

EFFECT OF DIFFERENT SURFACE BARRIER AGENTS ON THE MICROHARDNESS OF A LIGHT-CURED COMPOSITE RESIN

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Abstract

This study aims to evaluate the effect of the oxygen inhibition layer formed during the polymerization of composite resins on microhardness and to examine the changes in microhardness values after removing this layer using different finishing techniques. Disc-shaped specimens (10 mm in diameter and 2 mm in thickness) were prepared using Valux Plus Composite (3M ESPE, USA) and divided into four groups ($n = 12$) to evaluate the effects of different surface barriers. The control group was polymerized without any surface barrier. In the other groups, Glycerin gel, Mylar Strip, or BisCover LV were applied to the surface of the composite before light-curing. After polymerization, they were wet-finished and polished using 1200-grit SiO₂ sandpaper. The specimens were stored in distilled water at 37 °C for 24 h. Microhardness tests were performed at five different points on each specimen using a 300 g force. The data were analyzed using one-way ANOVA followed by post hoc tests ($\alpha = 0.05$). It was observed that different finishing techniques used for the removal of the oxygen inhibition layer resulted in significant differences in the microhardness values of the composite material. The control group showed significantly lower microhardness values (99.28 ± 6.50) compared to the other groups ($p < 0.05$). Among all groups, the Mylar Strip group demonstrated the highest microhardness values (117.43 ± 4.24), which were statistically significant. Different finishing procedures had significantly different effects on the surface microhardness of composite resins. Although the Mylar Strip demonstrated the most favourable results, all application procedures showed significantly better outcomes compared to the control group.

Key words: composite, microhardness, oxygen inhibition layer

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Introduction. The oxygen inhibition layer has a significant and multifaceted impact on the stability of composite resin restorations. This layer, formed by the interaction of oxygen with free radicals during polymerization, inhibits complete surface polymerization and leads to compromised mechanical properties [1]. As a result, the wear resistance and bond strength of restorations are negatively affected, jeopardizing their long-term clinical success. Furthermore, in multilayered restorations or repair procedures, the oxygen inhibition layer may compromise restoration integrity by impairing interlayer bonding [2]. Moreover, incomplete surface polymerization may lead to the release of residual monomers, raising biocompatibility concerns and potentially causing cytotoxic effects on surrounding tissue. Aesthetically, the presence of this layer makes composite surfaces more susceptible to discolouration, thereby diminishing the visual quality of restorations [3]. From a chemical stability perspective, the presence of the oxygen inhibition layer increases the solubility of composite materials in the oral environment and may lead to surface degradation. Collectively, these factors threaten both the mechanical and aesthetic success of restorations, thereby undermining their overall clinical performance. Therefore, minimizing the effects of the oxygen inhibition layer is critical for the clinical success of composite resin restorations.

The oxygen inhibition layer consistently forms on composite surfaces polymerized in the presence of air. This layer primarily consists of unreacted monomers and oligomers and generally exhibits a liquid-like structure. Its thickness typically ranges from 2 to 6 μm ; however, in some cases, it has been reported to extend up to 16–37 μm [4]. This layer facilitates the adhesion of a new material layer to the surface by enabling chemical copolymerization, which enhances interlayer bond strength. However, due to its adverse effects on properties such as surface hardness and mechanical durability, the removal of the oxygen inhibition layer is considered a critical step for the success of restorations [4].

Free radical polymerization is the key chemical process responsible for hardening composite materials and ensuring their mechanical durability. It consists of three main stages: initiation (formation of free radicals and their reaction with vinyl monomers), propagation (rapid addition of monomers to a growing polymer chain), and termination (cessation of polymerization through radical combination or disproportionation). Additionally, chain transfer may occur, involving the transfer of active centres between chains [4]. These processes significantly influence the final properties of composite materials and emphasize the critical role of the oxygen inhibition layer. In light of this information, our study aimed to evaluate the effectiveness of different finishing techniques in reducing the effects of the oxygen inhibition layer and provided significant findings on its impact on restorative applications.

The oxygen inhibition layer has been shown to significantly affect the stability of composites in previous research [1, 5, 6]. However, studies on the materials and techniques developed to eliminate or reduce the effects of this layer remain

limited. Therefore, the aim of this study is to investigate the effects of different finishing techniques on the oxygen inhibition layer and, consequently, on the surface hardness of composite materials. The null hypothesis was that no significant differences in surface hardness of the composite materials would be found among the groups treated with different finishing techniques.

Materials and methods. Disc-shaped specimens were obtained by applying Valux Plus Composite (3M ESPE, US) into acrylic molds with a diameter of 10 mm and a thickness of 2 mm. In this study, four different groups ($n = 12$) were established, including the control group, to evaluate the effects of three different surface barrier applications. In the control group, the composite material was applied without any surface barrier, and all specimens were polymerized using an LED curing unit (Demi Ultra, Kerr) with a wavelength range of 450–470 nm and an intensity of 1100 mW/cm² for 40 s. In the other three groups, Glycerin Gel, Mylar Strip, and BisCover LV were respectively applied on the composite surface, and each specimen was light-cured for 40 s. The compositions and manufacturers of these materials are listed in Table 1. In all groups, a constant distance of 1 mm was maintained between the tip of the light-curing unit and the specimen surface.

T a b l e 1
Composition and manufacturer information of the materials

Materials	Manufacturer	Composition
Valux Plus Composite	3M ESPE, US	BisGMA/TEGDMA, Zirconia/silica (66 wt%)
Glycerin Gel (Liquid Strip)	Ivoclar VivaDent, Light-enstein	Glycerin, water, highly dispersed silicon dioxide, aluminium oxide
Mylar Strip	Hawe Transparent Strip, Kerr Hawe, Switzerland	Polyester
BisCover LV	Bisco/US	Dipentaerythritol Pentaacrylate Ethanol

Following polymerization, all specimens were rinsed with distilled water and allowed to dry under ambient conditions. Prior to microhardness evaluation, surface finishing and polishing were carried out using 1200-grit silicon dioxide abrasive paper. Upon completion of the polishing procedure, the specimens were incubated in distilled water at 37 °C for 24 h to simulate oral conditions.

Before the microhardness testing, a load of 300 g was applied to five different points on the surface of each specimen for 15 s. Subsequently, the specimens were analyzed using a microhardness tester (Buchler, Japan). The obtained data were statistically analyzed using one-way ANOVA followed by post hoc tests.

Results. Statistical analysis revealed significant differences in microhardness values among all groups ($p < 0.05$). All results were considered significant at a 95% confidence level ($p < 0.05$), and adjustments for multiple comparisons were

made using the Bonferroni correction. Post hoc Bonferroni comparisons indicated that each group was significantly different from the others. The Control group exhibited a significantly lower microhardness value (99.28 ± 6.5) compared to the other groups. In contrast, the Mylar Strip group demonstrated a significantly higher microhardness value (117.43 ± 4.2) than the others. Based on the mean microhardness values, the groups were ranked from lowest to highest as follows: Valux Plus Composite, BisCover LV, Glycerin Gel and Mylar Strip. The mean microhardness values of the specimens are presented in Table 2.

T a b l e 2
Microhardness values of the groups

Materials	Mean $\pm$ SD	<i>p</i> *
Valux Plus Composite (Control group)	99.28 ± 6.5^a	< 0.05
Valux Plus Composite + Glycerin Gel	109.93 ± 4.7^b	
Valux Plus Composite + Mylar Strip	117.43 ± 4.2^c	
Valux Plus Composite + BisCover LV	104.94 ± 9.2^d	

*One-way ANOVA Test
^{a-d} There is a difference between groups with different letters

Discussion. Surface microhardness is widely accepted as an indirect indicator of the degree of polymerization in resin-based composites [7]. Since oxygen acts as a potent inhibitor that can hinder or terminate the polymerization reaction, the formation of the oxygen-inhibited layer (OIL) can negatively impact surface properties [8]. A lower degree of polymerization due to OIL results in reduced hardness, which compromises the material's resistance to permanent deformation [9]. In contrast, higher surface microhardness values are associated with enhanced scratch and wear resistance, thereby contributing to the long-term clinical performance of the material [10]. Considering that polymerization is influenced by multiple factors such as initiator type, filler content and size, shade, light intensity, and curing duration, this study was conducted under standardized conditions to isolate the effects of different surface barrier applications on microhardness and OIL formation [1]. As significant differences in surface microhardness were observed among the groups treated with different finishing techniques, the null hypothesis was rejected. These findings are consistent with previous studies, which have also reported that surface treatments can significantly influence the hardness and polymerization efficiency of resin-based composites [5, 6].

In the literature, studies investigating the effects of the oxygen inhibition layer have consistently emphasized its negative impact on the mechanical performance of resin-based restorative materials. Early research identified the inhibitory role of oxygen in free radical polymerization and described the underlying mechanism responsible for the formation of the oxygen-inhibited layer [11]. Subsequent studies further demonstrated that this layer can compromise the chemical integrity

and mechanical durability of restorative materials in clinical conditions [12]. In line with these findings, the present study shows that surface finishing techniques designed to reduce or eliminate the oxygen inhibition layer significantly improve surface microhardness. These results underscore the importance of controlling the oxygen inhibition layer to enhance the long-term clinical success of composite restorations.

A previous study reported findings consistent with those of the present research, identifying the use of a Mylar Strip as the most effective method for preventing the formation of the oxygen inhibition layer (OIL), while glycerin application was found to be the second most effective technique [1]. This can be attributed to the Mylar Strip's ability to fully isolate the composite surface from atmospheric oxygen, thereby limiting OIL formation to the oxygen already entrapped within the material. As a result, the extent of the OIL is significantly reduced. In contrast, when glycerin is applied, residual oxygen within the gel itself – as well as oxygen already present on the composite surface – may contribute to OIL development. Consequently, a higher amount of OIL may form compared to the Mylar Strip technique [1].

In another study using glycerin gel and Mylar Strips, the surface roughness of the specimens was compared, and no significant differences were found between the groups. The results of that study were attributed to the fact that the specimens had been polished prior to surface roughness measurements [5].

In this study, following Mylar Strip, the Glycerin Gel group exhibited the highest surface microhardness values. One of the reasons for the effectiveness of glycerin is its stable consistency, which does not harden during light curing or interfere with the intensity of LED devices [13, 14]. Its low-viscosity liquid form also allows it to reach difficult-to-access areas, such as occlusal surfaces of posterior teeth [13]. By limiting the interaction between oxygen and free radicals, glycerin may help reduce the formation of the oxygen inhibition layer, making it a clinically useful material in specific restorative applications [13].

Among the limitations of this study is its *in vitro* design; therefore, intraoral conditions could not be fully replicated. Factors such as the presence of saliva and the temperature of the oral cavity were not reflected in the results. Additionally, this study evaluated only a single type of composite material, curing device, and polishing protocol. Future studies should consider assessing the effects of different variations, as this would be clinically relevant and contribute to a more comprehensive understanding.

Conclusion. In this study, the effects of the oxygen inhibition layer on the microhardness properties of composite materials were investigated. The findings revealed that barrier agents used to prevent the formation of the oxygen inhibition layer had a positive impact on the microhardness of the composite. Although Mylar Strip demonstrated the most effective results, the use of other barrier agents also contributed to the improvement of microhardness values.

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