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**ORIGINAL ARTICLE**

## **Evaluation of Microhardness and Microleakage of Class II Silorane-Based Composite Restorations Post-Photoactivation Techniques**

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### **ABSTRACT**

**Objectives:** To evaluate the effect of additional light curing on microhardness and microleakage at cervical third of class II silorane-based composite restorations. **Methods:** Class II cavities were prepared on the proximal surfaces of 20 human premolars. The premolars were randomly and equally divided in two groups. In both the groups, class II cavities were restored incrementally with silorane-based composite and cured. Samples were immersed in 0.5% fuchsin. Dye penetration was recorded at four regions of the gingival floor. Vickers hardness test was performed 1 mm above the gingival margin. Data were statistically analyzed using ANOVA, post-evaluation of normal data distribution with Kolmogorov–Smirnov test at a significance level of 0.05. **Results:** The two photoactivation methods affected hardness and microleakage. Additional light curing increased Vickers hardness compared with occlusal curing. Comparison of microleakage in various areas of the first and second groups showed no significant difference with the first, but a significant difference with the second group. Additional light curing decreased dye penetration at all depths. The curing technique affected the results only in H3 and M3 regions. **Conclusion:** Additional light curing could lead to better hardness and lower microleakage. Therefore, we recommend this technique following initial occlusal curing.

**Key words:** hardness, microleakage, photoactivation technique, silorane-based composite resin

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### **INTRODUCTION**

In recent years, improvements in esthetic properties and tooth structure reinforcement have increased the use of composite restorations.<sup>1</sup> Polymerization shrinkage stress currently represents the main drawback in the use of composite resins. Specifically, it is responsible for gap formation and microleakage at the cavity surfaces.<sup>2</sup> Several techniques have been proposed to decrease contraction stress (e.g., use of stress-absorbing layers, modification of the chemical structure of organic matrices, regulation of light intensity).<sup>3</sup> Noticeable attempts have been made to establish new formulations of resin composite with reduced polymerization shrinkage to replace conventional methacrylate-based resin composites.<sup>4</sup> Silorane-based resin composite was

introduced as a low-shrinkage composite. It contains the cationic ring-opening monomers that compensate the polymerization shrinkage stress. In addition, it has been shown that silorane-based composites have lower degrees of conversion and polymerization depth than methacrylate-based composites.<sup>5</sup>

Compared to enamel, dentin structures have increased moisture and reduced mineral content. Microleakage commonly occurs at the gingival margins of class II cavities. Different incremental insertion methods have been proposed to significantly improve polymerization and to reduce the confinement effect on the shrinkage stress and microleakage.<sup>5</sup> The critical factors for the optimization of physical and mechanical characteristics of resin composites are curing depth and appropriate

polymerization.<sup>6</sup> In addition, irradiation time and photoactivation methods affect polymerization. Earlier studies have shown that, compared with continuous high-intensity curing methods, soft-start curing methods reduced shrinkage stress and improved marginal integrity.<sup>7</sup> To date, the formulation of new resin composites, adhesives, and various clinical techniques designated to enhance marginal adaptation have not resulted in a successful marginal integrity.

Some studies have evaluated the marginal adaptation and microleakage of silorane-based composites. In an earlier study, El-Eraky et al. showed that marginal discoloration and surface roughness were significantly higher for nano-hybrid than silorane-based resin composites.<sup>8</sup> Of note, Yamazaki et al. concluded that all resin composites showed microleakage regardless of the placement method, although incremental insertion significantly reduced microleakage.<sup>9</sup> Nevertheless, to date, studies on the transmitted light and its effect on microhardness and microleakage in different depths of class II restorations are limited. Therefore, evaluation of the gingival third of class II cavities restored by silorane-based resin composites under different photoactivation protocols is required.

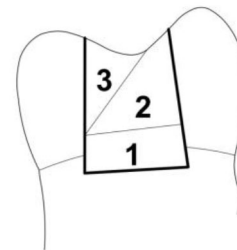
The aim of the current *in vitro* study was to assess the effects of two different photoactivation methods on the microhardness and microleakage of the silorane-based resin composite in different areas of class II cavities. Our null hypotheses were as follows: 1. The photoactivation method cannot influence the Vickers microhardness of different depths of gingival third; 2. Microleakage of the silorane-based resin composite cannot be influenced by the photoactivation method.

## METHODS

A total of 20 extracted human maxillary premolars without decay and cracks were selected. First, dental calculus, debris, and residual attached periodontal soft tissues were removed. Subsequently, the specimens were cleaned with slurry of pumice and rubber cup. They were then stored in 1% thymol solution at room temperature for not more than 14 days after extraction. A class II box-only cavity was prepared with dimensions of  $4 \times 1.5 \times 5 \text{ mm}^3$  on the distal surface of each tooth such that the butt joint gingival margin was located 1 mm below the cemento-enamel junction. Each preparation was performed using a new straight fissured diamond bur no.010 in a high-speed air-water cooling handpiece. Measurement of the dimensions of cavities and evaluation of uniformity between the preparations were performed using a digital caliper (Shinwa Digital Caliper, Niigata, Japan).



**Figure 1.** Class II cavities with matrix and wood wedge to establish appropriate contour and contact



**Figure 2.** Cross-sectional proximal view of the oblique incremental technique

The teeth were then embedded in the artificial maxillary arch with two adjacent teeth to simulate the proximal contacts. Subsequently, a mylar strip attached to a Tofflemire matrix retainer was placed around each tooth. Wood wedges were seated in the distal proximal areas to establish proper contour and contact, as well as perfect marginal adaptation (Figure 1).

Two-step self-etch silorane system adhesive (P90 system adhesive, 3M, USA) was used according to the manufacturer's instructions. The self-etch primer was applied to all surfaces of the preparation for 15 s, gently air dried, and then light cured for 10 s ( $650 \text{ mw/cm}^2$ ) using LED device (Blue Phase, Ivoclar Vivadent, Liechtenstein). Subsequently, a bonding agent was applied and light cured using a similar procedure. Specimens were randomly divided into two equal groups. In both groups, class II cavities were restored incrementally in three layers (gingival, oblique buccal, and oblique lingual) using a silorane-based resin composite (Filtek P90, 3M ESPE, St. Paul, MN, USA) (Figure 2).

In the standard direct curing group, each layer was cured exclusively from the occlusal surface. To this end, the soft-start curing technique was used with an initial intensity of  $650 \text{ mw/cm}^2$ , then increased to  $1200 \text{ mw/cm}^2$  for 20 s. In the mixed curing group, additional light curing was performed following matrix band removal. Additional light curing was performed for

**Table 1.** Composition of the silorane-based composite and adhesive system.

Composition	Filler type	Ingredients	Filler content, size, vol%, wt%	Photoinitiator
Filtek P90 (3M ESPE)	Silorane	3,4-Epoxy cyclohexyl ethyl cyclo polymethyl siloxane, bis-3,4-epoxy cyclohexyl ethylphenyl methylsilane	Silanized quartz, yttrium fluoride, 0.47µm, 55 vol%/ 76 wt%	Camphorquinone, Iodonium salt, electron donor
P90 System Adhesive (3M ESPE) Two-bottle self-etch adhesive system;		Primer: phosphorylated methacrylates, Vitrebond copolymer, Bis-GMA, HEMA, water, ethanol, silane-treated silica filler, initiators, stabilizers  Bond: hydrophobic dimethacrylate, phosphorylated methacrylates, TEGDMA, silane-treated silica filler, initiators, stabilizers		Camphorquinone, phosphine oxide  Camphorquinone

20 s from each direction of buccal and lingual, with an output intensity of 1200 mw/cm<sup>2</sup>. Table 1 describes the composition of the adhesive system and composite resin. The restorations received finishing and polishing procedures with medium, fine, and superfine grain disks (Soflex, 3M ESPE, USA) mounted on a low-speed handpiece with air–water spray.

Abbreviations: Bis-GMA: bisphenol-glycidyl-methacrylate; Bis-EMA: bisphenol-a-ethoxy dimethacrylate; UDMA: urethane-dimethacrylate; TEGDMA: triethyleneglycol dimethacrylate; HEMA: hydroxyethyl methacrylate. The brand name Filtek P90 is used in other countries as Filtek Silorane and Filtek LS.

Specimens were rinsed for 20 s and then dried with air and water spray. With the exception of the gingival margin and 1 mm around it, all tooth surfaces were covered with nail varnish to prevent dye penetration. Samples were then immersed in 0.5% alkaline fushin solution for 24 h. Finally, the specimens were embedded in acrylic resin and sectioned mesiodistally using the CNC machine (Nemo, Iran). For each restoration, four sections were prepared and numbered from buccal to lingual (from 1 to 4), respectively.

**Hardness test**

Cut surfaces were polished by an abrasive paper of 600, 800, and 1000 grit. Vickers hardness test (Buehler, USA) was performed for the silorane-based composite resin. Specifically, a force of 10 N for 10 s indentation time at a distance of 1 mm above the gingival margin was used. Vickers hardness values were classified as H1, H2, H3, and H4 from buccal to lingual, respectively.

**Microleakage evaluation**

Sections were observed under a stereomicroscope (Dino lite Pro; Anmo Electronics; Taiwan) to evaluate dye penetration. Photographs were taken and evaluated using Photoshop software (CS6) at 29× magnification. Dye penetration rate along the cervical dentin–resin interface was measured on the pixel scale. In addition,

dye penetration was recorded at the four regions of the gingival floor. These were named M1, M2, M3, and m4 from buccal to lingual, respectively.

**Statistical analysis**

Statistical analysis was performed using ANOVA, following the evaluation of normal data distribution with Kolmogorov–Smirnov test at a level of significance of 0.05. A p value of <0.05, with a 95% confidence interval, was considered statistically significant.

**RESULTS**

In the present study, two photoactivation methods were used. In addition, hardness and dye penetration were investigated in four areas at the gingival third of class II restorations. Based on a p value of >0.05 and the use of Kolmogorov–Smirnov test, the assumption of normalization for all variables was accepted in both groups. Two photoactivation methods significantly affected the variables (i.e., hardness and microleakage). In the hardness assessment, Hoteling test showed statistically significant differences between various locations of the first group (p=0.044). However, no significant differences were observed between various areas of the second group (p>0.05). According to the ANOVA test, additional light curing increased Vickers hardness values compared with standard occlusal curing (Table 2).

Hoteling test was performed to achieve leakage distribution in four locations. No significant difference was observed in the microleakage comparison of different areas in the first group (p>0.05). On the contrary, significant differences were found between various areas in the second group. Pair comparison showed a significant difference between M2 and other regions in the second group (p<0.05). Representative figures are shown in Figure 3. The ANOVA test showed that additional light curing decreased dye penetration at all depths. Table 3 outlines the values obtained for

**Table 2.** Mean Vickers hardness ( $\pm$ SD) in different depths of silorane-based composite resin, cured with two different photoactivation techniques

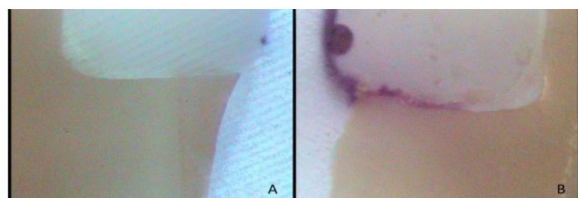
Curing method	Regions			
	H1	H2	H3	H4
Standard occlusal curing	81.88 $\pm$ 22.63 <sup>a</sup>	69.49 $\pm$ 8.88 <sup>a</sup>	62.99 $\pm$ 7.83 <sup>b</sup>	75.41 $\pm$ 9.93 <sup>a</sup>
Additional curing	76.49 $\pm$ 8.68 <sup>c</sup>	73.99 $\pm$ 10.96 <sup>c</sup>	76.81 $\pm$ 8.9 <sup>c</sup>	77.77 $\pm$ 9.97 <sup>c</sup>

**Table 3.** Microleakage values ( $\pm$ SD) on the pixel scale in different depths of composite resin following two photoactivation methods

Curing method	Regions			
	M1	M2	M3	M4
Standard curing	106 $\pm$ 67.08	153.11 $\pm$ 113.31	156.7 $\pm$ 89.9	139.1 $\pm$ 86.41
Additional curing	87.6 $\pm$ 74.77	136.7 $\pm$ 72.37	89.1 $\pm$ 20.8	77.6 $\pm$ 26.44

**Table 4.** Comparison of hardness and microleakage between two photoactivation techniques

Dependent Variable	Type III sum of Squares	df	Mean Square	F	Sig
H1	123.769	1	123.769	000.376	0.548 <sup>a</sup>
H2	122.722	1	122.722	01.114	0.307 <sup>a</sup>
H3	767.014	1	767.014	10.831	0.005 <sup>b</sup>
H4	013.869	1	013.869	00.126	0.728 <sup>a</sup>
M1	02568.056	1	02568.056	00.464	0.505 <sup>c</sup>
M2	02592.000	1	02592.000	00.287	0.600 <sup>c</sup>
M3	22330.889	1	22330.889	04.710	0.045 <sup>d</sup>
M4	09987.556	1	09987.556	03.225	0.091 <sup>c</sup>



**Figure 3.** Tooth sections of the specimens indicate dye penetration at the resin–dentin interface. (A) Presentation of no dye penetration in the resin–dentin interface; (B) Presentation of dye penetration at the resin–dentin interface

microleakage assessment. Only in the H3 and M3, photoactivation technique showed a significant effect on the results (Table 4).

## DISCUSSION

Hardness is an indicator of the degree of conversion (DC) when the same resin composites are tested in different polymerization situations. It has been shown that important data can be obtained by comparing hardness values at various depths of restoration.<sup>6</sup> Earlier studies have shown that light is not completely

transmitted through dentin and composite resin. The hardness at the bottom of restorations decreases with increased thickness of the resin composite. Small variations in the thickness of the composite restoration may change the light transmission, potentially affecting the DC, hardness, and other properties.<sup>10</sup> Increased irradiance has been recommended to enhance the DC during polymerization. Photoactivation in the silorane-based composite (Filtek P90) is achieved using a three-component initiating system, consisting of camphorquinone (CQ), an electron donor, and an iodonium salt.<sup>11</sup> Even with this program, it is noticeable that the silorane-based resin composite needs a minimum curing time of 20 s. This is because of the cumulative character of the initiator system of this resin, which cannot be compensated with higher intensities. Specifically, such characteristic is caused by its heat production, which plays a key role in this reaction, favoring the acceleration of the rings' opening.<sup>12</sup>

To verify whether polymerization was adequate at all regions of the gingival third, the authors verified the hardness. As a consequence, additional photoactivation technique improved polymerization at the bottom of the restorations. This is because light, even at reduced



irradiance, could reach the composite through the remaining buccal or lingual structure. Nevertheless, Vickers hardness values increased after additional curing at all regions. However, the difference was significant only at the H3 region.

In line with our findings, previous studies have also shown that higher exposure doses could cause the exothermic heat of composite resins, increase the monomer molecules mobility and determine a higher DC and microhardness.<sup>13-14</sup> Furthermore, an additional study explained that higher light intensities can generate more polymer growth centers. As a consequence, they can generate polymer cross-linking and higher strength of the composite resin.<sup>15</sup> In an earlier study, Nicola et al. concluded that an increased irradiation time produced higher DC at the bottom surface of the restorations.<sup>16</sup> In addition, Gritsch et al. explained that a longer exposure time with lower intensities improved the polymerization of the composite in depth. The authors found that this was caused by a delay in the rigid network formation.<sup>17</sup>

The difference between the Vickers hardness values of the surface and deep areas can be demonstrated by the fact that the light intensity provoking photoinitiators (CQ and others) tend to reduce from the surface to deep regions because of light attenuation and scattering. Based on this study's results, standard occlusal curing did not efficiently polymerize the deeper layers (H3 region). Following the standard photoactivation method, silorane-based resin composite at the H3 region resulted in significantly lower mean Vickers microhardness values than at H1 and H4 regions ( $p = 0.044$ ). Moreover, no significant differences were found between different regions in VHN values following additional light exposure.

In line with other studies, we found that the energy dose and photoactivation method control the DC in some way.<sup>3</sup> Here, a slight reduction in hardness was verified for direct occlusal compared with additional mixed curing. A possible explanation for this outcome is that the initial exposure at low light intensity from the occlusal surface for direct curing might result in the formation of short low-molecular-weight polymer chains. The latter may have less cross-linking interfering with the mechanical properties of the composite. Nevertheless, considering the magnitude of the difference verified between direct and mixed curing, prediction of its possible significant influence in clinical practice is complex. Furthermore, it should be noticed that the non-statistically significant increase in hardness might not be clinically relevant if the difference is  $<20\%$ .<sup>18</sup> In earlier reports, Moore et al. and Yap et al. tested the microhardness ratio from top to bottom of the restoration to estimate the depth of cure. The authors reported that this value should not exceed 10–20% in case of an adequate composite resin curing.<sup>18,19</sup> Polymerization of the silorane-based composite at H3 region following standard light curing

was insufficient to cause optimal physical properties. Various methods can be used to assess the microleakage around composite resin restorations. The most common and easy technique is dye penetration. This technique provides quantitative values for performance comparison of the various photoactivation protocols.<sup>20</sup> The aim of the microleakage evaluation is to obtain information about the marginal adaptation and sealing ability of the silorane-based composite resin.

Different techniques have been suggested to improve the sealing ability of the cervical margins in class II composite restorations. However, none of them completely eliminate the microleakage at the dentin–resin interface. Some authors have concluded that the incremental techniques, especially the oblique insertion, improve the marginal adaptation by shrinkage stress reduction.<sup>21,22</sup> However, other researchers advocate the bulk-fill insertion.<sup>23</sup> In addition, the three-sited photoactivation method has been proposed to improve the cervical marginal adaptation of class II resin composite restorations.<sup>24</sup> In a previous study, Lösche et al. concluded that following the transdental photoactivation technique, the marginal adaptation improved at the cervical margins of class II cavities. This was because of light intensity attenuation using the reflective wedge and shrinkage stress reduction.<sup>24</sup> On the contrary, in their report, Alves et al. concluded that mixed curing (i.e., occlusal + transdental curing) led to less gap formation, while not being detrimental for hardness of the composite resin.<sup>20</sup>

According to the results of the current study, additional photoactivation method provided less dye penetration than standard occlusal curing. However, no significant difference was observed between them except at M3 region. The smaller marginal adaptation observed for standard occlusal curing could be demonstrated using irradiance reduction when light transmitted from top to bottom.

However, we found that the Vickers hardness values did not differ significantly between the photoactivation methods. We believe that an additional explanation for the higher microleakage following occlusal curing may be linked to the lower mechanical properties of composites photoactivated with soft-start mode from occlusal surface. Some studies have shown that soft-start and pulse-delay photoactivation methods produce polymers with lower cross-linking density. These then affect their mechanical properties.<sup>25</sup>

In line with the hardness results, based on findings from the first group, the highest dye penetration was observed in M2 and M3 regions. Of note, these areas had the least amount of hardness. Furthermore, in the second group (additional curing from buccal and lingual) M1 and M4 regions exhibited lower dye penetration because of increased radiation exposure. In this group, the hardness enhanced due to increased

exposure. However, gap formation and dye penetration rate were not affected.

Our findings are in agreement with results of Guiraldo et al. study. Specifically, the authors did not find a sufficient polymerization in the deeper regions of silorane-based composite. They indicated that a higher exposure time or greater irradiance is efficient in obtaining better polymerization.<sup>26</sup> Previous studies have shown that improved polymerization on the gingival margin can be achieved by exposure to higher energy intensities. In addition, due to the ring-opening mechanism, the polymerization shrinkage of silorane-based composites does not immediately begin following light exposure, because longer time is needed to reach the gel point and vitrification. Furthermore, silorane-based composites have a slow polymerization rate because of the time required for the development of cations. As a consequence, more time is available for stress relaxation and flow.<sup>13</sup>

In summary, the results of our study show that a photocuring technique combining initial activation from the top surface of the restoration and final exposure through the dental structure might reduce the formation of gaps, while not being detrimental to composite hardness. Therefore, this technique could be encouraged in clinical practice.

## CONCLUSION

The present study demonstrates that additional light curing increases Vickers hardness values compared with standard occlusal curing. Furthermore, a significant dye penetration was observed between the different areas of the second group. Additional light curing decreased dye penetration at all depths. Photoactivation technique showed a significant effect exclusively on the results of H3 and M3.

## CONFLICT OF INTERESTS

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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