - Opening of the matrix; F - Removal of uncured resin with a plastic spatula.
Table 2. Depth of cure results achieved by resins stored in a refrigerator (5ºC) and at room temperature (23º): Depth of cure of resins used at different temperatures.

<table>
<thead>
<tr>
<th>Composite Resin</th>
<th>Opus Flowable</th>
<th>Opus APS</th>
<th>Filtek™ One</th>
<th>Filtek™ Flowable</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>23º</td>
<td>3.70 [0.06]Ca</td>
<td>4.18 [0.02]Ba</td>
<td>4.16 [0.12]Ba</td>
<td>4.82 [0.10]Aa</td>
</tr>
<tr>
<td>5º</td>
<td>3.78 [0.05]Ba</td>
<td>3.64 [0.08]Bb</td>
<td>4.1 [0.07]Ba</td>
<td>5.07 [0.02] Aa</td>
</tr>
</tbody>
</table>

* Means [SD] followed by the same uppercase letter in the lines and lowercase in columns do not differ by Two Way Anova (p <0.05) and Tukey test.

Storing the Filtek™ Bulk-Fill Flow resin in a refrigerator caused a greater depth of cure than the other resins, and the depth of cure decreased at room temperature. As for Opus Bulk-Fill APS, the depth of cure decreased proportionally to the temperature decrease. The temperature may have affected the depth of cure for some resins.

4. Discussion

The null hypothesis of this study was rejected because the groups presented differences among the materials and the temperatures.

Technological improvements are being achieved and applied daily in dental offices, especially regarding composite resins, which are extensively requested by patients for their esthetic properties. However, the results depend on the materials used. Attempting to improve the characteristics of these materials and increase their durability, new monomers and changes in the inorganic matrix in resin compositions have been proposed (Schmitt et al. 2016; Dantas et al. 2018). In bulk-fill composite resins, manufacturers have added components such as benzoyl peroxide, camphorquinone, and Ivocerin for enhanced capabilities that allow light-curing in deep cavities.

The use of bulk-fill composite resins is being claimed for posterior restorations, therefore, their physical properties such as tensile strength, elastic modulus, polymerization shrinkage, and degree of conversion must be understood to achieve a satisfactory restoration. Factors such as translucency and potent light sources can provide a high depth of cure for bulk-fill resins (Santis et al. 2020). Consequently, the depth of cure may have been affected by the characteristics and composition of the material.

Comparisons between the conventional technique with resin increments and the filling technique show that bulk-fill resins can be safely used in cavities from 4 to 5 mm in depth, filling the cavity faster, reducing working time, and facilitating material handling by the professional at the time of restoration (Ilie et al. 2014). Although both resins reached a depth of ± 4 mm, as recommended by the manufacturer, some performed better when the temperature changed.

According to reports, bulk-fill resins show low shrinkage after polymerization, but an increase in the depth of cure is still controversial (Lempel et al. 2021). It was impossible to verify the polymerization shrinkage in this study, but the consistency of flowable composite resins visibly changed when handling the material.

The mechanism used in new bulk-fill resins, which possibly reduces polymerization shrinkage, varies depending on the manufacturer. The factor that reduces this volumetric shrinkage is not very explicit, but these resins suffer less from this effect in posterior teeth restorations, promoting better longevity of restorations. Different manufacturers present materials in common, such as specific monomers, adjuvant monomers, different photoinitiators, and the addition of other inorganic fillers (Lempel et al. 2021). This would possibly explain the difference in the depth of cure of materials according to the manufacturer, verified in this study.

When comparing the same bulk-fill resins, verifying only the flowable consistency and paste (or regular consistency), some studies show that flowable resins have a higher polymerization shrinkage than the paste, and the less flowable ones behave better when polymerized, producing a lower degree of shrinkage. However, some studies still show higher efficacy for bulk-fill resins than conventional ones, resulting in restorations with higher longevity (Silva et al. 2019). Conventional and bulk-fill resins were not compared in this study, but there is a significant difference in the depth of cure of the material and its consistency.
The depth of cure of dental restorative materials depends on the light collimation in contact with the photoinitiator, which is extremely important for the final restoration outcome (Monterubbianesi et al. 2016; Iyap et al. 2016). The light source used can directly interfere with the results, but proper use was made according to the manufacturer’s instruction to prevent any final interferences.

The effective light-curing in increments of 4 mm of thickness required introducing photoinitiators in the bulk-fill resins, with a higher ability to absorb the light emitted in the light-curing process, along with the modification in the translucency and opacity of the composition and the reduced number of inorganic particles in the resins (Ilie et al. 2013; Souza Junior et al. 2014). The presence of photoinitiators such as benzoyl peroxide, camphorquinone, and Ivocerin makes a difference in the capacity of bulk-fill resins to reach a depth of cure of larger increments than conventional resins. This may be explained by the reduction of inorganic particles, as light penetration is closely related to the presence of such particles.

5. Conclusions

The depth of cure of bulk-fill resins indicated by the manufacturers was within the requirements described in the ISO 4049 standard.

Storing the Filtek™ Bulk-Fill Flow resin in a refrigerator resulted in a greater depth of cure than the other resins, and the depth of cure decreased at room temperature. As for Opus Bulk-Fill APS, the depth of cure decreased proportionally to the temperature decrease. The temperature may affect the depth of cure of some resins. Further studies are required to correlate these results to other material properties.

Authors’ Contributions: FERREIRA, A.F.E.: acquisition of data, drafting the article and critical review of important intellectual content; SILVA, I.M.: acquisition of data, drafting the article and critical review of important intellectual content; DIETRICH, L.: analysis and interpretation of data, drafting the article, and critical review of important intellectual content; COSTA, M.D.M.A.: analysis and interpretation of data, drafting the article, and critical review of important intellectual content; ANDRADE, C.M.O.: critical review of important intellectual content; MARTINS, V.M.: conception and design, acquisition of data, drafting the article and critical review of important intellectual content. All authors have read and approved the final version of the manuscript.

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