Abstract: The purpose of this study was to determine the depth of cure (DOC) of three resin-based composites (RBCs) using varying irradiance exposures with a corded light-emitting diode curing unit. DOCs for Filtek Z250, TPH Spectra, and Tetric EvoCeram Bulk Fill were determined using the International Organization for Standardization (ISO) Standard 4049. The RBCs were light-polymerized using three different power modes and manufacturer-recommended curing times. Irradiance was determined using a spectrometer sensor and the total energy density was calculated for each power mode and concomitant polymerization time. The DOC data were analyzed with a two-way analysis of variance and Tukey’s post hoc test. Tetric EvoCeram Bulk Fill produced significantly greater DOCs than TPH and Z250 ($P < 0.05$) for all three power mode settings. Overall, the DOC of Tetric EvoCeram Bulk Fill was greater than those of TPH and Z250 at all power settings, but the individual RBCs did not show a significant DOC difference among the three power settings ($P > 0.05$).

Keywords: polymerization; resin composite; dental materials.

Introduction

Visible-light photoinitiated resin-based composites (RBCs) are typically marketed with manufacturer guidelines for irradiance and resultant depth of cure (DOC). Depending on the type of light source and power output, light polymerization times for RBCs typically range from 10-40 s (1). The composition of modern-day RBCs usually includes an organic resin matrix, inorganic fillers, a coupling agent, inhibitors, a photoinitiator, and various pigments for color and opacity. The clinical performance of RBCs is largely determined by proper light polymerization. Undercured RBCs in deep preparations may lead to inferior cured material properties, resulting in water sorption, poor wear resistance, compromised adhesion, and decreased flexural strength (2). It is therefore extremely important to effectively cure RBCs to ensure that light polymerization results in an optimal clinical performance.

The International Organization for Standardization (ISO 4049) (3) has published guidelines for determining DOC of polymer-based restorative materials. Determining the DOC of a RBC using a light-emitting diode (LED) curing unit is important for optimal, predictable practices in the clinical setting. Improvement in the curing characteristics has the potential to provide a more efficient time management by clinicians, as it would not only decrease the amount of time required for light to polymerize the RBC but also allow for potentially greater increment depths of RBC to be placed at one time. This is precisely the claim that many manufacturers of newer bulk-fill RBCs make. They are marketed to allow the practitioner to save time by avoiding the standard 2-mm
incremental buildup procedure. Many bulk-fill RBCs available on the market are claiming an additional 4-mm DOC, sparing the patient and practitioner chair time (4). However, bulk-fills have yet to stand the test of time, and many practitioners are reluctant to adopt their regular clinical use without research and established clinical results backing up manufacturers’ claims.

Published studies provide mixed results concerning DOC measurement, time requirements for light polymerization, and the role of shade and/or translucency in effective polymerization (5-9). Understanding and employing optimal DOC parameters may also allow the patient to experience greater comfort through limited exposure to the curing unit and associated energy and heat transfer (10).

LED-curing units are relatively recent compared with quartz-tungsten-halogen (QTH) polymerization units (11). Efficiency and effectiveness of LED-curing units are showing comparable results to QTH units (6,12). Considering irradiance values of curing units, QTH tend to emit less than 1,000 mW/cm², whereas LED routinely demonstrates values over 1,000 mW/cm², with some manufacturers claiming values as high as 5,000-6,000 mW/cm² (13,14). Additionally, modern LED-curing units usually have average irradiance values above those that traditional handheld spectrometers can accurately measure (13). To ensure that a RBC is fully cured, a clinician must be able to accurately measure irradiance, as the relationship between the degree of RBC conversion and the amount of light exposure exhibits a reciprocal pattern (15), while DOC and the amount of light exposure exhibit a linear pattern (16-19).

Based on these observations, a laboratory study was designed to assess the DOC of light-polymerized RBCs with an LED-curing unit under varying irradiance exposures. The purpose of the study was to analyze RBC polymerization at different recommended light settings and ideally determine the appropriate setting for RBC light polymerization with an LED-curing unit.

The study’s null hypotheses were that 1) the RBC DOC is not the same using three different recommended power settings for light polymerization, and 2) that there is no difference in DOC between RBC materials at the same power setting.

Materials and Methods

Specimen preparation
Three RBCs (Shade A2) were evaluated in this laboratory study as follows: 1) TPH Spectra (Dentsply Sirona, Milford, DE, USA), 2) Filtek Z250 (3M Oral Care, St. Paul, MN, USA), and 3) Tetric EvoCeram Bulk Fill (Ivoclar Vivadent, Schaan, Liechtenstein). A unidose applicator gun (3M Oral Care) was used to dispense RBCs. In accordance with ISO Standard 4049, a stainless-steel mold with 4-mm diameter and 15-mm length was used. A mylar strip over a glass slide was placed on the bottom opening of the stainless-steel mold. To limit air incorporation, a dental condenser was used to insert RBCs into the mold. RBCs were slightly overfilled on the top surface of the mold. A mylar strip and glass plate were placed on the top of the mold and pressed to displace excess material. The glass slide was then removed, and the specimens were light-polymerized with a corded LED-curing unit (VALO, Ultradent, South Jordan, UT, USA) placed directly onto the mylar strip and activated in that position. The polymerized RBC materials were removed from the stainless-steel mold, and the uncured resin portion scraped away with a blunt spatula according to ISO Standard 4049. The length of the hardened resin was measured in millimeters using a Westward Digital Caliper (Model #2YNC6; Grainger, Lake Forest, IL, USA), and recorded. The DOC was then determined according to the ISO Standard 4049 by dividing the hardened light-polymerized length of the RBC by two.

Each RBC underwent three test conditions with the LED-curing unit: Standard Power Mode, High Power Mode, and Xtra Power Mode. A checkMARC spectrometer sensor (BlueLight Analytics, Inc., Halifax, Canada) was used to measure irradiance of the curing unit prior to testing (Table 1).

Ten samples of each RBC were prepared for each of the three power modes. The curing times and number of cycles for final cure (Table 2) were based on manufacturer-recommended curing times for optimal LED-curing unit results. The curing unit was set so that light would automatically shut off after the programmed polymerization time was completed. If the mode used required more than one cycle (Table 2), the curing unit was manually turned on immediately after the previous cycle and repeated until the number of cycles was completed.

Scanning Electron Microscopy observation
Ultrastructural observations were conducted on the polished surfaces of the tested RBCs after argon-ion etching. Three specimens per group were observed using field-emission scanning electron microscopy (SEM) (ERA 8800FE; Elionix, Tokyo, Japan). The RBC surfaces were wet ground with a sequence of silicon carbide papers (#600, #1200, #2000, and #4000 grit) using a grinder/polisher (Ecomet 4; Buehler, Lake Bluff, IL, USA). The surfaces were then polished with abrasive discs (Fuji Star Type DDC; Sankyo-Rikagaku, Okegawa, Japan).
followed by a series of diamond pastes down to 0.25-µm particle size (DP-Paste; Struers, Ballerup, Denmark) to bring the surfaces to a high gloss state. SEM specimens of polished surfaces were dehydrated by immersion in aqueous tert-butanol solutions with ascending concentrations (50% for 20 min, 75% for 20 min, 95% for 20 min, and 100% for 2 h) and were subsequently transferred to a critical-point dryer (Model ID-3; Elionix) for 30 min. These polished surfaces were etched for 30 s using an argon-ion beam (EIS-200ER; Elionix) directed perpendicular to the surface at an accelerating voltage of 1.0-kV and an ion current density of 0.4 mA/cm² to enhance visibility of the filler particles. The surfaces were coated with a thin gold film in a vacuum evaporator (Quick Coater SC-701; Sanyu Electron, Tokyo, Japan). SEM observations were carried out using a 10-kV operating voltage.

Statistical Analysis

Resulting DOC data were analyzed with the Shapiro-Wilk test to evaluate distribution normality and with the Levene test to evaluate equality of variance (α = 0.05). After checking for normality and equality of variance, mean DOC value testing was conducted using a two-way analysis of variance (ANOVA); Factors: 1) RBC and 2) power mode), followed by Tukey’s post hoc test (Systat 11; Systat Software, Inc., Richmond, CA, USA).

Results

The Shapiro-Wilk test did not reject the null hypothesis of a normal distribution of data, and the Levene test showed equal variance with similar scatter across all samples. The DOC results for the three RBCs light-polymerized with three different power modes using a corded LED-curing unit are presented in Table 3. The two-way ANOVA (Table 4) revealed a significant effect associated with RBC (F = 224.54; P < 0.001), power mode (F = 4.45; P = 0.015), and interaction of RBC and power mode (F = 2.77; P = 0.033) factors.

DOC using three curing power modes ranged from 2.69 (0.14) to 2.87 (0.12) mm for TPH, from 3.04 (0.07) to 3.15 (0.26) mm for Z250 DOC, and from 3.40 (0.09) to

| Table 1 LED-curing unit irradiance |
|-----------------------------------|
| Power mode LED-curing unit       | Manufacturer rated irradiance (mW/cm²) | Measured irradiance (mW/cm²)* | Percent difference |
|-----------------------------------|----------------------------------------|-------------------------------|--------------------|
| Standard                          | 1,000                                  | 823                           | 17.7               |
| High                              | 1,400                                  | 1,288                         | 8.0                |
| Xtra                              | 3,200                                  | 2,460                         | 23.1               |

*checkMARC, BlueLight Analytics, Halifax, Canada

| Table 2 Corded LED-curing unit’s power output (9.6-mm lens diameter) |
|------------------------------------|
| Power mode (settings) | Exposure time (s) | Number of cycles | Total exposure time (s) | Irradiance (mW/cm²) | Total energy density (J/cm²) |
|------------------------|-------------------|------------------|------------------------|---------------------|----------------------------|
| Standard               | 20                | 1                | 20                     | 823                 | 16.6                       |
| High                   | 4                 | 3                | 12                     | 1,288               | 15.6                       |
| Xtra                   | 3                 | 2                | 6                      | 2,460               | 14.8                       |

| Table 3 Depth of cure (mm; mean and standard deviation) |
|-------------------------------------|
| Power mode | Resin Composite |
|------------|-----------------|
|            | TPH             | Z250            | Tetric EvoCeram Bulk Fill |
| Standard   | 2.79 (0.08) aA  | 3.15 (0.26) aB  | 3.53 (0.08) aC           |
| High       | 2.87 (0.12) aA  | 3.04 (0.07) aA  | 3.53 (0.12) aB           |
| Xtra       | 2.69 (0.14) aA  | 3.10 (0.06) aB  | 3.40 (0.09) aC           |

Same small case letters in column for each RBC indicate no difference at the 5% significance level. Same capital case letters in row for same curing mode for the RBCs indicate no significant difference at the 5% significance level.

| Table 4 Two-way analysis of variance |
|--------------------------------------|
| Source | Sum-of-squares | df | Mean-square | F-ratio | P       |
|--------|----------------|----|-------------|---------|---------|
| RBC    | 7.419          | 2  | 3.709       | 224.541 | <0.001  |
| Power Mode | 0.147 | 2  | 0.074       | 4.452   | 0.015   |
| RBC Power Mode | 0.183 | 4  | 0.046       | 2.770   | 0.033   |
| Error  | 1.338          | 81 | 0.017       |         |         |
3.53 (0.12) mm for Tetric EvoCeram Bulk Fill (Table 3). Tukey’s post hoc test did not show a significant difference ($P > 0.05$) in DOCs for individual RBCs when the three power modes were compared. Tetric EvoCeram Bulk Fill produced significantly greater DOCs than TPH and Z250 ($P < 0.05$) for all three power mode settings. Overall, the DOC for Tetric EvoCeram Bulk Fill was greater than for TPH and Z250 at all power settings (Table 3), but the individual RBCs did not show a significant DOC difference among the three power settings ($P > 0.05$).

Representative SEM images of polished RBC surfaces with argon-ion etching are shown in Figs 1-3. The argon-ion etching revealed clear differences in filler particle size, shape, and distribution. RBCs exhibited a variety of filler particle sizes and shapes. SEM images of TPH showed a wide size range (<1 to 4 µm) of irregular particles. SEM images of Z250 showed a wide size range (<1 to 3 µm) of spherical particles. SEM images of Tetric EvoCeram Bulk Fill showed small spherical particles (<1 µm) and a wide size range (<1 to 2 µm) of irregular particles.

**Discussion**

LED-curing units have become very popular in clinical practice due to ease of use and time sparing for curing RBCs. The manufacturers’ recommendations for LED-curing units provide guidance to clinicians for radiant exposure to optimize light polymerization while reducing clinical chairside time. In the current study, a newly introduced LED-curing unit was evaluated for light polymerization of three RBC materials at power settings and exposure times recommended and provided by the manufacturer. Three power settings were used with
manufacturer-recommended curing times (Table 3), with each subsequent setting increasing in light irradiance and decreasing in total exposure time (Standard [20 s exposure]; High [12 s exposure]; Xtra [6 s exposure]). The average light irradiance to the RBC was calculated using a checkMARC spectrometer sensor (Table 2) and ranged from 14.8-16.6 J/cm² of radiant exposure to RBC surfaces. The total energy density for each of the three curing conditions used in this study was therefore similar.

The checkMARC spectrometer is accurate in measuring irradiance to within 2% of an Integrating Sphere, the gold standard in laboratory equipment (14). It is purportedly able to verify how well a curing unit performs when used intraorally at a distance of 0 and 6-mm from an RBC surface. Reports generated from the spectrometer manufacturer can provide useful information on minimum curing times, light-curing unit irradiance, and increment depths for various RBCs (www.checkmarc.net).

The results retrieved for each of the three RBCs did not show significant differences (P > 0.05) in DOC for the three curing conditions (Table 3). Therefore, the first null hypothesis that a RBC DOC is not the same using three different recommended power settings was rejected. The DOC between the three RBCs light-polymerized with the same curing conditions was significantly different (P < 0.05) for two of the three curing conditions. Therefore, the null hypothesis of no difference in DOC among RBC materials at the same power setting was partially rejected. However, it should be noted that it was not significantly different concerning curing condition (High, 12 s exposure), approaching significance (P = 0.094).

Based on the retrieved results, the DOC was different depending on the resin composite in most power modes (Standard and Xtra). The Tetric EvoCeram DOC was greater than both the Z250 and TPH DOCs in all three power modes. The difference in the curing characteristics of RBC materials may be related to the photoinitiators and/or filler particle composition, including shape, loading, shade, and opacity factors. In addition, from SEM observations, the filler particle size, shape, and distribution were different depending on the resin composite. This may be one of the reasons why DOC differed among resin composites. Photoinitiators of resin composite systems may have also played an important role in the resulting DOCs. Evidence using ISO Standard 4049 shows that Tetric EvoCeram Bulk Fill exhibited a greater DOC than both TPH and Z250, but caution suggests that increments of no more than 2.5-3.5 mm should be used in clinical settings to ensure an adequate DOC.

DOCs in this study were determined with stainless-steel molds according to ISO Standard 4049. While this data provides good comparisons in the laboratory, its relevance in the clinical setting is uncertain. Clinical DOCs in natural teeth may be greater due to light reflectance of natural tooth structures. Erickson and Barkmeier (20) recently published data from a study examining DOC of a light-polymerized RBC in stainless-steel, black, or white 6-mm cylindrical-shaped molds. The results suggested that DOCs are greater in white versus either black or stainless-steel molds. This may have significant clinical application since tooth structures may transmit and reflect light better than the typical metal molds used to assess DOC in the laboratory, and thus provide a better DOC for RBCs in the oral cavity. Similar studies using standardized natural tooth molds with additional bulk-fill and conventional RBCs are warranted to assess DOCs.

Conflict of interest
The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or companies that are discussed in this article.

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