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ORIGINAL ARTICLE

Influence of extended light exposure curing times on the degree of conversion of resin-based pit and fissure sealant materials

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KEYWORDS

Degree of conversion;
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Abstract *Purpose:* The aim of present study was to evaluate extended curing times on the degree of conversion (DC) of filled and unfilled resin-based materials used as pit and fissure sealants.

Materials and methods: The materials examined were a flowable composite (Filtek™ Z350 XT Flowable) and a pit and fissure sealant (Clinpro™ Sealant). Thirty disks of each material were prepared. The 30 made of the flowable composite were divided into three groups ($n = 10$ each) according to the three different curing times studied: 20 s (group 1), 40 s (group 2), and 60 s (group 3). Similarly, the 30 disks made of the pit and fissure sealant were divided into three groups ($n = 10$ each) according to the three different curing times: 20 s (group 4), 40 s (group 5), and 60 s (group 6). After polymerization, the disks were removed from the mold and stored in dry, lightproof containers in an incubator at 37 °C for 24 h. The DC was obtained using an Avatar 320 FTIR spectrometer. Then the data were analyzed using the Kruskal–Wallis test and the Fisher's least significant difference post hoc test for multiple comparisons (alpha = 0.05).

Results: DC values for the flowable composite (Filtek™ Z350 XT) were higher ($p = 0.002$) than those for the pit and fissure sealant (Clinpro™ Sealant). Group 2 and group 5 showed significantly higher DC values than group 1 and group 4, respectively. There was no difference between groups 2 and 3 or between groups 5 and 6 ($p = 2.93$).

Conclusion: An extended curing time improves the DC to some extent for both materials.

1. Introduction

In the recent era of preventive dentistry, pit and fissure sealants are considered an ideal method to prevent caries (Brown et al., 1996). Sealants prevent the initiation and progression of caries by creating a physical barrier that inhibits the entry of food particles into pits and fissures and the propagation of microorganisms (Beauchamp et al., 2009). In general, pit and fissure

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sealants are used in pediatric dentistry (Warnock and Rueggeberg, 2004).

Today, daylight-activated, low viscosity, resin-based materials such as sealants and flowable composites are generally used as pit and fissure sealants (Aguilar et al., 2007). The mechanical and physical properties of these materials are very important for maximizing their clinical longevity because the efficacy of these materials is directly linked to their retention (Nalcaci et al., 2004; Rode et al., 2009).

Polymerization is a process by which monomer molecules are converted into a polymer chain (Costa et al., 2009). Polymerization of the common composite monomer bis-glycidyl methacrylate (bis-GMA) takes place when a carbon–carbon double bond (C═C) forms between two methacrylate groups (Calheiros et al., 2008). During the polymerization process, the degree of conversion (DC) directly affects the mechanical and physical properties of the composite (Luciene et al., 2008). The DC is the magnitude to which the monomer is converted into a polymer (Boniek et al., 2011). It is calculated as the fraction of the amount of remaining aliphatic C═C in a cured material compared to the total number of C═C in the uncured material (Luciene et al., 2008). The DC depends upon many factors, including the composition of the composite material, the length of the curing time, the amount and type of photo initiator, the intensity of curing light, and the opacity of the material (Lohbauer et al., 2005; Obici et al., 2004). A sufficient DC is one of the most desirable physical properties of resinous materials because it has been shown that an insufficient DC is associated with increased solubility, and it facilitates the proliferation of cariogenic bacteria (Calheiros et al., 2008; Schneider et al., 2008).

It is assumed that the DC of a composite is directly proportional to the length of light exposure (Rastelli et al., 2008). Therefore, it is rational to investigate the shortest curing time that provides the highest DC without deleteriously affecting the physical properties of the composite (Beauchamp et al., 2009). In the literature, there are limited studies on this subject (Papagiannoulis and Eliades, 1989). Therefore, the present study evaluates the DC of filled and unfilled sealant materials cured for different lengths of time. The hypothesis of the present study is that extended light exposure will increase the DC of resinous materials used as pit and fissure sealants.

2. Materials and methods

The materials tested in this study included the filled sealant Filtek™ Z350 XT Flowable Restorative (3M™ ESPE™ St. Paul, USA) and the unfilled Clinpro™ Sealant (3M™ ESPE™ St. Paul, USA). Detailed descriptions of the materials are presented in Table 1.

2.1. Specimen preparation

Filtek™ Z350 XT and Clinpro™ Sealant were supplied in a unset paste form. A polytetrafluoroethylene mold, which was 5 mm in diameter and 1 mm thick, was placed on a substrate consisting of a glass slide covered with a polyethylene sheet. The mold was filled with low viscosity uncured paste. After the mold was filled, it was covered with another polyethylene sheet and glass slide, and light pressure was applied.

Both materials were cured with a Mectron Starlight Pro light-emitting diode (LED) curing lamp (Mectron, Italy) at an intensity of 1.000 mW/cm^2 and at a distance of 3 mm. The distance was standardized by using three 1-mm glass slides (Borges et al., 2010). After polymerization, the disks were removed from the molds and stored in dry, lightproof containers in an incubator at 37 °C for 24 h.

In total, 60 disks were prepared, with 30 disks of each restorative material. The 30 disks of each restorative material were further divided into three groups (n = 10) according to the three different curing times examined: 20 s, 40 s, and 60 s. The disks in groups 1, 2, and 3 were made of Filtek™ Z350 XT Flowable Restorative, and the disks in groups 4, 5, and 6 were made of Clinpro™ Sealant. Groups 1 and 4 were cured for 20 s (as recommended by the manufacturer), groups 3 and 5 were cured for 40 s, and groups 3 and 6 were cured for 60 s.

2.2. Degree of conversion

To determine the DC (%) of the materials, the number of C═C converted into single bonds was measured with an AVATAR 320 FTIR Spectrometer (Thermo Nicolet Inc., USA). Measurements were made using an attenuated total reflectance (ATR) accessory, and 16 scans were performed at 4 cm^{-1} resolution with a wave number range from 500 to 4000 cm^{-1}. The percentage of unreacted carbon–carbon double bonds (% C═C) was determined from the ratio of the absorbance intensities of aliphatic C═C (peak at 1638 cm^{-1}) against an internal standard (aromatic C─C, peak at 1608 cm^{-1}) before and after the specimen was cured. After the sample preparation, the background spectrum was taken by collecting an interferogram. Data were subsequently converted to frequency data by inverse Fourier transform. Then, we collected a single-beam spectrum of the sample, which contained absorption bands from the sample as well as the background. The ratio between the single-beam sample spectrum and the single-beam background spectrum gave the sample spectrum. Data analysis was performed by assigning the observed absorption frequency bands in the sample spectrum to appropriate normal modes of vibrations in the molecules (Smith, 1996). The DC was determined by subtracting the % C═C from 100%, according to the following equation (Arikawa et al., 1998):

Table 1 Materials used in this study.

Material	Manufacturer	Shade	Composition	Batch
Flowable composite	Filtek™ Z350 XT 3M™ ESPE™ St. Paul, USA	A_2	BIS-GMA/TEGDMA, BisEMA Zirconia/silica and silica; nanoparticle	7018A3D
Pit & fissure sealants	Clinpro™ Sealant 3M™ ESPE™ St. Paul, USA	Opaque white	BIS-GMA/TEGDMA	12637

Table 2 Mean (SD) degree of conversion (%) of flowable composite (Filtek™ 350 XT) and pit and fissure sealant (Clinpro™) at different time intervals.

Materials	Groups	N	Mean	STD	P value with respect to curing time	P value with respect to materials
Flowable composite (Filtek™ 350 XT)	1	10	45.28	6.431	$P = 0.002$	$P < 0.005$
	2	10	55.17	6.79	$P = 0.002$	
	3	10	58.83	6.338	$P = 2.93$	
Pit and fissure sealant (Clinpro™)	4	10	18.66	2.25	$P = 0.002$	
	5	10	45.00	3.46	$P = 0.002$	
	6	10	37.66	16.78	$P = 2.93$	

$$DC(\%) = 100 \times [1 - (R_{cured}/R_{uncured})].$$

where R is the ratio between Abs 1638 and Abs 1608 calculated for both cured and uncured resin cements.

3. Statistical analysis

Statistical analysis was computed with Statistical Package for Social Sciences (SPSS) software, version 16.00 (SPSS Inc., Chicago, USA). Descriptive analysis was carried out in the form of mean and standard deviation for DC. Significant differences between the values were evaluated using the Kruskal–Wallis test and least significant difference (LSD) post hoc test for multiple comparisons at a level of significance of 0.05.

3. Results

The findings indicated that the DC values for the flowable composite (Filtek™ Z350 XT) were higher ($p = 0.002$) than those for the pit and fissure sealant (Clinpro™ Sealant) as shown in Table 2.

For the flowable composite (Filtek™ Z350 XT), group 1 showed DC values that were significantly lower than group 2 ($p = 0.002$). Group 2 showed DC values that were significantly higher than group 1, whereas groups 2 and 3 were statistically similar ($p = 2.93$).

For the pit and fissure sealant (Clinpro™ Sealant), group 4 showed DC values that were significantly lower than group 5 ($p = 0.002$). Group 5 showed DC values that were significantly higher than group 4 but significantly lower than group 2. The DC values for groups 5 and 6 were statistically similar ($p = 2.93$).

5. Discussion

The clinical success of pit and fissure sealants is directly linked to their capacity to remain bonded to occlusal pits and fissures (Papacchini et al., 2006). The polymerized material forms a strong micromechanical bond to etched tooth enamel, thus physically protecting susceptible areas of the tooth surface and preventing dental caries (Christopher et al., 2009). Polymerization is a complex chemical mechanism in which two or more molecules combine to form a larger polymer molecule that consists of a repeating structural unit. Previous studies have reported DC values ranging from 45% to 85% in bis-GMA-based resin composites due to the complex mechanism of polymerization (Emami and Söderholm, 2005; Price et al., 2005; Soares et al., 2007). To date, the shortest curing time required to achieve a clinically satisfactory DC has not been specifically established, but the results of the present study indicate that a curing time of 40 s provided a satisfactory DC when compared to 60 s. However, a satisfactory DC was not achieved with a curing time of 20 s, which was recommended by the manufacturer.

The hypothesis in the present study that extended light exposure would increase the DC was partially accepted. Manufacturers recommended curing for 20 s, but curing for 40 s provided a higher DC than 20 s. Researchers have reported that an extended curing time using light sources generates more heat and leads to a higher DC in composite resin (Bagis et al., 2008; Trujillo et al., 2004). However, we observed no increase in DC upon curing for 60 s. A possible explanation for this result is that the light transmission may have decreased with time (Shortall et al., 2008). The filled flowable resin (Filtek™ Z350 XT) showed a higher DC than the unfilled pit and fissure sealant (Clinpro™). This result may be due to differences in the filler content of the two materials, or it may be due to differences in their organic content. Clinpro™ contains more bis-GMA monomers and had a lower DC than flowable Filtek™ Z350, which contains a lower organic content. The results are also influenced by several factors, such as monomer composition, material translucency, and the concentration of photo initiator (Calheiros et al., 2004). It is well known that there is a direct relationship between the DC of dental composites and curing light intensity (Rueggeberg et al., 1993).

The results of the present study are similar to those of Boniek et al. (2010) who demonstrated that a pit and fissure sealant showed a lower DC than a flowable composite. An extended curing time would prolong the excitation of photo initiator molecules, and filled flowable resin (Filtek™ Z350 XT) might contain more photo initiator molecules then the unfilled pit and fissure sealant (Clinpro™). This explanation could account for the higher DC in flowable Filtek™ Z350 than in the unfilled pit and fissure sealant. The results of the present study were in accordance with those of previous studies (Aguilar et al., 2007; Musanje et al., 2009; Silva et al., 2008). Moreover, a previous study demonstrated that an increased amount of triethylene glycol dimethacrylate (TEGDMA) in an experimental dimethacrylate-based polymer matrix led to an increased conversion of the monomer (Goncalves et al., 2008). Similarly, in the present study, the flowable composite showed a higher DC due to the increased TEGDMA content.

6. Conclusions

Within the limitations of this study, the following conclusions were obtained:

- The Clinpro™ Sealant showed a lower DC than Flowable Filtek™ Z350 XT at all three curing times examined. Filtek™ Z350 is preferred for use as a pit and fissure sealant because it showed a higher DC than Clinpro™.
- A curing time of 40 s was sufficient in providing a similar DC to a curing time of 60 s.

Ethical statement

This study employed only restorative materials and there were no ethical issues.

Conflict of interest

The authors have no conflict of interest to declare.

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