Effect of Light Curing Methods on Microleakage and Microhardness of Different Resin Sealants

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ABSTRACT

Purpose: This study's purpose was to evaluate the effect of light curing methods on the microleakage and microhardness of sealants.
Methods: The Elipar Free Light 2 light emitting diode (LED) with 10- and 20-second curing times, and the Elipar 2500 halogen light with a 20-second curing time were compared. Four different sealants were used: (1) Delton Clear; (2) Delton Opaque; (3) UltraSeal XT Clear; and (4) UltraSeal XT Opaque. Specimens were fabricated in a silicone mold (2-mm thick) and cured. Knoop hardness was measured at the bottom and top surfaces. For the microleakage evaluation, 120 human molars were divided into 12 groups and sealed with the sealants and curing methods, as stated previously. The teeth were thermocycled and immersed in 2% methylene blue for 24 hours. Each tooth was sectioned and examined for dye penetration.
Results: There were no statistically significant differences in the microleakage of sealants polymerized by either the halogen or LED curing methods. The microhardness of sealants varied according to the type of material and curing method.
Conclusions: A 10-second polymerization time with light emitting diodes was not sufficient to cure the 2-mm-thick opaque or high filler loaded sealants. Decreasing the curing time, however, had no effect on the microleakage of the sealants.
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The use of pit and fissure sealants is recognized as an effective way of preventing fissure caries.[1] Several long-term studies have shown that anticariogenic effectiveness is related to sealant retention (ie, caries is completely prevented when fissures remain sealed). There are a wide variety of sealant materials from which to choose. The classic and most widely used sealants, however, are visible light cured resin sealants. Dental visible light curing materials generally contain a diketone-type photoinitiator that absorbs light in the 400- to 500-nm range covered by the blue light from the visible spectrum. The most common photoinitiator used is camphorquinone (**CPQ**), which has peak absorption at a maximum of 465 nm. Quartz Tungsten halogen curing lights (**QTHs**) are the most frequently used polymerization source in dental offices.[2] They have several drawbacks, however, such as a relatively short working life span (40-100 hours) and degradation of the internal component over time (bulb, reflector, filter) due to the high operating temperatures and large quantity of heat produced during duty cycles.[3,4]

To overcome the problems inherent to QTHs, innovative light emitting diode (**LED**) curing units have been marketed. LEDs have several advantages. First, they can be operated for many thousands of hours without a significant reduction in light output.[3] The reduced temperature associated with LEDs also can prevent degradation of light guides and does not pose a threat to pulpal tissue.[5] No filter is required to produce

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the blue light because LEDs produce light in a very narrow wavelength and are, therefore, highly efficient light sources. Several studies, however, have shown that LEDs with relatively low irradiances may result in insufficiently cured composites and, therefore, inferior mechanical properties of the restorations.[6,7] The light output of the first-generation LEDs required improvement to match with the cure produced by QTHs.[8]

A number of second-generation LEDs with high power light sources are now available.The results of studies have shown that these are capable of curing CPQ-initiated composites in half the radiation time of their predecessor.[9-11] The curing kinetics of photo polymerized dental sealants using LEDs showed that the second-generation LEDs reached conversion similar to control in only 10 seconds.[11] Controversially, a 10-second curing time with LEDs was inadequate when bonding orthodontic brackets to tooth enamel.[12]

According to the manufacturers' guidelines, pit and fissure sealants should be light cured for at least 20 seconds. Prolonged curing time, however, is uncomfortable for children, impractical with young children, and inconvenient for the clinician. Various attempts have been made to accelerate the speed of the light curing process by using a larger light guide or laser devices.[13,14] The benefit of using LEDs with shorter curing times in the management of children would potentially be great provided if there are no adverse effects. Comparative data on the performance of different sealants with a rapid cure using LEDs, however, remain limited.

Among the various types of sealants, unfilled resin sealants, which had filler particles of approximately 0 to 10 weight %, were the most widely used sealants.[15] Recently, filled resin sealants (40-60 filler weight %) or flowable composites have been recommended for both sealing pits and fissures and filling small cavities as a preventive resin restorative material. As visual identification of sealant loss or sealant placement with a clear sealant is difficult, opaque materials have been developed to enhance detection. These materials have opacities, such as titanium dioxide, added to the resin matrix.

It has been recognized that the composite shade has a significant impact on the polymerization of a resin composite. It is assumed that the filler loading and sealant shade might be the important factors influencing polymerization as well. Questions remain, however, about the performance of different sealants cured with various methods. Investigations are needed to compare different filler loaded sealants (unfilled and filled) and sealant shades (clear and opaque).

The aims of polymerization have been stated to be: having high uniform conversion in depth, having the shortest radiation time, and having low shrinkage stress. The clinical success of a resin-based system depends upon adequate polymerization. A higher degree of conversion in a resin system provides increased mechanical properties that, in turn, should provide for the restoration's longevity. The microhardness of a composite resin has been shown to demonstrate a good correlation with the degree of monomer conversion. The Knoop hardness number (**KHN**) predicts the relative degree of conversion for a specific resin under variable conditions.[16] Because the impact of the light source is not well known, investigations are needed that examine the relationship using bottom/top ratios (B/T KHN).

Light irradiation involves polymerization with a large proportion of shrinkage stress. Clinically, shrinkage stress may compromise synergism at the sealant-tooth interface, leading to bacterial microleakage. While much research has been conducted on the properties of resin composite polymerized with different curing lights, little information has been reported about the relationship between the curing method and sealant microhardness and microleakage. Therefore, the hypothesis of this in vitro study was that a 10-second polymerization time might be sufficient to cure a 2-mm thickness of 4 different sealants with an optimal bond of sealing.

The purpose of this in vitro study was to evaluate the effect of curing sealants with different filler loads and sealant shades using light emitting diodes in half of the radiation time of quartz tungsten halogen curing units on the microleakage and microhardness of different sealants. Light exposure of 20 seconds with a conventional halogen light curing unit (LCU) was used as the control.

METHODS

MICROHARDNESS

Three different light curing protocols (Table 1) were used:

1. QTH type: Elipar 2500 with a light intensity of approximately 400 to 500 mW/cm^2 (3M ESPE Dental Products, St. Paul, Minn) with a 20-second curing time;
2. LED type: Elipar Free Light 2 with a light intensity of approximately 1,000 mW/cm^2 (3M ESPE Dental Products) with a 10-second curing time; and
3. LED type: Elipar Free Light 2 with a light intensity of approximately 1,000 mW/cm^2 (3M ESPE Dental Products) with a 20-second curing time.

The light intensity of the curing units was measured with a radiometer (Curing Radiometer, model no. 100, Demetron, Kerr, CA). The 4 different sealant materials (Table 1) were investigated using 3 curing protocols for each combination of light and material. All specimens were prepared by a single operator, with care taken to avoid bubble formation.

The sealant material was placed into a 2-mm-thick silicone mold with a 4-mm internal diameter. The mold was placed between 2 glass slides. Each specimen was cured for the appropriate amount of time from the top surface only. The specimens were stored in the dark under 100% relative humidity at 37°C for 48 hours. Five KHN readings were made at least 1 mm from the edge of each top and bottom surface after an indenter dwell time of 15 seconds and a load of 50 g (microhardness tester FM-700, Mitutoyo G3, Tokyo, Japan). A B/T KHN ratio was calculated for each specimen. Top and bottom surface hardness and B/T KHN means were determined for each group.

Table 1. Type of Sealant Materials, Curing Units Times Used in Experimental Groups

Group	Sealant materials	Curing units*	Curing times (secs)	No. of specimens
1	Delton Clear, unfilled sealant	QTH	20	10
2	Delton Clear, unfilled sealant	LED	10	10
3	Delton Clear, unfilled sealant	LED	20	10
4	Delton Opaque, unfilled sealant	QTH	20	10
5	Delton Opaque, unfilled sealant	LED	10	10
6	Delton Opaque, unfilled sealant	LED	20	10
7	UltraSeal XT Clear, filled sealant	QTH	20	10
8	UltraSeal XT Clear, filled sealant	LED	10	10
9	UltraSeal XT Clear, filled sealant	LED	20	10
10	UltraSeal XT Opaque, filled sealant	QTH	20	10
11	UltraSeal XT Opaque, filled sealant	LED	10	10
12	UltraSeal XT Opaque, filled sealant	LED	20	10

** QTH=quartz tungsten halogen; LED=light emitting diode.*

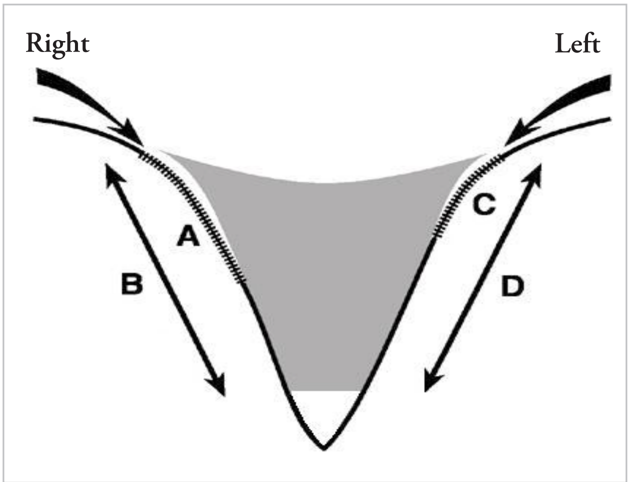

Figure 1. Scoring system employed for the evaluation of microleakage. A+C=length of dye penetration, B+D=length of resin–enamel interface, (A+C)/(B+D)=proportion of microleakage.

MICROLEAKAGE

A total of 120 extracted third molars—free of caries, fluorosis, fissure sealants, and restorations and which had previously been stored in chloramine T 2%—were selected by visual inspection. After that, the teeth were randomly assigned to one of 12 treatment groups (10 teeth each). All procedures were performed at room temperature (24±2°C). The treatment groups are shown in Table 1. The steps used for all procedures were standardized, as follows:

1. cleaning using a bristle brush with nonfluoridated paste for 15 seconds;
2. rinsing for 20 seconds with an air-water syringe;
3. drying with oil-free compressed air for 15 seconds;
4. etching with a 35% phosphoric acid gel (3M ESPE Dental Products) for 60 seconds;
5. rinsing for 30 seconds with an air-water syringe and dried with oil-free compressed air for 15 seconds;
6. applying 1 of 4 sealants (Table 1) following the manufacturer's instructions; and
7. polymerize with either a QTH curing unit (Elipar 2500) or LED (Elipar Free Light 2) for 10- and 20-second curing times (Table 1).

Care was taken not to place too much sealant material on each occlusal surface.

THERMOCYCLING AND DYE PENETRATION

Following sealant placement, the teeth were thermocycled in water for 1,000 cycles between 5±2°C and 55±2°C, with a dwell time of 30 seconds. The tooth surfaces were then coated with melted pink wax, leaving the sealant and approximately 1.5 mm uncovered enamel around the sealant. The coated teeth were immersed in 2% methylene blue for 24 hours to allow dye penetration into possible gaps between the tooth substance and the sealant.

MICROSCOPIC EXAMINATION

For further examination, the wax coatings were stripped off. The teeth were then sectioned into 4 fragments with 3 parallel cuts in the buccolingual direction with a low speed saw (Accutom 50, Struers, Copenhagen, Denmark). The thickness of the 4 sections per tooth was equal, with 6 sectioned surfaces obtained from each tooth. Microleakage was evaluated using a stereomicroscope (Zoom Stemi 200-C, Zeiss, Jena, Germany), at a 25X magnification, equipped with a digital camera linked to the computer. The examiner was blind to the groups. Image ProPlus software (Media Cybernetics 5.1, Media Cybernetics, Inc, Bethesda, Md) was used to measure the length of dye penetration and the enamel-sealant interface (mm; Figure 1).

Table 2. Means and Standard Deviations of Top and Bottom Hardness and Hardness Ratio for the Various Curing Lights and Sealant Materials*

Group	Sealant materials	Curing units (secs)	Top surface±(SD)	Bottom surface±(SD)	Hardness ratio±(SD)
1	Delton Clear	QTH (20)	14.26±0.35	13.57±0.27	0.95±0.03[a]
2	Delton Clear	LED (10)	13.96±0.39	13.59±0.30	0.97±0.03[a]
3	Delton Clear	LED (20)	15.64±1.03	14.70±0.64	0.94±0.04[a]
4	Delton Opaque	QTH (20)	15.04±0.61	1.08±0.15	0.07±0.01
5	Delton Opaque	LED (10)	13.08±0.27	NA	NA
6	Delton Opaque	LED (20)	13.82±0.26	2.87±0.35	0.20±0.03[b]
7	UltraSeal XT Clear	QTH (20)	21.51±0.99	14.09±1.07	0.65±0.04[c]
8	UltraSeal XT Clear	LED (10)	13.95±0.74	3.30±0.74	0.24±0.05[bd]
9	UltraSeal XT Clear	LED (20)	20.22±1.10	12.52±1.38	0.62±0.06[c]
10	UltraSeal XT Opaque	QTH (20)	24.57±1.23	5.21±0.91	0.21±0.04[b]
11	UltraSeal XT Opaque	LED (10)	19.6±0.92	NA	NA
12	UltraSeal XT Opaque	LED (20)	22.49±0.64	7.19±1.67	0.32±0.07[d]

** NA=not applicable. No statistically significant differences among groups are expressed by the same lower case letters (P>.05).*

STATISTICAL ANALYSES

The data were analyzed by general descriptive and multivariable methods using the general linear model of SPSS 11.5 software (SPSS Inc, Chicago, Ill). Microleakage and B/T KHN (dependent variables) were subjected to test whether the independent variables (sealant materials, curing methods) influenced the microleakage and microhardness of fissure sealants. The level of significance was set at $P<.05$.

RESULTS

MICROHARDNESS PART

The means and standard deviations of B/T KHN for the various curing lights and sealant materials are shown in Table 2. There were no data available for Group 5 (Delton Opaque with LED for 10 seconds) and Group 11 (UltraSeal XT Opaque with LED for 10 seconds) for B/T KHN or KHN, as the polymerization with LED for 10 seconds failed in the bottom surface of the opaque resin sealant. The data of the 10 remaining groups tested were abnormally distributed, and the variances were not equal. A robust test of equality of means (Brown-Forsythe) was used. There was a significant difference among the groups in terms of the hardness ratio ($P<.001$). A Dunnett T3 post hoc test was used to indicate the significant differences among the groups, as shown in Table 3. The results showed that the Delton Clear groups (Groups 1-3) presented a significantly higher hardness ratio than the other groups. No statistically significant difference was observed among the groups of Delton Clear regardless of the light source. In the UltraSeal XT Clear groups (Groups 7-9), polymerization with LED 10 seconds showed a significantly lower hardness ratio compared to the other light sources.

MICROLEAKAGE PART

The means and standard deviations of microleakage for the various curing lights and sealant materials are shown in Table 3. The microleakage data were abnormally distributed and the variances were not equal. The square root-transformed data were normally distributed; therefore, the square root of the values was used for 2-way ANOVA. The results showed that the type of sealing material had a significant influence on the microleakage ($P=.002$). When Tukey's test for pair-wise comparison was used in Figure 2, UltraSeal XT Clear exhibited significantly higher microleakage than UltraSeal XT Opaque and Delton Opaque, although no significant differences were found in microleakage between UltraSeal XT Clear and Delton Clear.

DISCUSSION

Light cured resin sealants have several advantages, such as better control of placement and a more complete polymerization, compared to chemically activated materials.[17] Polymerization shrinkage in these materials is an inherent disadvantage, resulting in gap formation between the tooth and material interfaces.[18] Gap formation contributes to microleakage, permitting the passage of bacteria and oral fluid from the oral cavity. This in vitro study demonstrated that microleakage was more evident in the UltraSeal XT Clear groups vs other sealant material groups.

The type of sealing material was found to have a significant impact on microleakage in our study. This finding might be explained in several ways. First, UltraSeal XT has high filler loading (approximately 58% by weight filled with an average particle size of 1.5 μm) as indicated for sealing pits and fissures and filling small cavities. Therefore, its high viscosity might lead to poor penetration of the material into the etched enamel and poor bond integrity between resin and enamel, resulting in higher microleakage compared to low filler loaded sealants. Secondly, high filler loaded sealants are usually placed in greater thickness than classical sealants because of the high viscosity of these materials.[15] It has been found that overfilled sealants cause significantly higher levels of microleakage.[19]

Finally, the opacity of sealant materials might have a significant impact on the control of application. As it is difficult to identify the margin of a clear sealant during placement, the clear and high filler loaded sealants might lead to poor adaptation of the sealant and higher shrinkage. As shown in the study, the UltraSeal XT Clear groups had significantly higher microleakage than the UltraSeal XT Opaque groups (Figure 2).

Interestingly, no significant difference was found in the amount of microleakage of sealants polymerized by a conventional QTH with a 20-second curing time compared to LEDs with either 10- or 20-second curing times. Therefore, it would appear that the rapid cure (10 seconds) by LEDs did not have an adverse effect on the microleakage of sealants. These findings are comparable to previous studies with composites[20,21] and sealants.[22,23] Contrary to the results of the hardness ratio in our study, a 10-second curing time with the opaque sealants indicated poor curing efficacy, as shown in Table 2.

Curing efficacy can be measured by direct and indirect methods. Direct methods that assess the degree of conversion, such as infrared spectroscopy, are complex, expensive, and time-consuming.[24] Indirect evaluation using hardness as a parameter for indicating the degree of conversion is widely accepted. A B/T KHN ratio is suggested to verify the efficiency of the cure in deep surfaces when compared to surfaces located closest to the light source.[25] If the polymerization is completely effective, the hardness ratio should be 1, as the hardness of the bottom surface should be the same as the top surface. The hardness ingredient should not exceed 10% to 20% (KHN ratio ≥0.8) for light-activated composites to be adequately polymerized.[26] According to this criterion, the hardness ratios obtained in this experiment were less than 0.8 for all irradiation protocols in the Delton Opaque and UltraSeal XT Clear and Opaque groups. Delton Clear was the only material in the experiment that achieved adequate polymerization.

In the case of opaque sealants with a 10-second curing time, no microhardness value was obtained for the bottom surfaces due to inadequate polymerization. Contradictory findings also have been reported. According to Warnock and Rueggeberg 2004, the second-generation LEDs reached a conversion similar to the control in only 10 seconds.[11] It should be noted, however, that the sealants were tested in 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 our study, the 2-mm thickness of sealants compromised the hardness ratio, especially when the opaque sealants were used. 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 pit and fissure sealants requires a 0.75-mm depth of cure[27] but ISO specification 6874 requires a cure twice as deep: 1.5 mm.[28]

In general, pit and fissure sealants are recommended to prevent dental caries in deep pits and fissures. Previous studies have demonstrated that the Y-fissure type (deep and narrow pits and fissures) is approximately 30% to 50% of all fissure types,[29] which might have a thickness of sealant covering up to 1.5 to 2.0 mm. Therefore, a 2-mm-thick sealant was employed in our study to ensure sufficient polymerization in the deep part. The question remains, however, as to what a proper thickness of testing sealant would be.

Table 3. Percentage of Microleakage and Standard Deviation in Each Group

Group	Sealant materials	Curing units (secs)	% microleakage ±(SD)
1	Delton Clear, unfilled	QTH (20)	22.51±15.17
2	Delton Clear, unfilled	LED (10)	31.76±30.91
3	Delton Clear, unfilled	LED (20)	22.94±35.13
4	Delton Opaque, unfilled	QTH (20)	34.41±34.17
5	Delton Opaque, unfilled	LED (10)	13.52±15.11
6	Delton Opaque, unfilled	LED (20)	9.61±10.57
7	UltraSeal XT Clear, filled	QTH (20)	36.23±28.76
8	UltraSeal XT Clear, filled	LED (10)	44.65±33.38
9	UltraSeal XT Clear, filled	LED (20)	43.83±33.19
10	UltraSeal XT Opaque, filled	QTH (20)	16.57±22.01
11	UltraSeal XT Opaque, filled	LED (10)	27.34±27.57
12	UltraSeal XT Opaque, filled	LED (20)	13.92±14.78

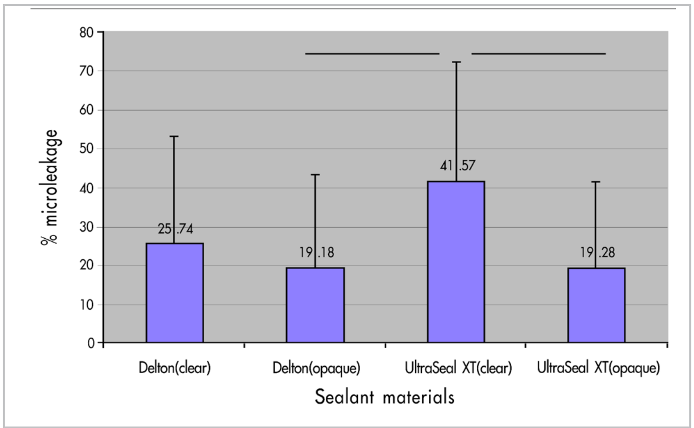

Figure 2. Percentage of microleakage and standard deviations of 4 sealant materials (lines between groups are statistically significant differences).

Several studies have demonstrated that high power LEDs cured the composite as effectively as conventional LEDs and QTHs even with a 50% reduction in time.[10,30,31] Contrary to the present study's results conducted with a 2-mm-thick sealant, UltraSeal XT (also used as a flowable resin composite) failed to meet the criterion (hardness ratio 0.8) in both shades for all curing methods. One possible explanation is that the flowable composite with smaller filler particles may be more difficult to cure compared to the resin-based composite with larger filler particles. The light might be more greatly attenuated and scattered by the submicron filler particles compared to the hybrid composite. Consequently, fewer photons of light were present to cure the composite at the bottom.[26]

As shown in Table 2, the clear unfilled sealants (Delton) demonstrated a significantly higher hardness ratio, regardless of the curing method, compared to the clear filled sealants (UltraSeal XT). The findings agree with a previous study showing that the ratio of filler to unfilled resin matrix is important.[4] It is more difficult for light to pass through the resin composite with a higher proportion of filler particles.

Surprisingly, the standard protocols with 20-second curing time with either QTH or LED were not sufficient to cure 2-mm-thick opaque sealants. Compared to the clear sealant, a greater degree of opacity for sealants seems to be an influential factor on microhardness. Apparently, the opacity or darker shade influenced the passage of the light through the composite.[32] The present study's results support the findings of previous investigations.[33,34] When opaque sealants were used, prolonged curing time (more than 20 seconds) with high output LCUs were recommended to overcome inadequate polymerization. It should be noted that different shades of the same brand of sealant can behave differently when irradiated with the same protocol. Statistically significant differences in hardness ratio, however, may not be imperative clinically. Further research should be done to verify the effect of different sealants and polymerization methods on the effectiveness of sealants.

A comparison of the microhardness performed on the top and bottom surface is shown in Table 2. All sealant materials demonstrated significant hardness reduction of the surface opposite the light exposure (bottom surface), except for the clear, unfilled sealant (groups 1-3). These results indicate that the curing degree decreased as a function of depth, as demonstrated previously in other studies.[28,29] With the increase of composite depth, the passage becomes more difficult because of the increase in the density of the polymer formed, which in turn reduces the activation of CPQ molecules.

As stated previously, the curing efficacy of the opaque sealants cured with LEDs for 10 seconds was impaired at the bottom surface, whereas no differences in microleakage were found. It could be that the higher the light intensity used, the higher the degree of conversion, which is associated with the improved mechanical properties of resin based restorative materials.[35] Conversely, these properties are in conflict with each other. An increased polymerization shrinkage and, thus, a higher stress level in bonded resin composites is expected, thereby increasing the amount of force acting on the bonded surface and inducing its detachment from the tooth enamel.[36]

Moreover, the degree of microleakage seems to depend on the integrity of the bond between the resin and the enamel at the orifices where the upper part of the sealant could be adequately polymerized. Likewise, if sealant retention is gained from the good adaptation of resin to fissure orifices, no dye penetration to the base of fissures (where the sealants might not be sufficiently cured) can be detected. Based on the inconclusive improvements at a 2-mm-thick hardness ratio, a reduction in polymerization time as short as 10 seconds is not recommended to cure opaque or high filler loaded sealants. It is possible that, when the deep layers of sealants are not adequately cured, the elastic modulus at the bottom will be lower than at the surface. This might increase the flexure of the sealant under masticatory forces, leading to an open margin and sealant loss in the future.

Currently, several LCUs and dental sealants are available in the market. Compared to earlier recommendations, the curing time for 2-mm resin composite layers can be limited to 20 seconds.[37] It is assumed that the same reduction also would apply when placing dental sealants. Clinicians should exercise caution when choosing these devices and sealant materials because the present study's results varied greatly with the type of sealant (filler loading and opacity), light source, and curing time. It should be stressed that the findings of this study are valid only for the specific materials and technology employed; they cannot be generalized to all sealants and curing protocols. Moreover, it should be remembered that in vitro microleakage and microhardness tests do not necessarily reflect clinical situations. Further studies are needed to clarify the relationship between light curing technology and sealant retention.

CONCLUSIONS

Based on this study's results, the following conclusions can be made:

1. Sealant microleakage was affected by the type of material. There were no statistically significant differences in the degree of microleakage in sealants polymerized by either quartz tungsten halogen or light emitting diode curing methods.
2. The microhardness of sealants varied according to the type of material (filler loaded and opacity) and light curing method.
3. A 10-second polymerization time with high power LEDs was not sufficient to cure 2-mm-thick opaque or high filler loaded sealants.

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