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Research Article

Effect of Multiple and Single Emission Peak Light Emitting Diode Light Curing Units on the Degree of Conversion and Microhardness of Resin-Based Pit and Fissure Sealant

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Received: August 29, 2017; **Published:** September 20, 2017

Abstract

Objective: To assess a multiple emission peak light-emitting-diode (LED) light-curing unit (LCU) by measuring the polymerization efficiency through the degree of conversion (DC) and Knoop microhardness (KH) of a resin-based pit and fissure sealant at various light curing times and two distances compared to a single emission peak LED LCU.

Method: Sixty disks of a resin-based pit and fissure sealant (Delton, DENTSPLY, York, PA) samples (6 x 1 mm) were fabricated. Prepared specimens were polymerized using 10, 20 and 40 second curing times at 2 or 4 mm curing distances (n = 5/LCU/group). A Managing Accurate Resin Curing System-Resin Calibrator was used to ensure that irradiance delivered from the LED LCUs remained consistent throughout the study. Samples were stored at 37°C for one hour. Then, the DC (n = 3/surface) and KH (n = 5/surface) measurements were obtained on the top and bottom surfaces using an Attenuated Total Reflection-Fourier transform infrared spectroscopy (Pike analytics and Jasco) and a Knoop microhardness tester (Instron) utilizing 25 gm at 10 second dwell time, respectively. Multiple-way ANOVA was performed followed by Fisher's test ($\alpha = 0.05$).

Results: The multiple emission peak LED LCU displayed significantly higher DC (27.7 - 81.4 %) and KH (10.0 - 23.0 kg/mm^2) compared to the single emission peak LED LCU; DC (20.4 - 74.8%) and KH (4.8 - 19.6 kg/mm^2) when curing specimens at 2 and 4 mm curing distances assessed using 10, 20 and 40-second curing times. The 10 second cure at 4 mm showed significantly lower DC and KH values compared to the other times and distances tested.

Conclusion: The multiple emission peak LED LCU demonstrated significantly higher DC and KH compared to the single emission peak LED LCU when using a resin-based pit and fissure sealant assessed regardless of the curing distance or curing time.

Keywords: *Fissure Sealants; Resin-Based Material; Light Emitting Diode; Multiple and Single Emission Peak; Degree of Conversion; Knoop Microhardness*

Abbreviations

LED: Light Emitting Diode; LCU: Light Curing Unit; KH: Knoop Microhardness; DC: Degree of Conversion; PFS: Pit and Fissure Sealant

Introduction

Pit and fissure sealants (PFS) were introduced in the 1960s as a relatively low viscosity resin-based material. A pit and fissure sealant is mainly applied on the occlusal pits and fissures of caries susceptible teeth and then polymerized, either chemically (autopolymerizing) or by exposing it to visible light (light-cured). This will form a micromechanically bonded protective barrier that prevents the invasion of the pit and fissures by caries-producing bacteria and simultaneously cuts off the access from their source of nutrients [1]. PFS are classified into two types of sealant materials: resin-based sealants, and glass ionomer (GI) cement. Currently resin-based sealants are the most commonly used [2].

Resin-based sealants contain photoinitiators (either Camphorquinone (CQ) or alternative photoinitiators) that absorb the incoming photons from the light-curing unit (LCU) to initiate the polymerization reaction [3]. This light activation process depends on matching the spectral emission of the LCU with the requirements of the photoinitiator system to produce free radicals leading to the conversion of monomers into a polymer network. Thus, different types of light-emitting-diode (LED) LCUs were developed [4,5]. The single emission peak LED LCUs are limited to 420 - 490 nm output to match the narrow absorption peak of CQ (465 - 470 nm). Multiple emission peak LED LCUs have an additional irradiance peak near the ultraviolet region that extends to the violet visible light spectrum and encompasses the maximum absorbance of the alternative photoinitiators (395 - 410 nm) [6,7].

There are several direct methods to determine the degree of conversion (DC) of resin-based materials. Attenuated Total Reflection-Fourier Transform Infrared Spectroscopy (ATR-FTIR) has proven to be a reliable method to determine DC. It detects the C = C stretching vibrations directly before and after curing of materials [8]. The degree of conversion is a crucial parameter as it is significantly correlated to other important material characteristics such as mechanical properties. A high percentage of DC is required to achieve high hardness [9], flexural strength and wear resistance [10]. The microhardness test provides an indirect indication of degree of conversion by measuring the ratio of the top to bottom surface hardness values. The values should be no more than 20% different between the hardness values of the bottom and top surfaces [11].

Although placing a PFS may appear to be a simple procedure, it is very technique sensitive. Attention to placement details, such as proper tooth isolation and curing light position may diminish the need to repair/replace the PFS in the future. The physical properties of sealants after polymerization is suggested to have a direct implication on their long-term clinical success in the oral cavity. Therefore, the presence of the appropriate wavelengths and sufficient intensity to activate the photoinitiator in the sealant material is needed for a successful polymerization process. Moreover, the distance of the light curing tip to the material and exposure duration are critical to achieve sufficient DC, and this can be controlled by clinicians to some extent [12]. Thus, more scientific evidence is needed concerning the efficiency of polymerization of the commercial resin-based fissure sealant cured with single and multiple emission peak LED LCU. Exploring this area may help clinicians decide whether single or multiple peak LED LCUs should be used for an effective and predictable clinical performance for resin-based fissure sealants [13]. The aim of the present study was to assess a multiple emission peak LED LCU by measuring the polymerization efficiency through the DC and KH of a resin-based pit and fissure sealant at various light curing times and two different distances compared to a single emission peak LED LCU.

Materials and Methods

A laboratory study was conducted using an opaque resin-based pit and fissure sealant (Delton, DENTSPLY, York, PA). Two LCUs were evaluated, a multiple emission peak LED LCU (VALO, Ultradent, South, Utah) and a single emission peak LED LCU (FLASH LITE 1401, Discus Dental, Culver, CA) (Table 1).

Material/unit	Product name and manufacturer	Composition
Pits and fissures sealant	Delton, DENTSPLY, York, PA	Aromatic an alphatic dimethacrylate monomers Titanium Dioxide (opaque) Silicon Dioxide (Opaque) Initiators Stabilizers
Light Curing Unit	Single emission peak LED (FLASH LITE 1401, Discus Dental, Culver, CA)	Wavelength Range: 460-480 nm Light Intensity: ≥1100 mW/cm^2
	multiple emission peak LED LCU (VALO, Ultradent, South, Utah)	Wavelength Range: 395–480nm Light Intensity: Irradiance (mW/cm^2) Standard Power: 1000 mW/cm^2 High Power: 1400 mW/cm^2 Xtra Power: 3200 mW/cm^2

Table 1: *Details of the composition of resin-based sealant and light-curing units used in the study as described by the manufacturers.*

Specimen Preparation

A total of 60-disc samples were fabricated using a Delrin mold (6 mm x 1 mm) and divided into twelve groups (n = 5/group). The sealant material was injected into the mold sandwiched between Mylar strips and microscope slides to create a smooth surface and avoid air entrapment. The LCUs were fully charged before testing. A Managing Accurate Resin Curing System-Resin Calibrator (MARC-RC) system (Blue light Analytics Inc., Halifax, Canada) with 4-mm diameter top sensor was used each time before testing, to monitor the irradiance delivered from the LED LCUs throughout the study. A mechanical arm was used to mount both LCUs in the same position and distance during specimen light curing. Reference points on the MARC-RC system, the rims of LCUs and transparent guide template were used to standardize the positions of LCUs throughout the measuring process. The LCU light guide tip was placed in perpendicular position and centered to the MARC-RC top and bottom sensors, and on the top surface of the specimens. Each specimen was placed over the 4-mm diameter MARC-RC bottom sensor to measure the amount of light irradiance delivered to the bottom surface of the specimen. Equally positioned markings on four corners of the mold were created to standardize the location of the specimen in the MARC-RC. Specimens were cured for 10, 20, and 40 seconds at 2 or 4 mm distance between the light guide and top of the specimen. The specimens were not removed from the mold to standardize the DC and microhardness measurements using the markings on the mold. Specimens were covered with a moist paper towel and the lid of the container closed to maintain 100% relative humidity. The container was wrapped in aluminum foil to keep specimens away from the light and stored at 37°C for one hour in the incubator. After this, the DC test was performed, followed by the KH microhardness test. The test groups were randomized for specimen fabrication and testing. Only specimens from one group were fabricated and tested each day.

Degree of Conversion

The DC of the resin-based fissure sealant was measured by attenuated total reflection Fourier transform infrared (ATR-FTIR) spectroscopy (JASCO 4100 International Co., Tokyo, Japan) using a 1.8 mm diameter diamond crystal plate (ATR-FTIR MIRacleTM, Pike technologies, Madison, WI, USA). The absorbance was measured using 64 scans and 4 cm^{-1} resolutions. Three uncured resin-based fissure sealant specimens were measured. For the cured specimens (n = 5/group), three non-overlapped standardized measurements on the top and bottom surfaces were collected on each specimen; one on the upper half, lower right and lower left side of each specimen. Each cured specimen was placed on the diamond crystal plate and secured with a swivel pressure clamp to insure its adaptation. The DC was determined by measuring the intensity (or area) decrease of the methacrylate aliphatic (C = C) stretch absorption band at 1,637 cm^{-1} as the methacrylate monomer was converted to polymer. The aromatic band present at 1,607 cm^{-1} was used as an internal standard. The areas under the curves (1607 and 1637 cm^{-1}) of uncured and cured resin-based pit and fissure sealant were used to calculate the DC percent according to the following equation:

$$Degree\ of\ conversion = \left(1 - \frac{cured\,(area\ under\, 1637\ /\ area\ under\, 1607)}{uncured\,(area\ under\, 1637\ /\ area\ under\, 1607)}\right) \times 100$$

The average DC values were calculated for each surface tested.

Knoop Microhardness (KH)

On the same specimens, the KH test was performed. Five indentations were obtained on both the top and bottom surfaces of each specimen. Indentations were located in the upper, lower, left, right, and center of each test surface with the indentations 1 mm from the periphery and 2 mm between indentations. The indentation location was standardized according to the markings placed on the mold. The hardness testing was performed using a hardness tester (Leco LM247AT, MI, USA, software; Confident V 2.5.2), with a diamond indenter utilizing 25-gram load and 10 second dwell time. The average KH values were calculated for each surface.

Statistical Analysis

The effects of LED LCU type (multiple or single emission peak), curing times (10, 20 and 40 seconds), distance (2, 4 mm), and surface (top and bottom) on the DC and KH was examined using ANOVA. The ANOVA included fixed effects for the three factors and their interactions and a random effect to correlate the measurements from the top and bottom surfaces of the same specimen. Pair-wise comparisons were made using Fisher's Protected Least Significant Differences to control the overall significance level at 5%. A sample size of 5 for each group, for 80% power to detect a KH or DC difference of 1.8 between LED LCU types was determined.

Results and Discussion

In the present study, the radiant exposure values were higher at the 40 second exposure time than at the 20 and 10 second times (Tables 7, 8). Also, the DC of both LED LCUs was significantly lower at 4 mm than 2 mm within the 10 second curing time. Furthermore, the 10 second time for each LED LCU at 4 mm was significantly lower than 20 and 40 seconds. However, there was no significant difference on the top surfaces between the single and multiple emission peak LCUs that were tested (Table 2). Moreover, the results in (Table 3) displayed that there were significant differences in the DC at the bottom surfaces of specimens among the curing times and distances of each LCU. The DC of the single emission peak LED LCU was significantly lower than the multiple emission peak LED LCU at all curing times at 4 mm curing distances and only at 2 mm curing distance with a 20 second curing time.

	Multiple emission peak LED LCU		**Single emission peak LED LCU**	
	2 mm	**4 mm**	**2 mm**	**4 mm**
10 sec	78.6 (7.2) ^{Aa}	64.0 (11.3) ^{Bb}	77.6 (3.7) ^{Aa}	64.6 (11.0) ^{Bb}
20 sec	79.0 (7.9) ^{Aa}	83.8 (2.4) ^{Aa}	82.3 (2.6) ^{Aa}	79.6 (4.6) ^{Aa}
40 sec	84.0 (2.2) ^{Aa}	81.8 (5.3) ^{Aa}	85.2 (2.3) ^{Aa}	76.6 (7.8) ^{Ab}

Table 2: *Mean (standard deviation) for the degree of conversion values (%) of the top surface of the resin-based sealant specimens cured by each light-curing unit explored at the different curing distances and curing times.*

Different lowercase letters between different curing distances in each row and uppercase letters in the column between different curing times indicates statistically significant differences in each LCU.

	Multiple emission peak LED LCU		Single emission peak LED LCU	
	2 mm	4 mm	2 mm	4 mm
10 sec	45.4 (2.8) Ba	27.7 (4.9) Cb*	45.7 (3.5) Ba	20.4 (2.9) Cb
20 sec	77.9 (2.6) Aa*	76.4 (3.2) Ba*	71.5 (4.0) Aa	56.5 (6.4) Bb
40 sec	79.2 (4.2) Aa	81.4 (1.5) Aa*	74.8 (3.7) Aa	73.6 (1.9) Aa

Table 3: *Mean (standard deviation) for the degree of conversion values (%) on the bottom surface of the resin-based sealant specimens cured by each light-curing unit explored at the different curing distances and curing times.*

*Different lowercase letters between different curing distances in each row and uppercase letters in each column between different curing times indicate statistically significant differences in each LCU. *represents significantly different values between two LCUs at the specific time and distance.*

The KH results revealed that both LED LCUs were significantly lower at 4 mm than 2 mm within the 10 second time (Table 4). Furthermore, KH at 10 and 20 seconds for the multiple emission peak LED LCU at 2 mm was significantly lower than at 40 seconds, but at 4 mm, the 10 second curing time was significantly lower than 20 and 40 seconds. In the single emission peak LED LCU at both distances, 10 seconds was significantly lower than 20 and 40 seconds. In addition, the single emission peak LED LCU was significantly lower than the multiple emission peak LED LCU for all curing times and both distances. There were significant differences in hardness at the bottom surfaces of the specimens among both LCUs, curing times and various distances (Table 5). For the single emission peak LED LCU, KH was significantly lower than the multiple emission peak LED LCU within the 40 second curing time at both distances and the 20 second at 4 mm curing distance as well. Finally, the Bottom/Top (B/T) hardness ratios obtained were less than 0.8 for all irradiation protocols except the 40 second at 2 mm curing distance group for both curing units (Table 6). No microhardness values were obtained from the bottom surfaces for the specimen groups cured for 10 seconds at 4 mm distance.

	Multiple emission peak LED LCU		Single emission peak LED LCU	
	2 mm	4 mm	2 mm	4 mm
10 sec	20.0 (2.1) Ba*	14.9 (3.1) Bb*	17.6 (0.7) Ba	5.9 (1.9) Bb
20 sec	21.2 (1.0) Ba*	18.4 (2.1) Aa*	19.2 (3.2) Aa	16.6 (2.0) Aa
40 sec	23.0 (3.9) Aa*	21.5 (1.1) Aa*	19.6 (0.6) Aa	17.6 (2.1) Aa

Table 4: *Mean (standard deviation) Knoop microhardness values (KH, kg/mm^2) on the top surface of the resin-based sealant specimens cured by each light-curing unit explored at the different curing distances and curing times.*

*Different lowercase letters between different curing distances in each row and uppercase letters in each column between different curing times indicate statistically significant differences in each LCU. *represents significantly different values between two LCUs at the specific time and distance.*

	Multiple emission peak LED LCU		Single emission peak LED LCU	
	2 mm	4 mm	2 mm	4 mm
10 sec	4.9 (0.6) Ca	0.0 (0.0) Cb	5.2 (2.4) Ca	0.0 (0.0) Cb
20 sec	11.8 (1.7) Ba	10.0 (2.1) Ba*	10.0 (1.7) Ba	4.8 (3.0) Bb
40 sec	19.9 (1.4) Aa*	16.5 (2.2) Ab*	16.9 (1.4) Aa	10.3 (1.1) Ab

Table 5: *Mean (standard deviation) Knoop microhardness values (KH, kg/mm^2) on the bottom surface of the resin-based sealant specimens cured by each light-curing unit explored at the different curing distances and curing times.*

*Different lowercase letters between different curing distances in each row and uppercase letters in each column between different curing times indicate statistically significant differences in each LCU. *represent significantly different values between the two LCUs at the specific distance.*

	Multiple emission peak LED LCU		Single emission peak LED LCU	
	2 mm	4 mm	2 mm	4 mm
10 sec	0.24	0.0	0.29	0.0
20 sec	0.64	0.47	0.52	0.28
40 sec	0.87 *	0.76	0.86 *	0.58

Table 6: *Microhardness bottom/top ratios.*

**represent KH Bottom/Top more than 80%*

Two regulating bodies specifying requirements for many dental products are the American National Standards Institute/American Dental Association (ANSI/ADA) and the International Organization for Standardization (ISO). ANSI/ADA specification 39 for PFS requires a 0.75-mm depth of cure [14] whereas ISO specification 6874 requires a cure twice as deep at 1.5-mm [15]. In the clinical environment, PFS usually present up to a 1-mm thickness. The light tip of the curing unit may be placed at different distances from the sealant surface [16]. This is mostly dictated by the cusp size and the morphology of pits and fissures, which may lead to an increase in the light dispersion and decrease in the irradiance of the light that reaches the material. Therefore, both distance and specimen thickness were considered in the present study design to simulate clinical conditions [17]. The 2 and 4 mm curing distances were selected due to variations in accessibility, cusp size and shape of posterior teeth, as it may be difficult clinically to place the light tip at 0 mm distance over the resin-based materials surface. It is recommended that the distance should not exceed 3 mm to sufficiently cure a 2 mm layer of the composite material [18]. Previous studies found a difference when the distance was less than 4 mm [19] or 6 mm [20]. The control of distance by the clinician between the LCU light guide and the surface of the resin was reported by Price., *et al*. 2000 as a factor influencing the light intensity [21].

In the present experiment, specimens were kept in a dark environment at 37°C under 100% relative humidity for one hour to approximate clinically relevant conditions as heat energy may induce the decomposition of initiators into free radicals or direct excitation of monomer molecules [22].

Curing the resin-based fissure sealant with short curing times might be helpful to accelerate the process of placing the fissure sealant for pediatric patients, without jeopardizing the sufficient polymerization of the material. In the present study, the resin-based sealant was tested with a 10 second curing time for both LED LCUs to confirm that a short curing time would not adversely affect the physical and mechanical properties of the resin based fissure sealant. However, the 10 second curing time results showed that the mechanical and physical properties of resin sealant were inferior compared to resin sealant cured to 20 and 40 seconds. Moreover, from the results of the present study, a simple comparison of the difference in the values of DC and KH between the 10 second and 20 second exposure times showed higher differences than 20 second and 40 second exposure time values. This observation might be explained by the fact that the efficiency of the polymerization reaction is limited or reaches a saturated maximum state above which an increase in the irradiance or exposure time no longer leads to a significant increase in DC [23,24].

The radiant exposure values were higher at the 40 second exposure time than at 20 and 10 seconds (Tables 7, 8). Thus, the increase in exposure time caused an increase in radiant exposure, resulting in higher DC and KH values at the 40- second exposure time compared to 10 and 20 second on both the top and bottom surfaces. A previous study by A Peutzfeldt., *et al*. 2005 discussed that when the radiant exposure increases, the mechanical properties will be higher. Also, A Catelan., *et al*. 2014 stated that even without changes in the irradiance, the radiant exposure will be higher if exposure time is longer [24,25].

	Multiple emission peak LED LCU		Single emission peak LED LCU	
	2 mm	4 mm	2 mm	4 mm
10 sec	13.0 (0.3)	9.1 (0.1)	7.7 (0.5)	2.4 (0.0)
20 sec	26.3 (0.1)	18.2 (0.1)	15.3 (0.1)	7.2 (0.2)
40 sec	49.0 (1.0)	36.0 (0.1)	30.6 (0.2)	14.4 (0.1)

Table 7: *Light Emitting Diode Light Curing Units Mean (standard deviation) Radiant exposure – Top (J/cm^2).*

A KH B/T ratio is suggested to verify the efficiency of the cure in deep surfaces when compared to surfaces located closer to the light source [26]. The B/T hardness ratio results should be ≥ 0.8 to indicate adequate polymerization [27]. The hardness ratios obtained in the present study were less than 0.8 for all irradiation protocols with the Delton Opaque except the 40 second at 2 mm curing distance for both curing units (Table 8). In the case of a 10 second at 4 mm curing time, no microhardness value was obtained for the bottom surfaces due to inadequate polymerization. These findings support a previous study by Duangthip., *et al* 2011 [28]. However, contradictory findings have been reported [29]. According to Warnock and Rueggeberg 2004, the second-generation LEDs reached a conversion similar to the control in only 10 seconds [29]. It should be noted, however, that the sealants in their study were tested at only a 0.5-mm-thin layer. This higher conversion could have resulted from less light attenuation of the thinner sealant (0.5 mm). In the present study, the 1-mm thickness of sealants may have compromised the hardness ratio, especially because an opaque dental sealant was used.

	Multiple emission peak LED LCU		Single emission peak LED LCU	
	2 mm	4 mm	2 mm	4 mm
10 sec	1.7 (0.0)	1.2 (0.1)	1.0 (0.0)	0.5 (0.0)
20 sec	3.5 (0.0)	2.4 (0.2)	1.9 (0.1)	1.0 (0.0)
40 sec	8.0 (0.1)	4.9 (0.9)	4.0 (0.1)	1.9 (0.1)

Table 8: *Light Emitting Diode Light Curing Units Mean (standard deviation) Radiant exposure – bottom (J/cm^2).*

The opacity of the opaque white dental sealant that was used in this study is related to the opacifying agents present in its composition. This probably causes substantial reflection, scattering, and absorption of the light energy, which may prevent a more thorough cure through the sealant. A previous study by Yue C., *et al*. 2009 has reported greater depth of cure for Delton Clear than Delton Opaque irrespective of the curing time or distance [30,31]. Thus, the polymerization reaction may be diminished in opaque sealants and the DC and

KH decreased if the irradiation is deficient. This may be associated with the presence of titanium oxide fillers in the opaque version of this sealant which may interfere with the light penetration through the material. Shortall., *et al*. 2008 have attributed this type of effect in composite materials due to the changes in refractive index mismatch between filler material and resin during the curing [32].

The present study had some limitations. The type and amount of photoinitiators included in resin-based pit and fissure sealant that was used were not clearly identified. The manufacturers' information did not specify the type of photoinitiator that was used in the material. Therefore, further studies will be needed to determine accurately the performance of single and multiple emission peak LED LCUs on resin-based pit and fissure sealant formulated with different concentrations and ratios of CQ and alternative photoinitiators.

Conclusion

Within the bounds of the present study, the multiple emission peak LED LCU demonstrated significantly higher DC and KH than the single emission peak LED LCU at specific curing distances and curing times. Radiant exposure, DC and KH were shown to be significantly influenced by the exposure time, exposure distance and type of curing unit. Based on these findings an exposure time should be encouraged to be at 40s and an exposure distance less than 4 mm, since it can reach a sufficient polymerization that would lead to higher mechanical and physical properties at least within the same material and light curing units that were used in the present study. In addition, it should be stressed that the findings of this study are valid only for the specific material and LCUs studied; these results cannot be generalized to all sealants and curing protocols. Thus, more studies are needed to clarify the relationship between newer light curing technology and sealant polymerization.

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