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Fatigue stipulation of bulk-fill composites: An in vitro appraisal

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ABSTRACT

Objectives. The aim of this study was to determine the Weibull and slow crack growth (SCG) parameters of bulk-fill resin based composites. The strength degradation over time of the materials was also assessed by strength-probability-time (SPT) analysis.

Methods. Three bulk-fill [Tetric EvoCeram Bulk Fill (TBF); X-tra fil (XTR); Filtek Bulk-fill flowable (BFL)] and a conventional one [Filtek Z250 (Z250)] were studied. Seventy five disk-shaped specimens (12 mm in diameter and 1 mm thick) were prepared by inserting the uncured composites in a stainless steel split mold followed by photoactivation (1200 mW/cm^2/20 s) and storage in distilled water (37 °C/24 h). Degree of conversion was evaluated in five specimens by analysis of FT-IR spectra obtained in the mid-IR region. The SCG parameters n (stress corrosion susceptibility coefficient) and σ_{f0} (scaling parameter) were obtained by testing ten specimens in each of the five stress rates: 10^{-2}, 10^{-1}, 10^{0}, 10^{1} and 10^{2} MPa/s using a piston-on-three-balls device. Weibull parameter m (Weibull modulus) and σ_{f0} (characteristic strength) were obtained by testing additional 20 specimens at 1 MPa/s. Strength–probability–time (SPT) diagrams were constructed by merging SCG and Weibull parameters.

Results. BFL and TBF presented higher n values, respectively (40.1 and 25.5). Z250 showed the highest (157.02 MPa) and TBF the lowest (110.90 MPa) σ_{f0} value. Weibull analysis showed m (Weibull modulus) of 9.7, 8.6, 9.7 and 8.9 for TBF, BFL, XTR and Z250, respectively. SPT diagram for 5% probability of failure showed strength decrease of 18% for BFL, 25% for TBF, 32% for XTR and 36% for Z250, respectively, after 5 years as compared to 1 year.

Significance. The reliability and decadence of strength over time for bulk-fill resin composites studied are, at least, comparable to conventional composites. BFL shows the highest fatigue resistance under all simulations followed by TBF, while XTR was at par with Z250.

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1. Introduction

Bulk-fill resin composites were introduced with claims to save restorative procedure time by enabling up to 4-mm thick placements that can be photopolymerized in one step. As increment size variability greatly depends on the skill level of clinicians [1], these materials can possibly overcome problems related to traditional layering techniques such as the inclusion of voids or contamination between layers [2].

The physical, chemical and mechanical properties of bulk-fill resin composites have been extensively studied. These materials may present degree of conversion (DOC) as high as high as 76–86% at 1 mm reaching up to 64% at 4 mm depth similar to conventional resin composites in the 55–60% range at 1 mm [3–7]. The marginal quality to enamel and dentin as well as internal dentin adaptation of bulk-fill resin composites has been found to be similar to conventional composites [8,9]. Some low-viscosity bulk-fill resin composites however, present less gap-free marginal interface and compromised internal adaptation to dentin [10,11]. Moreover, bulk fill resin composites present nanoindentation and bulk compressive creep comparable to conventional resin composites [12,13]. Conversely, the elastic modulus and flexural strength of bulk-fill resin composites vary as a function of the resin formulation and filler type and loading, making it impossible to generalize for the entire class of material. The elastic modulus of bulk-fill resin composites, can vary from 3.3 to 9.4 GPa [7]. Higher elastic modulus were reported for some bulk-fill composites (~15 GPa) but the values obtained were lower than the ones measured for conventional resin composites (up to 20 GPa) [14].

Though some studies have not found discrepancy in the flexural strength while comparing only bulk-fill resin composites [5,15], others have reported significant discrepancies when comparing various bulk-fill resin composites and conventional ones [7,16]. Fracture strength alone cannot predict structural failure as it provides only insight into the stresses that the material will withstand for a given flaw size distribution [17]. Alternatively, Weibull distribution, however, takes into consideration the scatter in strength measurements to describe the reliability of materials (i.e. stress required to fracture a given percentage of specimens) as they are scaled-up in size (larger volume or surface under stress) [17–19]. The Weibull modulus (m) is a dimensionless material-specific parameter that describes the variability of strength of brittle materials. Since it is inversely related to the standard deviation in a normal distribution, high Weibull modulus relates to higher reliability of materials [20]. The second parameter, characteristic strength (σ_0), is a location parameter that corresponds to the stress level for a 63.2% probability of failure [19]. Since it is related to the fracture strength of a material, it may vary with specimen geometry and test set-up [17,20]. The m values for some bulk fill composites can fluctuate from 10.4 up to 26.7 [5,21].

Characterization of a material's fracture resistance is important for screening, however, because that property is generally determined under static or quasi-static loading and it may not be representative of the material's strength when in function [5,7,22,23]. This is especially true for dental applications since, in clinical situations, direct and indirect restorative materials are subjected to cyclic masticatory forces under a corrosive environment that ultimately may lead to strength degradation [18,23–27]. In fact, resin composites do experience fracture strength degradation while defying fatigue scenarios [26]. The flexural strength of composites can decrease up to 27% after being stressed under rotating fatigue [22]. Likewise, dramatic strength degradations ranging from 32% up to 64% were observed for resin composite subjected to a flexural fatigue regimen of 10^4 cycles [28,29]. Similar strength degradation (37% up to 67%) were reported by Lohbauer *et al.* for resin composites after a fatigue challenge of 10^5 cycles [30].

The strength degradation over time is related to the material's susceptibility to slow crack growth (SCG) [17]. The SCG parameters n (stress corrosion coefficient) and σ_{f0} (scaling parameter) can be obtained by the dynamic fatigue test. This methodology relies on mathematical relationships among fracture resistance of specimens tested at different stress rates [18,26,27,31,32]. Usually, n values ranging from 5 to 30 indicate a high susceptibility to strength degradation under corrosive environment over time. For resin composites, n values ranging from 7 to 34.7 have been reported [26,32].

The objective of this study was to determine the strength (m and σ_0) and SCG (n and σ_{f0}) parameters of bulk-fill composite materials. In addition, the reliability degradation of the materials over time was assessed by the analysis of a strength–probability–time (SPT) diagram. The null hypothesis to be tested is that bulk-fill resin composites present similar Weibull and SCG parameters to the conventional resin composite tested.

2. Materials and methods

The three bulk-fills and one conventional resin composite used in this study are listed in Table 1. Disk-shaped specimens (12 mm in diameter and 1.0 ± 0.1-mm thick) were prepared by inserting the uncured composites in a stainless steel split mold. The top surface was covered with a Mylar strip. A glass slide was placed over the mold and manual pressure was applied to extrude excess material. The glass slide was removed and the material was photoactivated for 20 s at 1200 mW/cm^2 (Elitedent Q-4, Elitedent Enterprise Inc, USA). The tip of the curing light was kept 2 mm from the composite by a spacer to standardize curing distance. The specimens were subsequently removed from the mold and stored in distilled water at 37 °C for 24 h prior to testing.

The degree of conversion (DOC) was assessed to confirm the efficacy of the polymerization method. Five specimens were dry stored for 24 h at 37 °C and the FT-IR spectrum was measured at the bottom of the specimens in mid-IR region (IFS66 v/S, Bruker, USA) equipped with universal ATR sampling accessory (MIRacle, PIKE, USA) under the following conditions: 4 cm^{-1} resolution and 138 scans per spectrum. DOC was obtained by measuring the difference in the ratio of the absorbance strength of the vinyl peak at 1638 and a reference peak at 1608 cm^{-1} corresponding to aromatic absorption before and after photoactivation according to previously reported [3].