



EFFECT OF LIGHT CURING UNITS AND STORAGE TIME ON THE DEGREE OF CONVERSION OF FISSURE SEALANTS

Efeito das unidades fotopolimerizadoras e do tempo de armazenamento no grau de conversão dos selantes de fissuras

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Abstract

OBJECTIVE: To investigate the effect of light curing units and storage time on the degree of conversion of fissure sealants. **MATERIAL AND METHOD:** Three different fissure sealants were used: a resin-based pit and fissure sealant (Clinpro Sealant™), a polyacid modified resin composite (Dyract Seal™), and a resin modified glass ionomer cement (Vitremmer™). Each fissure sealant was polymerized with a halogen, high intensity quartz tungsten halogen, LED. Half of the specimens were tested to assess the degree of conversion immediately after polymerization and the others after one month's storage in artificial saliva. The degree of conversion was measured by FTIR. The data were analyzed using two way ANOVA. **RESULTS:** Statistical analysis of Dyract Seal™ showed significant differences between the DC values of samples tested immediately and again after one month's storage ($p < 0.05$). The DC values of Clinpro™ increased after the storage period ($p < 0.05$). The DC of Vitremmer™ showed no change after storage ($p > 0.05$). Results of the curing units testing showed no significant differences among the DC of samples polymerized with LED, HQTH, OTH tested immediately and again after one month for Dyract Seal™, Vitremmer™ and Clinpro Sealant™ ($p > 0.05$). **CONCLUSION:** Light curing units had no effect on the degree of conversion of the fissure sealants. But storage after initial light activation and polymerization for one month in artificial saliva can affects on DC of fissure sealants.

Keywords: Degree of conversion. Fissure sealant. Light curing unit. Storage. Artificial saliva.

Resumo

OBJETIVO: Investigar o efeito de unidades de polimerização por luz no grau de conversão dos selantes de fissuras. **MATERIAL E MÉTODO:** Três diferentes selantes foram utilizados: um selante resinoso de fissuras (Clinpro SealantTM, uma resina composta poliácida modificada (Dyract SealTM) cimento de ionômero de vidro modificado (VitremerTM). Cada selante de fissura foi polimerizado por LED halógeno de quartzo/tungstênio. Metade dos espécimes foi testada para assegurar o grau de conversão imediatamente após polimerização e outros após um mês de armazenamento em saliva artificial. O grau de conversão foi medido por FTIR. Os resultados foram analisados por meio do ANOVA. **RESULTADOS:** A análise estatística do Dyract SealTM demonstrou diferenças estatisticamente significantes entre os valores DC dos espécimes testados imediatamente e novamente após um mês de armazenamento ($p < 0,05$). Os valores do ClinproTM aumentaram após o período de armazenamento ($p < 0.05$). O DC do VitremerTM não demonstraram alterações após o armazenamento ($p > 0.05$). Os resultados das unidades de fotopolimerização não mostraram diferenças significantes ente DC de amostras polimerizadas com LED, HQTH, OTH testados imediatamente e novamente após um mês para o Dyract SealTM, VitremerTM e Clinpro SealantTM. **CONCLUSÃO:** Unidades de fotopolimerização não tiveram efeito no grau de conversão dos selantes de fissuras. Porém, armazenamento após ativação inicial e polimerização por um mês em saliva artificial pode afetar o DC dos selantes de fissuras.

Palavras-chave: Grau de conversão. Selantes de fissuras. Unidades fotopolimerizadoras. Armazenamento. Saliva artificial.

INTRODUCTION

The procedure of pit and fissure sealing, introduced more than thirty years ago, has proved to be effective for caries prevention in several studies (1-3) and has a primary role in minimal intervention dentistry (4). The variety of materials available to clinicians for pit and fissure sealing is then evidently wide (2). Resin composites with Bis-GMA monomer are traditionally used for sealing in combination with 37% phosphoric acid for etching (5).

The composites are lightly filled in order to keep viscosity low, thus allowing for deep penetration of the materials into pits and fissures (5), where a resin impregnated layer of enamel is formed, preceding the sealing effect (6). In the 1970's, glass ionomer cement was developed which had as one of its properties, the release of fluoride. However, research has demonstrated low retention rates for these materials when they are used as fissure sealants (7). Resin was incorporated to improve the mechanical properties of this material which led to the development of resin modified glass ionomer cement. These materials have been used for a long time as fissure sealants (8-10).

An innovative acid-conditioning procedure utilizing a non-rinse conditioner followed by a light cured compomer sealant has been introduced in recent years. The photochemically induced polymerization mechanism is the same for light-curing composite materials and compomers (11). Currently, visible light curing sealants are preferred because it is easy to control the working time and their application and can achieve proper polymerization in a shorter time (4, 11, 12). Most of these systems are sensitive to light with a wavelength of 460-480 nm and have an initiator like camphoroquinone and a reducing agent like tertiary amine (13). Conventionally, a quartz tungsten halogen light has been used to light-cure sealants. These units generally have a power density of 600-800 mW cm⁻² (14). Despite their popularity the halogen units present several limitations such as gradual reduction in energy output over time (due to bulb and filter degradation), limited depth of cure and a relatively long exposure time is required (15).

Quartz tungsten halogens with higher light intensities (HQTH) hold significant potential for use in dentistry. But the increased power of

recently developed light-curing units has renewed concerns about the biological safety of curing units (16). A solid-state light-emitting diode (LED) technology was proposed in 1995 for the polymerization of light-cured dental materials to overcome the shortcomings of halogen visible light-curing units. LEDs require no filters to produce blue light, are resistant to shock and vibration, and take little power to operate (17). LEDs longer life span and more consistent power density, compared with halogen bulb technology, show promise for dental applications (1, 18, 19).

Light cured materials have recognized disadvantages such as limited depth of cure and poor distribution of degree of conversion (DC) in cured resin. The degree of conversion defined as the percentage of reacted C=C bonds, affects many properties including mechanical properties, solubility, dimensional stability, color change and biocompatibility of the resin materials (20, 21). The unreacted double bonds may either be present in free monomer or as pendant groups on the network. The unreacted monomer may leach from the polymerized material and irritate the soft tissue (22).

The clinical performance of the fissure sealant to withstand chewing forces depends on its physio-mechanical, chemical and biological properties (23). These properties are related to the polymer matrix and adequate polymerization parameters such as the degree of conversion and

rate of polymerization (24, 25). However, in the literature there are few published studies which examine the degree of conversion of fissure sealants (14, 26).

Among the several methods to determine the DC of resin composite, Fourier transformation infrared spectroscopy (FTIR) has been proven to be a powerful technique and has been widely used as a reliable method (27-29) as it detects the C=C stretching vibrations directly before and after the curing of materials (28).

The objective of this study was to investigate the influence of three light curing units and storage in artificial saliva on DC of fissure sealants. The null hypothesis tested was that:

1. The DC does not improve after storage for a one month period in artificial saliva.
2. The DC is directly dependent on the light irradiance.

MATERIALS AND METHODS

The fissure sealants used in this study were Dyract Seal™ (compomer), Clinpro™ Sealant (composite) and Vitremer™ (resin modified glass ionomer cement). The details are shown in Table 1.

TABLE 1 - Compositions, manufactures and lot numbers of materials studied

| Materials | Lot Number | Manufacturer | Composition |
|-----------------|------------|------------------------------|--|
| Dyract Seal | 0410818 | Dentsply De Trey Germany | Strontium-amino-fluoro-silicate glass, Ammonium salt of PENTA and N,N-dimethyl aminoethyl ethacrylate Carboxylic acid modified macromonomers Diethyleneglycoldimethacrylate |
| Clinpro Sealant | 5GH | 3M ESPE, St.Paul. MN, USA | Bis-GMA, TEGDMA, EDMAB, CQ, BTH, silica, TBA TFB, TiO ₂ , rose Bengal sodium. |
| Vitremer | 20061101 | 3M ESPE, St.Paul MN, USA | Fluoro-aluminosilicate glass, potassium persulfate, ascorbic acid. 50% polyacrylic acid copolymer, 20 % HEMA, water, 13 % carboxylic acid copolymer. |

The three curing units used in this study were: a HQTH (Hilux 550, Express Dental Products, Toronto, Canada); a QTH (Optilux 501, Kerr, Danbury, CT, USA), a LED (Elipar Freelight 2, 3M Espe, Germany). Manufacturers' names and addresses, light intensities and applied polymerization time are listed in Table 2.

TABLE 2 - Visible light curing unit used

| Brand | Type of unit | Output of the light tip (mW/cm ²) | Diameter of the tip (mm) | Applied polymerization time (s) | Manufacturer |
|-------------------|---------------------------|---|--------------------------|---------------------------------|------------------------------------|
| Hilux Toronto, | Conventional Halogen | 450 | 10 | 20 | Express Dental Products, Canada |
| Optilux 501 | High Intensity Halogen | 810 810 | 8 8 | 20 20 | Kerr, Danbury, CT, USA |
| Elipar-Freelight2 | Light Emitting Diode | 920 | 8,5 | 20 | 3M ESPE-AG, Seefeld, Germany |

Specimen preparation

The specimen disks of fissure sealants were prepared using a teflon mold 5 mm in diameter and 1 mm in height to determine DC. Dyract Seal, and Clinpro Sealant were directly applied into the mold. Vitremer was placed into the mold after mixing the material in a powder/liquid proportion of 1:2. (8, 30). Five specimens were prepared for each group. A small (0.7mm) Mylar™ strip was placed over the resin and pressed with glass to remove any excess. The light source was placed in contact with the glass surface and the specimens were polymerized with the curing units according to the manufacturers' instructions. The light outputs of the curing units were measured before each testing procedure using a digital curing radiometer (Radiometer, Demetron, Danbury, CT, USA).

Degree of conversion measurement

The degree of conversion was measured twice in this study; the first immediately after curing and again a month after polymerization. The polymerized specimens were divided into two groups. Half of the specimens were tested for the degree of conversion immediately after polymerization and the others after one month's storage in light-proof boxes containing an artificial saliva solution to avoid further exposure to light irradiation.

Each specimen was individually pulverized into fine powder with a mortar and pestle and 50 micrograms were mixed with 5 mg of potassium bromide powder (Cario-Erba Reagenti, Milan, Italy). This mixture was placed into a pelleting device and then pressed in a hydraulic press with a load of 8 tons to obtain a pellet which was then placed into a holder attachment within the spectrophotometer. The absorbance peaks were recorded with the diffuse-reflection mode of FTIR (Perkin-Elmer, 1600 series) using 1 scans at 4 cm⁻¹ resolution and wavelength range between 400 and 4000 1 cm⁻¹. Spectra were also acquired from the same number of uncured adhesives. Uncured pastes were smeared onto thin potassium bromide disks for this purpose.

The number of double vinyl bonds remaining in the sample exposed to irradiation is shown by the intensity of the peak at 1.637 cm⁻¹ referring to the C=C stretching of vinyl the group and have been used the study of polymerization of acrylates and methacrylates (22). The DC was directly related to the decrease of this net peak absorption on the FTIR spectra. After curing, DC was determined with respect to the uncured resin composite using the following equation:

$$DC = ((A_0 - A_t) / A_0) \times 100$$

where A₀ is the peak area of the uncured fissure sealant, A_t is the absorption of the peak immediately after polymerization and after the one month storage period (22, 29-32).

Statistical analysis

The results of testing were registered into an Excel (Microsoft, Seattle, WA) spread sheet for calculation of descriptive statistics. The data were normally distributed, and parametric tests were employed. The values determined for different light sources (QTH, HQTH, LED), and the storage period were compared using two-way analysis of variance (ANOVA) at a preset alpha of 0.05 followed by pair wise comparison with Tukey HSD. Statistical significance was considered as $p < .05$.

RESULTS

FTIR spectra of the Clinpro Sealant, Dyract Seal, Vitremer after setting are shown in Figures 1, 2 and 3, respectively. Tables 3, 4 and 5 show the DC values obtained and the results of their multiple comparisons for fissure sealants.



FIGURE 1 - Spectra of the Clinpro Sealant™

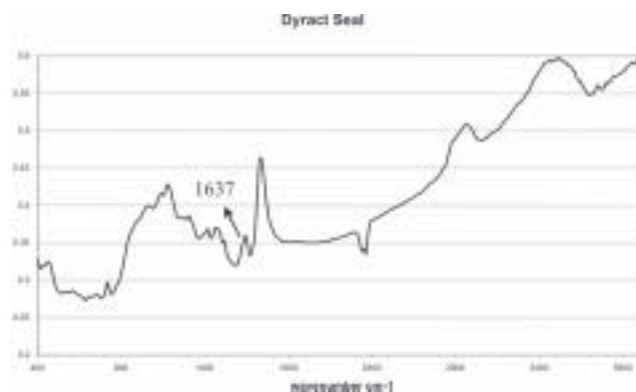


FIGURE 2 - Spectra of the Dyract Seal™

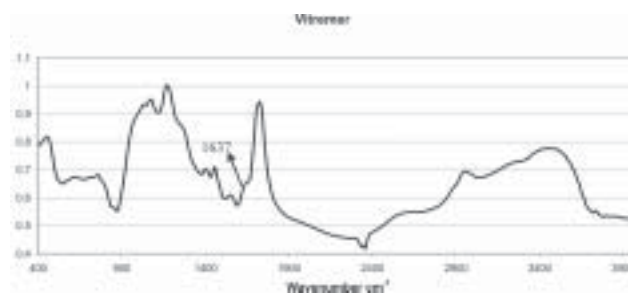


FIGURE 3 - Spectra of the Vitremer™

TABLE 3 - The results of the degree of conversion of Dyract Seal polymerized with different curing units

| Material | Curing units | *Immediately DC % ± SD | **1 Month DC % ± SD |
|-------------|--------------|------------------------|---------------------|
| Dyract Seal | Elipar | | |
| | Freelight2 | 80.35 ± 5.80 A,a | 80.35 ± 3.41 A,a |
| | Hilux | 85.47 ± 3.35 A,a | 85.05 ± 1.74 A,B,a |
| | Optilux 501 | 83.15 ± 3.01 A,a | 84.77 ± 1.32 A,a |

* Mean values followed by different small letters in the column differ statistically for the light curing methods.

** Mean values followed by different capital letters in the line differ statistically for the storage periods n: 5 for each group.

TABLE 4 - The results of the degree of conversion of Vitremer™ polymerized with different curing units

| Material | Curing units | *Immediately DC % ± SD | **1 Month DC % ± SD |
|----------|--------------|------------------------|---------------------|
| Vitremer | Elipar | | |
| | Freelight2 | 80.26 ± 0.76 A,a | 82.58 ± 3.28 A,a |
| | Hilux | 78.60 ± 2.65 A,a | 79.13 ± 3.28 A,a |
| | Optilux 501 | 81.52 ± 3.32 A,a | 80.52 ± 1.43 A,a |

* Mean values followed by different small letters in the column differ statistically for the light curing methods.

** Mean values followed by different capital letters in the line differ statistically for the storage periods.

TABLE 5 - The results of the degree of conversion of Clinpro Sealant™ polymerized with different curing units

| Material | Curing units | *Immediately DC % ± SD | **1 Month DC % ± SD |
|-----------------|--------------|------------------------|----------------------|
| Clinpro Sealant | Elipar | | |
| | Freelight2 | 84.59 ± 3.38 A,a | 82.14 ± 4.59 A,a |
| | Hilux | 79.85 ± 4.38 A,a | 87.85 ± 1.42 A,B,b,a |
| | Optilux 501 | 82.74 ± 3.27 A,a | 84.59 ± 1.98 A,a |

* Mean values followed by different small letters in the column differ statistically for the light curing methods.

** Mean values followed by different capital letters in the line differ statistically for the storage periods.

Dyract Seal™

Two-way ANOVA shows that there was no significant interactions between light curing units and the storage period ($p > .05$). There were significant differences between DC values of Dyract tested immediately and after one month ($p < 0.05$). The DC value decreased after storage. With regard to the curing units, there were no significant differences among the DC of samples polymerized with LED, HQTH, OTH both immediately and after one month ($p > .05$) (Table 3).

Vitremer™

Statistical analysis shows that significant interactions were not present between the light curing units and the storage period for Vitremer ($p > 0.05$). There were no significant differences between DC values of samples tested immediately and tested after one month ($p > 0.05$). Of the materials examined, it appeared to be the most chemically stable. All light curing units produced similar conversion values both immediately and after one month ($p > 0.05$) (Table 4).

Clinpro Sealant™

Statistical analysis shows significant interactions were presented between light curing units and the storage period ($p < 0.05$). The DC values of Clinpro increased with storage for one month and this increase was statistically significant ($p < 0.05$). With regard to the curing units, DC values promoted by the source were not statistically different immediately after polymerization. After one month, HQTH produced statistically higher DC values than LED ($p < 0.05$). However, LED did not differ statistically from QTH ($p > 0.05$).

Comparing DC values immediately and after one month for each light curing method, only DC values promoted by HQTH immediately after polymerization were statistically different from the values promoted by HQTH after one month ($p < 0.05$). The DC values, promoted by the other sources, for both sets of samples were not statistically different ($p > 0.05$) (Table 5).

DISCUSSION

The physical, mechanical and biological properties of fissure sealant are strongly influenced by the DC achieved by the organic matrix. Ideally, a resin material would have all of its monomer converted to polymer during polymerization (33-35). Problems associated with inadequate polymerization include inferior physical properties, solubility in the oral environment and increased microleakage with resultant recurrent caries (36). In addition to these, the process of monomer elution from fissure sealants is related to the extent of the polymerization reaction, i.e., the degree of conversion (37). The greater the extent of polymerization, the less unconverted monomer is available to be leached (38).

The DC values of fissure sealants observed in this study were limited from 78 to 87%. These values were higher than those typically seen for resin composites. This higher conversion could have resulted from less light attenuation of the thinner sealant than that seen in 2 mm composite increment (14). Another factor in the higher DC might be due to lower viscosity as fissure sealants have less filler content to penetrate into fissures and pits and lower viscosity results in greater DC % increasing the mobility of molecules (1, 34, 39). Previously, DC was reported to decrease progressively with increasing filler content, which limits the mobility of radical chain ends, and impair the mobility of the reactants (27).

The minimum DC for clinically satisfactory resin materials has not been established precisely. Nevertheless, a negative correlation of *in vivo* abrasive wear depth with DC has been established for DC values of composite resins in the range of 55-65%. This suggests that, at least for occlusally restorative layers, DC values below 55% may be contraindicated.²⁵ There appears to be no widely accepted DC value for fissure sealants. But it is accepted that the higher the conversion of double bonds, the greater the mechanical strength. In this study, high DC values for fissure sealants were obtained and the results indicate that these values may be sufficient for a clinically satisfactory fissure sealant. But this is an indirect assumption and further investigations are needed.

Of the three fissures sealant materials tested, exhibited different behavior for one month

due to different polymerization mechanisms. The DC mean value of Clinpro Sealant was 80.3% immediately after irradiation. DC continued to develop through one month when the DC increased to 84.3%. This is because polymerization reaction of light-activated composites continues even after the end of light irradiation and the DC shows a gradual increase after light exposure (30).

Conversely, the DC value of Dyract Seal, a polyacid modified resin composite, decreased with time. These materials are set primarily by light catalyzed polymerization and continue at a measurable rate for several hours after the light source is removed. After this time water sorption continues with the composites. The decrease in DC of Dyract Seal with time can be attributed to water absorption (31). These results are similar to a previous study (31) which showed decreased DC of acid-resin modified composites under wet storage as a result of water absorption which may have negatively affected the post-polymerization.

The setting reactions of resin modified glass ionomer cements include a radical chain polymerization and an acid-base cross-linking reaction. These materials are polymerized initially more rapidly due to the addition of chemical cure initiators. The full and rapid polymerization of the monomers in resin modified glass ionomer cements during light exposure may be due to a gel and the Trommsdorff-Norrish effect (40). It was suggested that the fast and complete polymerization of these materials can reduce concern over monomer toxicity (41).

Our results show that the DC of Vitremer remained stable after one month in light-proof boxes containing an artificial saliva solution. Thus, the first null hypothesis was not rejected for all materials tested.

The changes of DC of fissure sealants which are based on resin composite and polyacid modified resin composite when stored for a one month period in artificial saliva after irradiation are clinically important in terms of stability and performance after initial polymerization. Further investigations need to examine long-term storage periods on DC.

Three different light curing units to polymerize fissure sealants were used in this study. However there were no statistically significant differences in DC values among these light curing units, regardless of the power density of the light

source. Only for Clinpro Sealant, HQTH produced statistically higher DC values than LED after one month ($p < 0.05$). Thus, the second hypothesis was disproved. These results are in agreement with Warnock & Rueggeberg's findings (25). They also concluded that light units having power densities greater than that of the conventional QTH light did not necessarily provide a higher conversion rate. Again, this could result from the very thin sealant layer and low light attenuation values that tended to provide high levels of light energy within the sealant (25).

Although there were no statistically significant differences in the DC values among all light curing units, LEDs have certain advantages over other curing units. They are cordless, smaller, lighter, do not require a cooling fan and have an estimated life of over 10 000 hours. Moreover, LED technology is still developing and high intensity LED curing lights are available, which might be even more efficient. Given the fact that the LED technology continues to develop, it seems that it will play a leading role in the future.

All light sources have their own advantages and disadvantages and the individual clinicians should make a selection according to their clinical needs and expectations.

CONCLUSION

Within the limitations of this *in vitro* study, the following conclusion were drawn:

- 1 - The DC of the materials examined, with the exception of Vitremer, changed following a one month storage period in light-proof boxes containing an artificial saliva solution;
- 2 - No statistical difference was observed in DC values of fissure sealants polymerized with QTH, HQTH and LED.

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