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BASIC RESEARCH:

Mathematical-Physical Description of the Oxygen-Inhibited Layer (OIL) in Nanofilled Dental Polymers

Descripción físico-matemática de la capa inhibida por oxígeno en polímeros dentales nano rellenados

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ABSTRACT: This research evaluates the presence of the oxygen-inhibited layer (OIL) on the top surface of a photopolymerized dental composite resin protected with a glycerin layer. This evaluation was conducted using physical and mathematical methods. Polymerized discs were fabricated to evaluate Vickers microhardness (VHN), and pre-and post-polymerized samples were used for the calculation of C-O/C-H ratios through Fourier Transform Infrared Spectroscopy (FTIR) (n=10), using two types of glycerin, one for medical use (MG) and another for dental use (DG). Surface hardness decreased from MG to DG to CO, and the increase in C-O/C-H ratios decreased in the same order (p<0.05). Samples protected with medical and dental glycerin layers are harder and exhibit lower C-O/C-H bond ratios than the control group.

KEYWORDS: Polymers; Dental composite; Glycerol; Hardness test; FTIR.

RESUMEN: La presente investigación evalúa la presencia de la capa inhibida por oxígeno OIL en la última superficie fotocurada de una resina compuesta de uso odontológica que fue protegida con una capa de glicerina. Esta evaluación se hizo a partir de métodos físicos y matemáticos. Se fabricaron discos polimerizados para evaluar la microdureza Vickers (VHN) y pre y post polimerizados para el cálculo de tasas C-O/C-H por medio de Espectroscopía Infrarroja Transformada de Fourier (FTIR) (n=10) usando dos tipos de glicerina, una de uso médico (MG) y otra de uso dental (DG). La dureza superficial disminuyó de MG a DG a CO y el aumento de tasas C-O/C-H disminuyó en ese mismo orden (p<0,05). Las muestras

protegidas con capas de glicerina médica y odontológica son más duras y presentan menos cantidad tasas de enlaces C-O/C-H que el grupo control.

PALABRAS CLAVE: Polímero; Resina compuesta; Glicerol; Prueba de dureza; FTIR.

INTRODUCTION

Oxygen is a potent inhibitor of polymerization (1,2). The diffusion of oxygen into the resin during polymerization inhibits the propagation of the reaction by binding to free radicals, forming the oxygen-inhibited layer (OIL) (3). In dental resins oxygen can generate peroxyl radicals, which are less reactive for breaking double bonds, reducing the triggering effect of chemical or light-activated initiators resulting in a lower degree of conversion in this zone (4). The interaction described above induces the formation of the OIL, which presents a composition similar to that of uncured resins. Its thickness varies between 11.6 and 200 μm (4-9). This layer compromises several desirable characteristics of the final polymer such as surface hardness, wear resistance, and chromatic stability (10), and increases the possibility of elution of unreacted monomers in saliva, which can cause adverse reactions in the mucosa (11).

As polymerization progresses, the rate of free radical diffusion decreases causing some dimethacrylate molecules to remain unreacted, resulting in 25% to 50% of unpolymerized methacrylate groups (12). However, several studies suggest that the presence of an inhibited surface layer increases the bonding strength between composite resin increments (7,13-15). It is also suggested that this does not promote a significant increase in incremental bond strength (4,15) or even imparts a detrimental effect on the layer integrity compared to the surfaces without it (9). On the other hand, Xia and Cook mention that Bis-MGA and TEGDMA harden after the radical-induced attack during polymerization. Although this reaction is fast, monomer conversion is high, and there are no solvents present; it is also strongly inhibited by radical scavengers such as oxygen (14).

It is important to emphasize that the properties described above depend on the absence or presence of OIL and the formation of polymeric chains (16). Fourier-transform infrared spectroscopy (FTIR) examines the conversion of methacrylate-based polymers (17,18) and is considered a reliable technique to analyze the bonds before and after the chemical reaction of the materials, being an indirect and useful method to determine the degree of polymer conversion (14,19-21). For resinous materials, the intensity in the band at 1638 cm-1 can be measured and correlated with an internal reference peak between 1635 cm-1 and 1649 cm-1, whose intensity remains unchanged during the polymerization process, to monitor the intensity of the methacrylate C=C and between 1605 to 1610 cm-1 to monitor the intensity of the phenyl in absorbance (17). Additionally, readings of oscillations from 1310 cm-1 to 1250 cm-1 for the C-O bonds of aromatic ester compounds and between 3000 cm-1 to 2840 cm-1 for C-H bonds of alkane compounds have been reported (12). These readings are recorded in search of the wave intensity in the bonds of interest to confirm their presence in the study samples.

Based on it, we know which peaks correspond to the carbon double bonds, which are formed by the monomers before the initial polymerization reaction.

Initially, the decrease in the intensity of the methacrylate C=C absorbance (Ameth) at 1635-1640 cm-1 was monitored, and the phenyl absorbance (Aarom) at 1605-1610 cm-1 was used

as an internal standard (17). However, the percentage of double bonds that reacted (conversion of double bonds) did not represent the response to the hypothesis. The goal was to find out how much C-O bonds increased when the samples were polymerized.

This conversion calculation has been repeated in several studies where an approach to clinical protocol versus controlled laboratory protocol is attempted without success (22). The method has been exposed for many years, with Rueggeberg establishing in 1990 that the analysis methodology of conversion by infrared methods is achieved with the calculation of aliphatic C=C bonds at 1640 cm-1 and aromatic C=C bonds at 1608 cm-1 (8,17,23-26).

Another indirect method used to evaluate the polymerization of materials is through the measurement of surface microhardness, which allows an analysis of the polymer´s quality and degree of conversion based on surface properties (22) and the efficiency of light-curing processes of different polymeric materials (22,27-29). Studies have shown that inadequate polymerization can affect the hardness and roughness of the material and influence biofilm retention, favoring gingival inflammation, secondary caries formation, and superficial staining, thereby compromising the longevity of the restoration (30). In that regard application of glycerin has been recommended as a method to improve surface polymerization by inhibiting the formation of OIL in composite resins for direct restorations (6,10,31-33) and three-dimensional printing (34). Glycerin (C3H8O3) or glycerol 1,2,3-propanediol is a colorless, odorless, and viscous liquid alcohol. It is the main component of triglycerides found in animal fats and vegetable oils. It is also a derivative of the petrochemical industry and must be refined and purified for use in dentistry (35).

Therefore, given the clinical importance of minimizing the presence of the OIL, the purpose of this study is to evaluate the effect of different glycerin preparations when light-curing a dental resin.

The research hypotheses are 1. Using an intermediate layer of glycerin will produce differences in VHN and 2. Using an intermediate layer of glycerin will produce lower C-O/C-H bond ratios.

METHODOLOGY

For all experiments, prefabricated Teflon molds 12 mm in diameter by 2 mm in thickness were used to fabricate samples in a single increment on a glass slide with Filtek Z350 XT resin (3M ESPE®) and photocured with an LED curing unit with a peak emission of 450 nm and radiant output of 1505 mW/cm², (3M ESPE Elipar Deep Cure-L) for 20 s at a distance of 1 mm.

For all experiments, three groups were evaluated: (1) CO (control) specimens were cured without any intermediate layer, (2) MG specimens were cured through a layer of medical-grade glycerin (KY®), and (3) DG specimens were cured through a layer of dental-grade glycerin (Liquid Strip®). For all specimens, the surface opposing the curing light, to ensure that all measurements were made on the top surface of the disc. The characteristics of each material used as an intermediate layer are detailed in Table 1 and the materials used in the study are described in Table 2.

Table 1. Study groups.

Group	Name	Light curing
CO	Control	Direct light curing
MG	Medical glycerin	Light curing through a layer of medical glycerin
DG	Dental glycerin	Light curing through a layer of dental glycerin

Table 2. Products, manufacturers, and product composition.

Product	Liquid Strip® Glycerin	KY® Glycerin	Z350 XT A2D Composite
Manufacturer	Ivoclar Vivadent (Schaan, Liechtenstein)	Reckitt Benckiser Healthcare Manufacturing (New Jersey, USA)	3M ESPE (Minnesota, USA)
Product composition	Glycerin Water High-dispersion silicon dioxide High-dispersion aluminum oxide	Water Glycerin Hydroxyethyl cellulose Gluco-nolactone Methylparaben Sodium hydroxide Chlorhexidine gluconate	UDMA TEGDMA bis-EMA non-agglomerated/non-aggregated silica 20nm non-agglomerated/non-aggregated zirconia 4 to 11 nm aggregated cluster of zirconia 4 to 11 nm and silica 20 nm
Batch	Z00925	1186V2	N968258

MICROHARDNESS MEASUREMENTS

For microhardness analysis, the VHN of 10 specimens from each group was analyzed, for each specimen three measurements were made and the mean VHN of the specimen was calculated. The surface where the indentation was made was marked, and a visual inspection was carried out to find a smooth surface that would allow for quality indentation. All surface hardness tests were performed with a load of 19.6N for 20s (36) using a Vickers microhardness tester (Micromet® 2100, Buehler).

MEASUREMENTS OF C-O/C-H RATIOS WITH FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

To determine the ratio of C-O/C-H bonds, 10 specimens from each group were analyzed. Measurements were made with a Fourier Transform Infrared Spectrophotometer (Thermo Scientific Nicolet 6700 Waltham, Massachusetts, USA) with gold optics.

For all groups and based on Hook's law exposed in Silvesrtein's book (12), the peaks of

1113 cm-1 for C-O bonds and 2872 cm-1 for C-H bonds were chosen and recorded. C-O/C-H ratios were calculated before and after the polymerization of each study group to detect changes in the readings of the C-O bond with respect to the constant C-H bond, and with this data, the percentage increase in the intensity reading of the C-O bond was calculated.

STATISTICAL ANALYSIS

All statistical analyses were performed using the statistical software Minitab18 (State College, PA). The VHN and C-O/C-H rate data were analyzed. Normality and homoscedasticity (Anderson Darling, Levene test, respectively) and subjected to a one-way ANOVA (factor: type of glycerin) and Tukey post hoc ($\alpha=0,05$).

RESULTS

SURFACE HARDNESS

Mean VHN values for all groups are presented in Table 3. Regarding VHN statistical analysis showed significant differences between MG and DG, also between MG and CO, but not between CO and DG. The MG group presented a mean VHN of 119 ± 24.4, while the CO and DG groups presented a mean VHN of 68.9 ± 7.8 and 75.9 ± 12.03, respectively. These results suggest that using MG before light curing results in a higher VHN than CO and DG.

Of note, the difference between the CO and DG groups is not significant, indicating that both groups have a similar surface hardness.

DIFFERENCES IN C-O/C-H BOND RATES

Mean C-O/C-H rate values are presented in Table 3. The statistical analysis detected significant differences between the study groups ($p=0.023$). The Tukey analysis found significant differences between CO and MG. The MG showed the lowest C-O/C-H bond rate of 0.45, compared to the highest rate of 0.78 for CO.

The rate results indicate that the mean bond rate in CO samples was significantly higher than in MG samples with a 42% higher bond rate in the CO group. Additionally, there were no statistically significant differences between CO and DG or between the two groups polymerized through any type of glycerin.

Table 3. Average VHN, difference in bond rates followed by the standard deviation (in parentheses) corresponding to each measurement group CO, OG, and MG.

Group	VHN		Difference in bond rates	
CO	68,9 (7,8)	B	0,78 (0,35)	A
MG	119 (24,4)	A	0,45 (0,21)	B
DG	75,9 (12,03)	B	0,56 (0,17)	AB

DISCUSSION

The results suggest that using an intermediate layer of glycerin to isolate the final increment of RBC from the oxygen may increase the VHN and DC of RBCs. Nonetheless, the results are product dependent (37). Therefore, the first hypothesis stating that using an intermediate layer of glycerin will produce differences in VHN was accepted, because the use of MG resulted in significantly higher hardness than that obtained in the DG and CO groups. In our study, the most interesting finding is that using DG as an intermediate layer resulted in a VHN increase of only 9.2% compared to the control group which was not significant. At this point, results suggest that it is equally effective to photocure through an intermediate layer of glycerin manufactured for specific dental use as it is to photocure without any protection to stop the oxygen-free radicals present in the air that produces OIL formation. A previous study confirms that there were no significant differences in the surface hardness of samples protected with various methods to neutralize free oxygen, and other authors agree with this line of results regarding surface roughness (38,39).

Studies have tested various types of intermediate layers to protect the final increment of restorations with glycerin being the most accessible and immediate medium, although not necessarily the most effective (28,40). In the present study, it is noted that when the surface is not protected by an intermediate layer, the VHN was 42% lower than that of samples protected with medical-use glycerin (MG), consistent with studies that reiterate that the use of an intermediate layer during polymerization may improve the surface hardness of polymers (41,42). We also found a difference between MG and DG groups although some authors find that surface hardness depends on the type of material used (37).

The second research hypothesis stating that using an intermediate layer of glycerin will produce lower C-O/C-H bond ratios is accepted because when a polymer is protected with glycerin the increase in the C-O/C-H rate is lower than in polymers not protected by any physical intermediate layer. Although the rates of polymerized bonds through MG also increase, they do not do so significantly as in CO and DG. The amounts of C-O bonds are 73% higher in control samples, which have not been protected to prevent the action of oxygen as a free radical, than in samples that have been protected with MG, which, in turn, is 19,6% lower than samples protected with DG. The C-O/C-H rate is always lower in samples protected with MG.

The present investigation started from the fact that bonds change during polymerization, so the calculation of the C-O/C-H ratio was based on the variation of C-O bonds, versus the C-H bond that remains constant during polymerization.

Whenever comparisons were made between groups, the control samples showed an increase in the percentage of bond intensity, supporting the readings of differences in rates, where intensity readings are always lower when measured in samples protected with MG. This increase in bond intensity suggests that samples without intermediate polymerization barriers may have a lower degree of conversion on their surface, which is essential for optimal physical properties of the material and biocompatibility (43). Although in some cases the differences are not significant.

The measurements do not ignore the fact that it has been reported in several studies that total polymerization of materials is never achieved. Peutzfeldt has reported conversion degrees not greater than 75% since 1997, and other authors have even found low degrees of conversion ranging

from 30.6% to 47.4%, depending on the studied polymers (44,45).

In Marovik's study, there is an inverse correlation to our results, finding that polymers with higher conversion degrees are softer (43), while others do not find significant correlations between hardness and polymer conversion degree (37).

On the other hand, other authors indicate a correlation between OIL and adhesion strength, (46), and finally, those who have similar results to ours, where for a dental restorative resin, there is a good correlation between the increase in hardness and the increase in conversion degree during the setting reaction (47-49).

Research on the surface hardness and bond conversion rates of dental-use resins is of great clinical importance because these properties are directly related to the quality and durability of dental restorations.

The study demonstrating that the protection of the final resin layer with different types of glycerin increases surface hardness and decreases C-O bond rates is especially relevant, as it suggests that this process could improve the quality and longevity of dental restorations made with resins.

Additionally, this research could also have important implications in terms of the selection of materials and techniques used in daily clinical practice, as it could suggest changes in resin protection strategies that could significantly improve long-term restoration results. These findings could have a significant impact on the information available to the dental community, academia, and the quality of dental care, and therefore on the health and well-being of patients.

This study not only describes the quality of an OIL but also mathematically quantifies the existence of an oxygen-inhibited layer in polymers through the calculation of C-O bond rates.

A limitation of this study was the inability to visually identify the inhibited layer to characterize and measure it.

CONCLUSIONS

Applying glycerin can influence the surface hardness of polymers, the greatest increase was observed in MG.

Applying glycerin can influence the increase of the C-O/C-H bond rate, the smallest increase was observed in MG.

There is a statistically significant difference in VHN between MG and CO. There are no significant differences between CO and DG.

A significant difference in VHN was observed between MG and DG. The intensity of C-H/C-O bonds increased in all groups. However, the statistical difference was found only between CO and GM.

It is concluded that both hypotheses raised are accepted since the glycerin layer applied prior to final polymerization increases surface hardness and decreases the amount of carbon-oxygen bonds characteristic of the inhibited layer, thus improving the long-term quality of the restoration.

AUTHOR CONTRIBUTION STATEMENT

Conceptualization and design: I.F.G.
Literature review: I.F.G. and A.H.M.
Methodology and validation: I.F.G.
Formal analysis: I.F.G.
Investigation and data collection: I.F.G.
Resources: I.F.G.
Data analysis and interpretation: I.F.G. and A.H.M.
Writing-review and editing: I.F.G. and A.H.M
Supervision: I.F.G.
Project administration: I.F.G.

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CONFLICT OF INTEREST

No potential conflict of interest relevant to this article was reported.

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