



EVALUATION OF THE THICKNESS OF OXYGEN INHIBITION LAYER PRODUCED ON TWO DIFFERENT COMPOSITE MATERIALS: A STEREOMICROSCOPIC STUDY.

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Abstract:

Aim: aim of this study was to evaluate the thickness of oxygen inhibition layer (oil), produced on two composite materials

Objective: To evaluate the thickness of oxygen inhibition layer (oil), produced on two different composite materials.

Material and method: The thickness of oil on 2 different composite materials group1 n=30 - fiber reinforced composite and group 2 n=30 - nanohybrid composite were evaluated. The materials were divided into 3 subgroups depending on the surface treatment given i.e - (control group- a, water spray group -b, ethanol group -c. The depth of the inhibition layer was measured using stereomicroscope around the periphery at a magnification of 40x. The results were recorded in micrometers between the outer boundary of the specimen and the polymerized-unpolymerized resin interface. Results: compared to composites of (Group 1b and Group 1c fiber reinforced composite), the (Group 2c and Group 2b nanohybrid composite) displayed a maximum thickness of OIL.

Conclusion: The oxygen inhibition layer, which serves as an intermediary layer, is kept on the composite's surface after being exposed to either ethanol or water spray. However, whether the oxygen inhibition layer was present or not, the short fiber-reinforced composite shown a propensity for cohesive fractures.

Keywords: oxygen inhibition layer, surface treatment, short fiber composite, nanohybrid composite

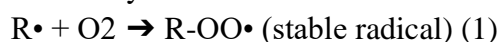
Introduction

Currently, Resin composites continue to be the most widely used restorative materials due to their excellent esthetics, functional capacity, and mechanical properties. Restorations based on resin composites can be affected by the

Formation of rough surfaces that can cause staining, plaque accumulation, gingival irritation, recurrent caries, and wear kinetics, among other problems(5)

Typically, commercial dental composites are random copolymers of 2,2-Bis[4-(2-hydroxy-3-methacryloxypropoxy)phenyl]propane (Bis-GMA) And triethyleneglycol dimethacrylate (TEGDMA), filled with various types of inorganic particles. Bis-GMA and TEGDMA are bi-functional methacrylate monomers that harden following a free-radical-induced polymerization reaction.

The oxygen diffusing from the atmosphere into curing resins is responsible for the Inhibited surface layers commonly found on freshly polymerized unfilled Resins (Finger *et al.*, 1996; Vallittu, 1999; Yatabe *et al.*, 2001). This is due to The oxidation of radicals into stable species known as peroxides (Reaction 1) (Andrzejewska *et al.*, 1998; Schulze and Vogel, 1998), which have low Reactivity toward monomers.



A principal drawback associated with the use of resin composites is the requirement of an incremental placement technique to overcome a limited curing depth (~2–3mm for the majority of commercial material types of regular shade)(6)

During the light-curing procedure, oxygen in the air interferes with the polymerization reaction (due to higher reactivity of oxygen with a radical as compared to reactivity of monomer with the radical). This results in the formation of an OIL on the surface of the composite(8)

The oxygen inhibition layer (OIL) that forms on the surface of methacrylate based resins cured in the presence of oxygen has received much attention in the literature, both positive (Kim *et al.*, 2006; Truffier-Boutry *et al.*, 2003) and negative (Eliades and Caputo, 1989; Rueggeberg and Margeson, 1990; von Beetzen *et al.*, 1996).(7)

The OIL is similar in composition to that of an uncured resin with consumed or reduced amounts of photoinitiator, hence also known as an unpolymerized (uncured) layer of resin. The thickness of the OIL could influence the interlayer bond strengths of the composite resins, as OIL is known to (i) impair the interfacial homogeneity; (ii) permit complete interdiffusion of the freshly overlaid composite through the oxygen inhibition zone (if OIL is thin); and (iii) compromise the mechanical strength (if OIL is thick). Hence, the thickness of the OIL is crucial for the integrity of the layer itself and the quality of the interlayer bonding. When a new increment of the composite is added, OIL formed on the previous layer readily adapts the overlying material. It, thereby, increases the contact area and allows increments of composite on both sides of OIL to cross the interface and blend, to form an interdiffused zone, where copolymerization can take place to produce a chemical bond. All these actions will tend to fortify layer–layer interaction.(8)

The amount of diluent (TEGDMA) also affects the oxygen inhibition depth (in this study, as in microhybrid and short glass fiber-reinforced (SGFR) composite). When a composite is reinforced with fibers, the fibers and their orientation may also influence the oxygen inhibition depth.(8)

Previous investigations have demonstrated an oxygen inhibition layer (OIL) thickness for dental resins and resin composites ranging from ~4 [3] to 40_μm (6)

With the evolution of these materials, bulk-fill resin composites appeared, which allow a monoblock technique to be used, placing a restoration with a 4–5 mm thick layer and light-curing easily, as they are more translucent and have less filler.(5)

Bulk-fill resin composites have become a good product of choice due to their quality in terms of strength and durability, presenting high biocompatibility and better physical properties, such as greater wear resistance and surface hardness, as they are formed by nanoparticles and ceramic metal fillers that improve the resin surface, thus facilitating modeling and polishing with a better esthetic finish(5)

Enhancing the physical and mechanical properties of resin composites by incorporation of millimeter-scale short-fiber fillers and resin matrix encompassing a semi-interpenetrating polymer network (semi-IPN) structure has proved promising(4)

The short-fiber-reinforced composite is a dimethacrylate-based composite, in which Bis-GMA and TEGDMA are used as monomers and millimeter-scale short-fiber fillers with a semi-IPN structure (1, 2) are incorporated into the resin matrix. Fibers are known to restrict the polymerization shrinkage (24, 25), but as a result of the monomer composition, the polymerization reaction still proceeds by free radicals. Thus, formation of an oxygen inhibition layer on the surface of the short-fiber-reinforced composite is expected, although the viscosity of the resin Matrix is high. (4)

The effect of oxygen on the bonding properties of the newly developed short-fiber-reinforced composite (everx Posterior) is unknown.(4)

The present investigation included dimethacrylatebased composites (short-fiber-reinforced composite and nanohybrid and The study aims were: (i) to evaluate the thickness of the oxygen inhibition layer on the surface of the short-fiber-reinforced composite in comparison with freshly polymerized nanohybrid-, resins when treated with distilled water and ethanol(4)

Material and method

The thickness of oil of 2 different composite materials (60 specimen n=30 each **group1- fiber reinforced composite group2- nanohybrid composite ,further divided into 10 each under 3 sub-groups**).The 3 subgroups (a,b,c) depending on the surface treatment given-no surface treatment (control group ,water spray ethanol. Equal amount of each type of composite resin was applied on the middle of a horizontally placed glass microscope slides.A constant sample thickness of 0.1mm was ensured by placing two glass plates (20 mm × 20 mm)on each side of the composite resin.The specimens were polymerized for 40s using a light-emitting diode light-curing unit.The prepared groups for testing were as follows:

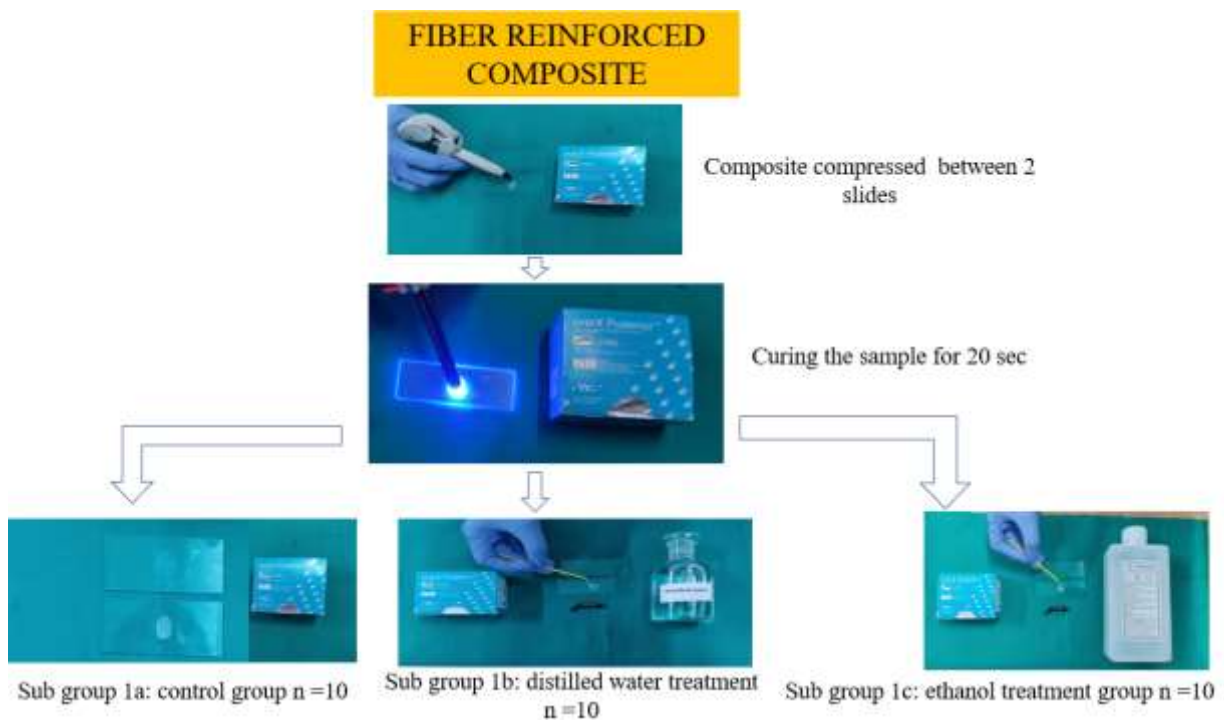
Subgroup 1a and 2a, the oil was not treated,

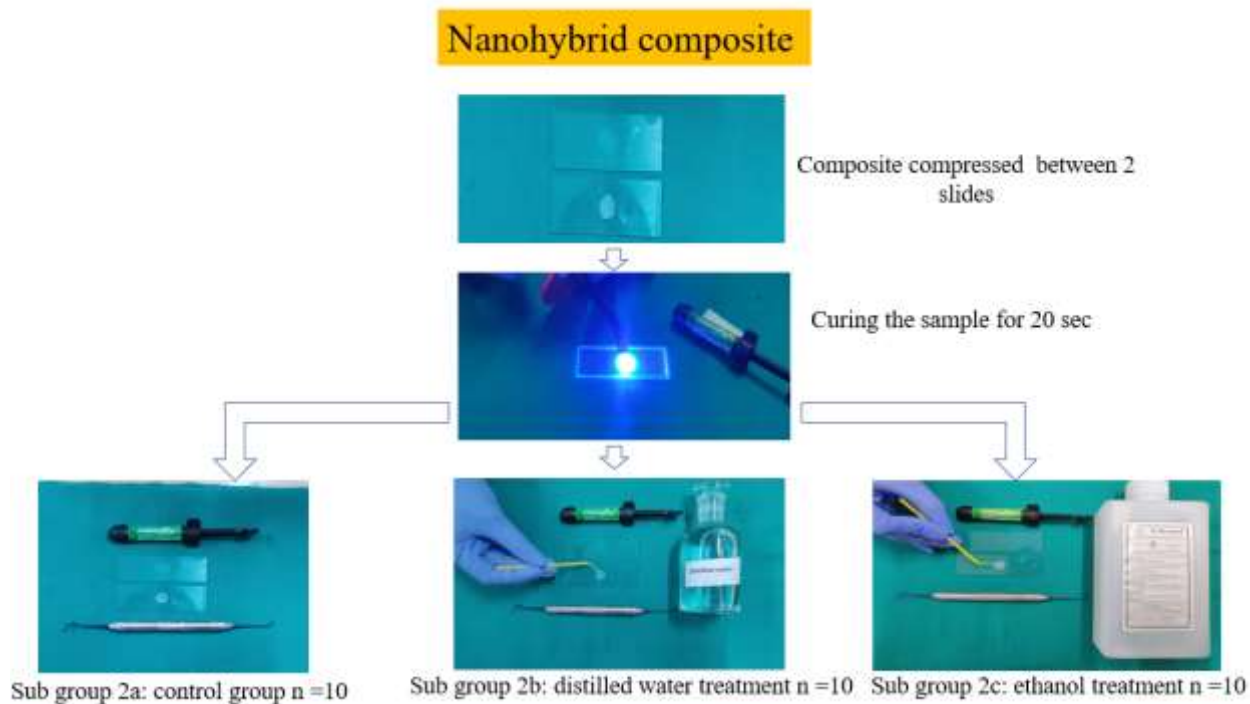
Subgroup 1b and 2b ,the oil was treated with water applied as a water spray for 20 s and gently air dried for another 20 s.

Subgroup 1c and 2c-the oil was treated by wiping the surface of the cured specimen using alcohol sponges soaked 20s and then gently air dried for another 20 s;

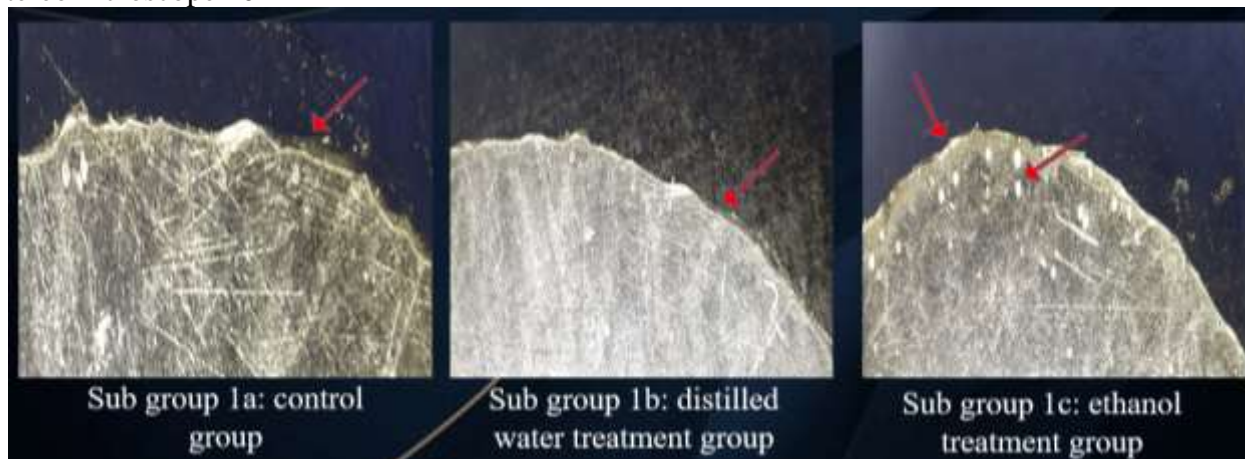
The depth of the inhibition layer was measured using stereomicroscope around the periphery at a magnification of 40x.The results were recorded in micrometers between the outer boundary of the specimen and the polymerized–unpolymerized resin interface.

Materials

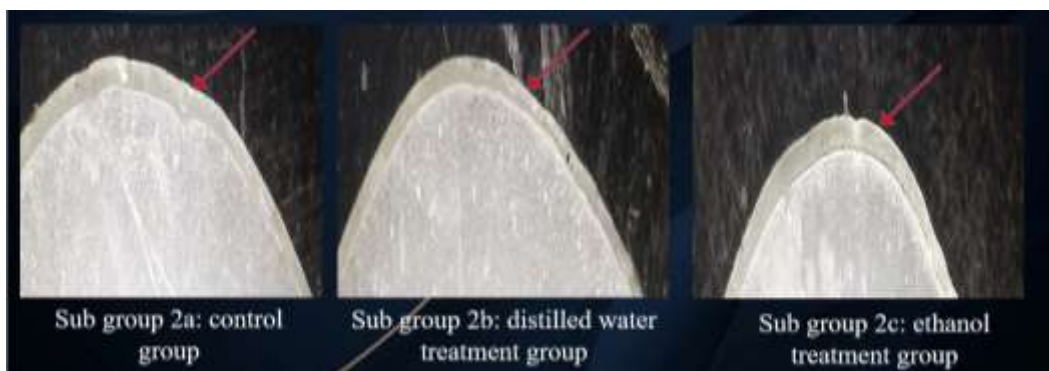




Stereomicroscope 40X



Fiber reinforced composite



Nano hybrid composite

Graph and result

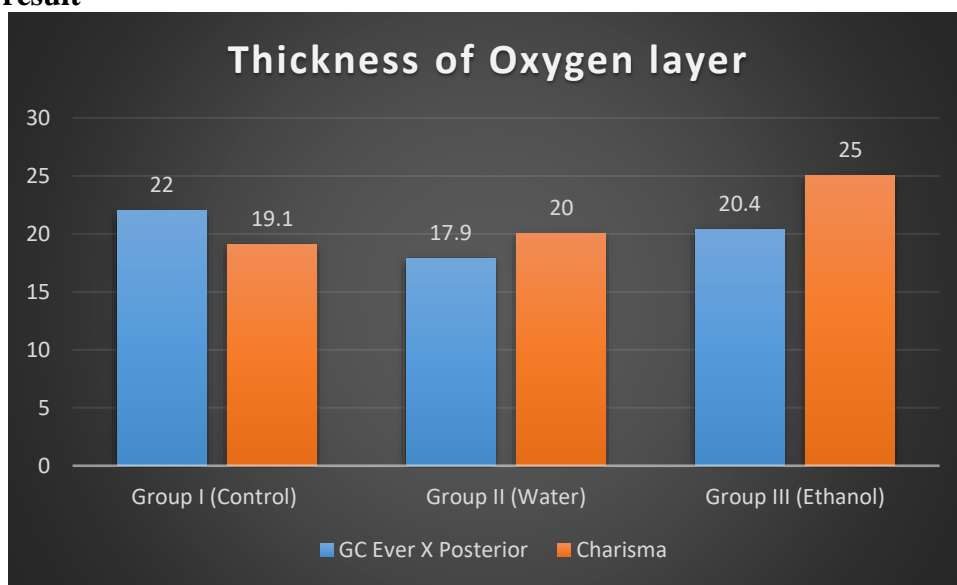


Table 1: Intra group comparison of thickness of oxygen inhibition layer of GC Ever X Posterior composite material in different solvents

GC Ever X Posterior composite material	Mean	SD	One way Anova F test	P value, Significance
Group I (Control)	22.0	1.33	F = 29.336	p < 0.001**
Group II (Water)	17.9	1.19		
Group III (Ethanol)	20.4	1.07		

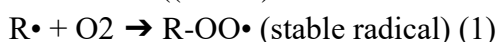
Table 2: Intra group comparison of thickness of oxygen inhibition layer of Charisma composite material in different solvents

Charisma	Mean	SD	One way Anova F test	P value, Significance
Group I (Control)	19.1	1.19	F = 70.126	p < 0.001**
Group II (Water)	20.0	0.81		
Group III (Ethanol)	25.0	1.49		

Discussion

The surface properties of resin composites, roughness, and microhardness have gained great clinical importance, as they are related to the esthetics and function of restorations. The absence of these properties results in periodontal disease and the development of secondary caries due to increased plaque accumulation and wear of the restoration, compromising long-term clinical success. Therefore, any restorative material should reproduce the biological, functional, and esthetic properties of a natural tooth. Commercial dental composites are random copolymers of 2,2- bis[4-(2-hydroxy-3-methacryloxypropoxy)phenyl]propane (Bis-GMA) and triethylene glycol dimethacrylate (TEGDMA), filled with various types of inorganic particles. Bis-GMA and TEGDMA are bi-functional methacrylate monomers that harden following a free-radical-induced

Polymerization reaction. While this type of reaction has several distinct advantages (fast reaction rates, high degrees of monomer conversion, and absence of solvents), it is strongly inhibited by free-radical scavengers such as oxygen (Xia and Cook, 2003). The inhibition resulting from oxygen diffusing from the atmosphere into curing resins is responsible for the inhibited surface layers commonly found on freshly polymerized unfilled resins (Finger *et al.*, 1996; Vallittu, 1999; Yatabe *et al.*, 2001). This is due to the oxidation of radicals into stable species known as peroxides (Reaction 1) (Andrzejewska *et al.*, 1998; Schulze and Vogel, 1998), which have low reactivity toward monomers. ((11111)



When composites are cured in air, in clinical practice, an oxygen inhibition layer is formed on the surface of the freshly cured composite resin. In the present study, the inhibition of polymerization by oxygen was determined as the thickness of the low-polymerized outer layer on composite resin specimens, which were cured in the presence of air and at ambient temperature.(4)

Ruyter (1981) reported that the thickness of unpolymerized surface films on resin following free-radical polymerization is a function of the resin viscosity, since the rate of oxygen diffusion in resin decreases with increasing viscosity of the liquid.(12)

Based on the principle of molecular interaction, the oxygen-inhibited layer improve the interfacial bonding between two contacting polymers.1

The oxygen-inhibited layer is primarily composed of unreacted monomers and oligomers, and it possesses a liquid-like consistency. This layer not only readily adopts the overlying material to increase the contacting area, it also allows the materials on both sides to cross the interface and blend together to form an interdiffused zone, where copolymerization can take place to produce a chemical bond. All of these actions would tend to strengthen the layer layer interaction.(2)

Eliades and Caputo explained their results by observing that the resin in the oxygen inhibited layer has the same composition as the uncured resin, except that the photoinitiator system, commonly camphorquinone (CQ) and amine, has been consumed or decomposed (2).

When a composite is reinforced with fibers, then the fibers and their orientation should also be considered as factors influencing the oxygen inhibition depth (27). The short-fiber composite used in the present investigation consisted of a cross-linked polymer network derived from dimethacrylate monomers (Bis-GMA and TEGDMA) and PMMA. During the polymerization process, this polymer matrix forms a semi-IPN structure (28). The differences between the short-fiber-reinforced composite and both dimethacrylate-based composites may be explained by the presence of fibers in short fiber-reinforced composite, which are shown to affect the oxygen inhibition depth (27) and the internal void space formation (29). Orientation of the fibers in the previous study favoured the passage of oxygen (27), but it should be noted that the short-fiber-reinforced composite used in this study had randomly oriented short fibers, with length varying between 1 and 2 mm, and their influence on the oxygen inhibition depth could be less than in composites with oriented fibers.(4)

Oxygen inhibition layer-free surfaces have been produced for research purposes by curing the specimens at elevated temperatures or air-free argon, nitrogen, or carbon dioxide atmospheres.

Water spray and ethanol treatments were used in the present study as momentary treatments for cleaning the surface of the oxygen inhibition layer, because they are clinically more practical methods. All dimethacrylate-based composites studied showed an insignificantly thicker oxygen inhibition layer after both treatments, which means that those treatments did not remove the oxygen inhibition layer from the exposed surface.(4)

Due to this, Both the composite material and the surface layer treatment may affect the interlayer shear bond strength.

Therefore, it can be suggested that the surface wettability provided by the oxygen inhibition layer is crucial for adhesion of the adherent surface. The ethanol and the water-spray treatments were applied for a controlled time of 20 s, and adverse bonding effects were not observed following either surface treatment. Cleaning the surface of the oxygen inhibition layer with ethanol and water-spray may have extracted some unpolymerized monomers. This might have influenced the bond strength, which was also evident in the more frequent occurrence of cohesive breaks observed with these groups. Predominantly cohesive fractures were observed for the ground short-fiber-reinforced composite surface (i.e. The oxygen inhibition layer was completely removed). This could be a result of the Micro-mechanical interlock between the monomer from the overlaying composite and the fibers of the underlaying composite, which were exposed during the grinding procedure.

In general, our results are in agreement with the majority of other studies, in which the presence of an oxygen inhibition layer with adequate thickness at the surface between the adjacent composite layers improved the interfacial bonding.

Conclusion

In conclusion, the oxygen inhibition layer, acting as an intermediate layer, is retained on the surface of the composite after treatment with either ethanol or water spray. The water-spray treatment would probably have no effect on removal of the oxygen inhibition layer, because of the oxygen dissolved in water, whereas treatment with ethanol could remove the oxygen inhibition layer, but probably a longer application time is needed. The present study shows results in accordance with the study of Koga *et al.*, which showed that both the physical and chemical surface properties of the OIL depend on its thickness if relatively thin, the OIL allows diffusion of the photoinitiator into the overlaying composite, thus improving the bond strength.(8)

The presence of an oxygen inhibition layer improved the interlayer shear bond strength of adjacent composite layers and led to more durable adhesion, whereas the absence of an oxygen inhibition layer adversely affected the bond strength and led to adhesive interfacial failures. Hence, the oxygen inhibition layer should

Be provided and left intact after polymerization.

Within the limitations of this study it is recognized that since it is an in vitro investigation, the obtained results could not be extrapolated to the clinical field; for this reason, it would be advisable to develop randomized clinical trials with the same proposed aim. It is recognized that the present study procedure may be different from a clinical situation, since changes in temperature, the presence of saliva, enzymes, and changes in pH could affect microhardness and surface roughness over time.(5)

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