A micro-computed tomography evaluation of the change in volume of different bulk-fill composite materials caused by polymerization shrinkage

Suzan Cangül1, Özkan Adıgüzel2, Savaş Sağmak3, Begüm Evran1

1 Dicle University, Faculty of Dentistry, Department of Restorative Dentistry, Diyarbakir, Turkey
2 Dicle University, Faculty of Dentistry, Department of Endodontics, Diyarbakir, Turkey
3 Adıyaman University, Faculty of Dentistry, Department of Restorative Dentistry, Adıyaman, Turkey

Abstract

Aim: Composite resins contain different monomers and fillers that are directly affected by polymerization shrinkage. Therefore, an accurate and reliable method is needed to measure the volume changes. The aim of this study was to evaluate the amount of volume change associated with polymerization shrinkage in four different bulk-fill composite materials in class II restorations using a micro-CT device, which has high resolution and provides 3-dimensional images.

Methodology: A total of 40 human 3rd molar teeth were used. First, standard class II cavities were opened on the mesial surfaces of all the teeth, and then the first micro-CT images were obtained. The same adhesive material was applied to all the teeth. The teeth were then separated into four groups, and a different bulk-fill composite was applied to each group; Filtek (FTK), X-tra Fil (XTF), Tetric Evo Ceram (TEC), and Filtek One (FLO) and the second micro-CT images were obtained. Then after polymerization of the materials, the final micro-CT images were taken, and analyses were made according to the scanning results. The Kruskal Wallis and Mann Whitney U-tests were used in the statistical evaluation of the data.

Results: The volumetric gap formed after polymerization of the composite resins was not determined to be statistically significant (p>0.05).

Conclusion: The volumetric difference (%) between the composite resin and the dental tissue following polymerization was seen to be greatest in XTF and least in FTK. It was concluded that the volumetric gap caused by the polymerization shrinkage of the tested materials may be due to the structure of the material.

Keywords: Micro-CT, bulk-fill composite, volumetric gap, polymerization shrinkage

Introduction

The primary aim in the restoration of dental tissues is the coverage of the dental tissue that is exposed following removal of the dental decay (1, 2). The restoration will provide the necessary tightness around the edges to prevent leakage, recurrent caries and potential pulp damage which may occur (3). The most important factor determining the resistance of the restoration is the provision of a prepared compatible surface and the capacity to cover the cavity walls. Ideally, it should result in a robust bond between the restorative material and the tooth surface with minimal marginal leakage. Effective marginal
adaptation is of critical importance especially in posterior restorations (4). Despite current technological developments, no restorative material binds perfectly to the tooth surface. By resulting in a fracture along the edges of the restorative material, this failure in bonding leads to the emergence of a gap and microleakage (5).

Composite resins, which are currently often used as restorative material because of superior mechanical properties, aesthetic results, and the possibility of minimally invasive treatment, have the significant disadvantage of stresses occurring in dental tissues as a result of polymerization shrinkage (6,7). By creating tension between the tooth and the material, polymerization shrinkage weakens the integrity of the restoration (4). As a result of volumetric changes occurring during polymerization, there may be a separation in the tooth-composite interface, causing secondary caries and cracks in the enamel (8,9). For successful composite restorations, the composite-tooth merging is of great importance in protecting the marginal integrity. One of the most important factors in the failure of current composite resin restorations is microleakage associated with polymerization shrinkage, which cannot be prevented (4). Microleakage is defined as the penetration of fluid, bacteria, molecules, and ions that cannot be clinically determined between the cavity wall and the restoration (10). As a result of this type of microleakage, there is discoloration of the restoration, sensitivity in the tooth, the formation of recurrent caries, and eventually failure of the restoration (2).

The configuration factor (C-factor) is used to describe the relationship between the form of the cavity and the level of stress occurring as a result of polymerization shrinkage (11,12). The C-factor is calculated from the ratio of the number of bonded surfaces to unbounded surfaces and is affected by the extent of the shrinkage and the stress that occurs. It is assumed that in cavities of larger dimensions with a higher C-factor, polymerization shrinkage is increased (7). The C-factor is accepted as a significant marker of shrinkage stress in dental composite restorations. However, the regression of shrinkage cannot be evaluated according to the C-factor alone (12).

It is extremely important to reduce polymerization shrinkage in respect of the success of the restoration. Therefore, it is recommended that composite resins are placed in the cavity in layers not exceeding 2mm. However, this method causes an increase in the number of light applications, especially in deep cavities, and prolongs the procedure for the patient (13).

Developments in composite resins have supported the development of bulk-fill composites, which can be polymerized in a single application up to 4-6mm in depth according to the producers because of modifications to the content (10,14). Thus, the material can be placed in the cavity at a greater thickness and greater mass (13). In addition to minimum polymerization shrinkage, the placement of bulk-fill composites to the cavity walls, which have superior physical and mechanical properties, is extremely easy because of the fluid texture (15-17).

Although bulk-fill composites have lower viscosity compared to traditional composites, the polymerization shrinkage is not as high as that of fluid composites (13). They have a low elasticity modulus to reduce polymerization stress. As they have polymerization modulators allowing a sufficient degree of polymerization, it is not necessary to follow the layering technique (15,18,19).

Previous studies of microleakage analyses have presented valuable information about restorative materials and the techniques used. Various methods have been used for the evaluation of microleakage, including the staining method, micro-computed tomography (µCT), scanning electron microscope, radioactive isotopes, air pressure, neutron activation analysis, and bacteria activity analysis (2). Compared to other methods, µCT has several advantages. First, as it is unnecessary to take slices from the samples, it allows the same sample to be used several times. The imaging procedure can be easily repeated, or changes can be made on the image using special software. This method allows the imaging of the material together with mineralized tissues (20). Therefore, the microleakage and gap that occurs between the tooth and the composite resin after polymerization can be successfully revealed using µCT (21).

It can be used to measure the gap between the composite resin and dental tissues and to determine the amount of microleakage. The gap that is formed, in other words, the predicted microleakage, which is associated with the C-factor, has been revealed in several studies using µCT. These studies have shown that with the use of µCT, 3-D quantitative evaluation of the amount of microleakage can be successfully made without the disadvantages of traditional techniques (22,23).

The aim of this study was to evaluate with µCT the volumetric change associated with polymerization shrinkage of different bulk-fill composite materials.

### Materials and Methods

#### Sample Preparation

A total of 40 human 3rd molar teeth were used in the study. The teeth had no restorations, defects, cracks, or decay and had been extracted for periodontal or surgical reasons. Approval to use these teeth in the study was granted by the Ethics Committee of Dicle University, Faculty of Dentistry (Decision no:2017/26). After cleaning all the hard and soft tissue remnants from the teeth, they were kept in distilled water at room temperature (25˚C) before and after the preparation. Using a high revolution diamond fissure burr (KG Sorensen, São Paulo, Brazil) under abundant water cooling, standard class II cavities were opened to be 2.5 mm deep, 4 mm wide, and with a mesial mass depth of 4 mm. These burrs, which have standard active tips and vertical stopper, were changed after each three cavity preparations, and the accuracy of the cavity dimensions was checked with digital calipers and periodontal probe at the end of the preparation, and thus standardization has been achieved.
To provide standardization of all the prepared cavities, the same adhesive material (G-aenial Bond (GC, Tokyo, Japan)) was applied with a brush. After each bonding procedure, the cavities were lightly dried with air and polymerized using an LED light source (Elipar, 3 M ESPE, St. Paul, MN, USA) for 10 secs. After completion of the stages of adhesive material application, the samples were randomly separated into four groups, each to be applied with a different bulk-fill composite material, placed in accordance with the manufacturer’s instructions. Bulk-fill composites that are frequently used by clinicians were preferred for the study. Group 1 was applied with FTK, Group 2 with X-tra Fil, Group 3 with TEC, and Group 4 with FLO. The components of the composite resins and the manufacturers are shown in Table 1.

### Forming the Restoration and the Micro-CT Scanning and Analysis

All the teeth were placed in a high-resolution micro-CT device (Skyscan 1172, Bruker, Kontich, Belgium) immediately after cavity preparation. Each sample was rotated 360˚ and before each scan, air calibration of the detector was performed. Approximately 1 hour after the scan, the first analyses of the images of each tooth were made. Before polymerization, the samples were kept in the dark and were placed in the micro-CT device for the second imaging. As the internal chamber of the device is dark, there was no shrinkage in the material. The micro-CT device was fixed at operating energy 100 Kvp, 100µA, cameral pixel size 9µm, and rotation angle 2˚ for all the samples. Images were recorded at 1000x 1000-pixel resolution.

After the second scanning, all the teeth were polymerized with the LED light source for 40 secs. Then the samples were placed in the micro-CT device again for the third image and evaluation of volume change (Fig. 1). Since the radiodensity of the tooth and resin composite was similar, 3 scan images were superimposed. Thus, possible scattering was prevented. After the final images were obtained, micro-CT analyses were made of all the samples. Percentage of the volumetric gap formed after polymerization was calculated using the volumes before and after polymerization. In this analysis, the micro-CT CTAn (ver.1.16.1.0, SkyScan, Aartselaar, Belgium) software was used. First, the region to be analyzed was determined, then 3-D imaging was performed to determine the accuracy of the selected region. The volumetric differences between before and after polymerization of the samples related to these images were calculated (Fig. 2).

### Statistical analysis

Statistical evaluation of the volumetric changes associated with polymerization shrinkage was made using the Kruskal Wallis and Mann Whitney U-tests. Analysis of the data was carried out with IBM SPSS Version 22 (IBM SPSS Inc., Armonk, NY, USA). p<0.05 was considered statistically significant in all tests.

### Table 1. The components of the composite resins used in the study and the manufacturers

<table>
<thead>
<tr>
<th>Bulk-fill composite resin</th>
<th>Components</th>
<th>Manufacturer</th>
<th>Filler %wt</th>
</tr>
</thead>
<tbody>
<tr>
<td>FTK (Posterior)</td>
<td>AUDMA UDMA 1.12-dodecane-DMA, Silica filler, Zirconia filler Zirconia/silica cluster filler, ytterbium trifluoride filler</td>
<td>3M ESPE, St. Paul, MN, USA</td>
<td>76.5</td>
</tr>
<tr>
<td>X-tra Fil</td>
<td>BIS-GMA, UDMA, TEGDMA Barium, Boron, Aluminosilicate glass</td>
<td>Voco GmbH, Cuxhaven, Germany</td>
<td>86</td>
</tr>
<tr>
<td>TEC</td>
<td>Dimethacrylates, prepolymer, bariumglass filler, ytterbium trifluoride, mixed oxide, additive, initiators, stabilizers, pigments</td>
<td>Ivoclar Vivadent, Schaan, Liechtenstein</td>
<td>78</td>
</tr>
<tr>
<td>FLO</td>
<td>AFM, AUDMA, UDMA, and 1, 12-dodecane-DMA</td>
<td>3M ESPE, St. Paul, MN, USA</td>
<td>76.5</td>
</tr>
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</table>

*BIS-GMA, bisphenol A glycidyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.
Figure 1. The schematized version of the study steps, cavity preparation, followed by the first micro-CT scan, the cavity was filled with composite, and the second micro-CT scan was performed. The composite was polymerized with the LED light source for 20 sec, and a third micro-CT scan was taken.

Figure 2. Micro-CT image of the change in volume of caused by polymerization shrinkage of FTK, XTF, TEC, FLO composites, respectively
Results

The differences in bulk-fill composite resin volume from before polymerization to after polymerization were found to be statistically significant. The volume of the gap formed associated with shrinkage after polymerization of the resins was not found to be significant in any of the four different composites according to the Kruskal Wallis test (Table 2).

According to the Mann Whitney U-test, when the groups were compared in pairs, there were determined to be significant differences before polymerization between FTK and TEC ($Z=2.797$ ($p=0.005$)) and between FTK and FLO ($Z=2.797$ ($p=0.004$)). After polymerization, statistically significant differences were determined between FTK and TEC ($Z=2.948$, $p=0.002$) and between FTK and FLO ($Z=2.797$, $p=0.004$).

The volumetric difference (%) between the resin and the dental tissue following polymerization was seen to be greatest in XTF and least in FTK (Table 3).

### Table 2. Average values of the volumes of the materials before and after polymerization (mm$^3$)

<table>
<thead>
<tr>
<th></th>
<th>N</th>
<th>Mean</th>
<th>Std. Deviation</th>
<th>95% Confidence Interval for Mean</th>
</tr>
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<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Lower Bound</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Before polymerization</td>
<td>40</td>
<td>43.0488</td>
<td>9.30883</td>
<td>40.0716</td>
</tr>
<tr>
<td>FTK</td>
<td>10</td>
<td>48.8020</td>
<td>7.55416</td>
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<td>46.6760</td>
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<tr>
<td>FLO</td>
<td>10</td>
<td>38.8890</td>
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</tr>
<tr>
<td>Total</td>
<td>40</td>
<td>43.0488</td>
<td>9.30883</td>
<td>40.0716</td>
</tr>
<tr>
<td>After polymerization</td>
<td>40</td>
<td>41.7125</td>
<td>9.06712</td>
<td>38.8126</td>
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<tr>
<td>FTK</td>
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<tr>
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<tr>
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<td>34.7447</td>
</tr>
<tr>
<td>Total</td>
<td>40</td>
<td>41.7125</td>
<td>9.06712</td>
<td>38.8126</td>
</tr>
</tbody>
</table>

### Table 3. Evaluation as a percentage of the volumetric gap formed after polymerization

<table>
<thead>
<tr>
<th></th>
<th>N</th>
<th>Mean</th>
<th>Std. Deviation</th>
<th>95% Confidence Interval for Mean</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Lower Bound</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FTK</td>
<td>10</td>
<td>2.4795</td>
<td>.86753</td>
<td>1.8589</td>
</tr>
<tr>
<td>XTF</td>
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<td>4.1501</td>
<td>3.68761</td>
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</tr>
<tr>
<td>TEC</td>
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<td>.85866</td>
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<tr>
<td>FLO</td>
<td>10</td>
<td>3.0452</td>
<td>1.70974</td>
<td>1.8222</td>
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<tr>
<td>Total</td>
<td>40</td>
<td>3.1518</td>
<td>2.13148</td>
<td>2.4701</td>
</tr>
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</table>

Discussion

The aim of this study is to evaluate volumetric gap and the amount of shrinkage that occurs after polymerization of different bulk-fill composites using micro-CT. A direct correlation between polymerization stress, polymerization shrinkage and marginal gap has been reported in many studies (6,24,25). As a result of this study, it was observed that a volumetric gap was formed in each bulkfill composite, but there was no statistically significant difference between the polymerization shrinkage of the materials.

Conventional methods such as microscopic evaluation can be used to measure the volumetric gap that occurs between the material and dental tissues. However, the fact that this method contains time-consuming steps such as cutting and staining of samples and being destructive makes it difficult to use (26,27). In recent years, micro-CT has been used to image the volumetric gap formed associated with polymerization shrinkage. This imaging method allows reconstruction of the tissues around the restoration and the tooth. The
advantage of this method is that it allows non-destructive and quantitative measurements while obtaining 3D images with the help of suitable programs. It also helps to ideally determine the localization, type, and volume of the resulting gap. However, this method is very time-consuming and expensive (20,26). Studies have shown that micro-CT is successful in detecting gaps between restoration and tooth and can be used for such evaluations (14). In this study, micro-CT was used to evaluate the volumetric gap.

One of the most important problems related to composite materials is the volumetric shrinkage that is formed during the transformation of monomers to polymers (28). Several problems occur related to this, such as microleakage, the formation of secondary caries, postoperative sensitivity, and bonding failure associated with the formation of stress between the restoration and the tooth (29,30). The main factors affecting the polymerization shrinkage of composite resins are the filler content of the composite resins, the chemical structure of monomers, the shape and depth of the cavity, the technique of composite placement, and light polymerization. In recent years, new composites have been developed to reduce polymerization shrinkage stresses. The most important property of these resins, known as bulk-fill, is the higher degree of polymerization than traditional composites because of the development of translucent structures. This allows the composites to be placed in the cavity in a greater mass (31,32). In addition to the low volume of shrinkage shown by these resins, which can be placed in a single layer of up to 4-6mm, there are several advantages such as ease-of-application, high resistance to wear, and color compatibility (33). The use of different bulk-fill composites was selected in the current study because of the good surface properties.

Some of the studies have shown that bulk-fill composites with high filler ratios exhibit the most advantageous shrinkage strength. In composite resins with high filler content, the amount of organic matrix is less, and consequently, the number of reactive methacrylate groups is reduced. This results in lower polymerization shrinkage and hence lower shrinkage stress development (34,35). In this study, the polymerization shrinkage of Tetric Evo Ceram composite is less than Filtek One can be explained by the high filler ratio. However, some studies have shown that not only the amount of filler but many other factors are effective in polymerization shrinkage (33,36).

A modified molecular weight urethane dimethacrylate (AUDMA-aromatic urethane dimethacrylate) was added to the Filtek Posterior composition. Although manufacturers have used this monomer to reduce polymerization shrinkage, some studies have shown that composite resins containing urethane dimethacrylate form more interface cavities (37,38). In this study, the minimum volumetric gap was found in Filtek Posterior with AUDMA. These results also support manufacturers.

Another property of composite resins is the formation of air pockets in the material associated with more manipulation during condensing. Some studies have shown that spaces can be formed when placing a composite in layers on top of each other, and the gaps formed can have a negative effect on the torsion strength of the composite (39). Demirel et al. used a horizontal technique in a study that examined the gaps formed in bulk-fill composites in class II cavities with micro-CT. In the current study, the standard technique was used (40).

The C-factor is defined as the ratio of unbonded surfaces to bonded cavity surfaces in composite resin restorations, and as the C-factor increases so shrinkage stress increases. In a previous study, it was shown that polymerization shrinkage in class I restorations was less than in class II restorations (41). Other studies have reported a direct association between polymerization stress shrinkage and a marginal gap (42). Less polymerization shrinkage is expected in class II restorations as there are more free surfaces that can undergo deformation. Cavities with a high C-factor show higher shrinkage stress, and this has a negative effect on mechanical properties.

In a study by Ersen et al., all the bulk-fill composites examined showed shrinkage, but there was seen to be a statistically significantly greater loss of volume in Filtek and SDR than in Tetric Evo Ceram (7). In the current study, the volumetric difference between the resin and the tooth tissue after polymerization was seen to be similar to that reported in other studies, with the greatest loss seen in X-tra Fil and the least in Filtek Posterior. Garcia et al. examined the polymerization of bulk-fill composite resins and the depth of polymerization. Polymerization shrinkage was reported as 3.43% in Filtek Ultimate resin and 3.57% in SDR (32). This difference in polymerization shrinkage is thought to be due to differences in the organic matrix of the materials.

The use of adhesive affects volume loss to a significant extent. Under normal conditions, in restorations made without the use of adhesive, resin composites can be bound to the cavity surfaces as the surfaces are irregular (43). However, not using adhesive increases polymerization shrinkage. In a study on this subject by Algamaiah et al., the use of adhesive was found to reduce polymerization shrinkage of bulk-fill composites by 13%. Furthermore, in studies using Tetric Evo Ceram bulk-fill (3.65%), Filtek Flowable bulk-fill (3.78%), and SDR, the greatest shrinkage was seen in SDR (44). Another study reported a high level of polymerization shrinkage, and this decreased with increasing depth (22).

Conclusions

The results of this study, which used micro-CT to evaluate the volumetric change that formed associated with polymerization shrinkage in bulk-fill composite resins, were as follows:

1. A volumetric gap was seen to form associated with polymerization shrinkage in all the bulk-fill composite resins used in this study.
2. The greatest volumetric difference was seen in X-tra Fil resin and the least in Filtek Posterior.

3. As there are few studies that have been conducted with micro-CT, which is a three-dimensional detailed imaging method, there is a need for further studies on this subject.

Ethical Approval: Ethics committee approval was received for this study from Dicle University, Faculty of Dentistry Ethics Committee in accordance with the World Medical Association Declaration of Helsinki, with the approval number: 2017/26.

Peer-review: Externally peer-reviewed.


Conflict of Interest: No conflict of interest was declared by the authors.

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References


