The Effect of Oxygen-Inhibited Layer and Its Inhibition Technique on Diametral Tensile Strength Values of Various Nanofilled Composite Resin Types

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Submission date: 16-May-2024 12:47PM (UTC+0700)
Submission ID: 2380788124
File name: the_effect_of_oxygen_inhibited_layer_and_its.8_1.pdf (2.07M)
Word count: 5097
Character count: 28777

Original Research

The Effect of Oxygen-Inhibited Layer and Its Inhibition Technique on Diametral Tensile Strength Values of Various Nanofilled Composite Resin Types

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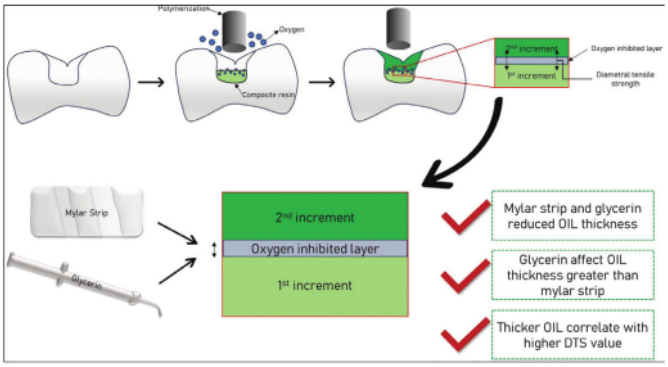
Abstract

Aim: To investigate the impact of different inhibitory techniques on the thickness of oxygen-inhibited layer (OIL) and diametral tensile strength (DTS) value in various types of nanofilled composite resins. **Materials and Methods:** Thirty-six nanofilled composite resins specimens, consisting of packable, flowable, high-viscosity bulk-fill (HVBF), low-viscosity bulk-fill (LVBF), shaped as half disk (diameter: 6mm and height 3mm) and randomly allocated to three groups ($n = 3$): with Mylar strip, glycerin application, and without OIL inhibitors. OIL thickness was observed with an optical microscope. Furthermore, 60 specimens of composite resins were incrementally created in disk-shaped molds (diameter: 6mm, height: 1.5mm × 2mm). DTS measurements were carried out using a universal testing machine. Data were statistically analyzed using a two-way analysis of variance (ANOVA) test and Pearson's correlation test ($P < 0.05$). **Results:** OIL inhibitor techniques (Mylar strip and glycerin) significantly affected OIL formation across various types of nanofilled composite resins ($P < 0.05$). Changes were also observed in how these techniques influenced DTS values. Correlation analysis indicated a positive relationship between OIL thickness and DTS value. **Conclusions:** Application of Mylar strip and glycerin reduced OIL thickness and DTS values in packable, flowable, HVBF, LVBF nanofilled composite resins. Glycerin proved to be more effective than Mylar strips in reducing OIL thickness, which is reflected in the DTS values of nanofilled composite resins. Greater OIL layer thickness on the outermost layer of the nanofilled composite resin correlated with a higher DTS value.

Graphical Abstract



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Access this article online

Quick Response Code:

Website: www.jioh.org

DOI: 10.4103/jioh.jioh_198_23

How to cite this article: Hadi S, Tjandrawinata R, Fibryanto E. The effect of oxygen-inhibited layer and its inhibition technique on diametral tensile strength values of various nanofilled composite resin types. J Int Oral Health 2024;16:150-7.

Keywords: Composite Resin, Diametral Tensile Strength, Oxygen-inhibited Layer

Received: 23-Aug-2023, **Revised:** 15-Jan-2024, **Accepted:** 22-Jan-2024, **Published:** 29-Apr-2024.

Introduction

Composite resin constitutes one of several dental materials that may be utilized for restoration.[1] Tooth-colored restoration such as composite resin can be applied to the anterior and posterior teeth.[1,2] Composite consists of two or more materials with distinct characteristics.[3] Composite resins include a resin matrix or monomers, inorganic fillers or fillers, coupling agents, initiator-activator systems, and coloring agents.[1,4] The most recent advancement in nanotechnology is in resin-based composite materials with more favorable mechanical properties, namely nanocomposites.[5] Nanofilled composite resins have a minimum filler size of about 40 nm silica dioxide and/or zirconium dioxide, resulting in a highly smooth surface and more resistance to discoloration.[6]

Many of the mechanical characteristics of composite resins are crucial to the material's durability including compressive strength, tensile strength, bond strength, and hardness. This mechanical force generated during the chewing process has a significant impact on the longevity of the composite resin in the cavity.[7] However, when polymerization is performed on composite resins in the presence of oxygen in the atmosphere, a sticky superficial layer develops on the outermost layer of the resin, delaying or inhibiting the polymerization reaction and forming an oxygen-inhibited layer (OIL).[8-10] Earlier studies have demonstrated that the presence of OIL could either reinforce, decrease, or have an insignificant effect on the shear bond strength of composite resins.[11-13] Clinical approaches used to decrease the formation of OIL layers include the use of Mylar strips, glycerin between composite resin layers before polymerization, and polishing, which acts as a physical barrier and improves the degree of conversion (DC).[6,14,15]

A reduced DC can result in numerous unreacted monomers, affecting the composite resin's physical, chemical, and mechanical properties.[16,17] One factor influencing the DC of composite resins is the material's viscosity, which is proportional to the amount of matrix resin and inorganic filler.[18-20] Flowable composites contain a more significant proportion of matrix resin than traditional composite resins.[18] The lower or higher viscosity of the composite resin may affect DC during polymerization, thereby influencing the mechanical properties of the composite resin.[19-22] Bulk-fill composite resin, which features a novel matrix component, has been developed. The matrix component of bulk-fill composite resins has longer molecular bonds and shorter intermonomer distances, resulting in decreased volume shrinkage during polymerization.[18,23] Growing number of composite resins with varying compositions calls for more research. No previous research compared the effect of OIL in various nanofilled composite resins on the bond strength of composite resins as measured by diametral tensile strength (DTS) values.

Based on the background of the subject's overview, the objective of this research was to:

- investigate the impact of OIL inhibition techniques (Mylar strip and glycerin) on OIL thickness formation in various nanofilled composite resins (packable, flowable, high-viscosity bulk-fill [HVBF], and low-viscosity bulk-fill [LVBF] resin composite);
- investigate the impact of different OIL inhibition techniques (Mylar strip and glycerin) on the DTS value of various nanofilled composite resins (packable, flowable, HVBF, and LVBF resin composites); and
- determine the relationship between OIL thickness and DTS value.

Materials and Methods

This is *in vitro* laboratory-based experimental research with a posttest-only control group design. The investigation was conducted in the Dental Materials and Testing Center of Research (DMTCore) Laboratory, Trisakti University, Jakarta, Indonesia. The sample size for this study was calculated using the following formula for unpaired numerical and analytical research:[24]

$$n_1 = n_2 = 2\left(\frac{(Z\alpha + Z\beta)S}{\bar{x}_1 - \bar{x}_2}\right)^2$$

Analysis of OIL formation on four nanofilled composite resins using three samples per group. The total number of specimens required for the three categories of OIL inhibition techniques was 36. In addition, five specimens per group were used for the DTS analysis of four distinct nanofilled composite resin types. A total of 60 specimens are required for the three categories of OIL inhibition techniques.

Sample preparation and measurement of oxygen-inhibited layer formation

Thirty-six specimens were prepared in half disk-shaped stainless steel molds with a diameter of 6 mm and a height of 3 mm. Twelve specimens for each type of composite resin: packable (Filtek™ Z350 XT Universal Restorative, 3M ESPE, St. Paul, MN, USA), flowable (Filtek™ Z350 XT Flowable Restorative, 3M ESPE), HVBF (Filtek™

Table 1: Difference in means and significance of OIL thickness between composite resin type groups

Nanofilled Composite Resin	Flowable		HVBF		LVBF	
	P value	Difference in means	*P* value	Difference in means	*P* value	Difference in means
Packable	<0.001*	3.1133	<0.001*	1.1460	<0.001*	2.9507
Flowable	-	-	<0.001*	-1.9673	0.921	-0.1627
HVBF	-	-	-	-	<0.001*	1.8047

DTS=diametrical tensile strength, HVBF=high-viscosity bulk-fill, LVBF= low-viscosity bulk-fill, OIL=oxygen-inhibited layer
*Tukey ($p< 0.05$)

One Bulk Fill Restorative, 3M ESPE), LVBF (Filtek™ One Bulk Fill Restorative, 3M ESPE), and LVBF (Filtek™ Bulk Fill Flowable Restorative, 3M ESPE) were grouped into three distinct categories at random ($n = 3$) according to each composite resin type.

Group I was the group treated with 0.05 mm Mylar strip (Polyester Matrix Strip, Tdv, Pomerode, Brazil), Group II was the group treated with glycerin Oxygen Barrier Gel: DeOx (Ultradent, South Jordan, UT, USA), and Group III was the negative control (no OIL inhibitor). The specimens were polymerized for 20 s at a distance of 1 mm with a light-emitting diode (LED) light-curing unit with an intensity of 1200 mW/cm^2 (LY-B200, Guangdong, China). The specimens were then measured for OIL thickness at 10× magnification using an optical microscope (Shimadzu, Tokyo, Japan).

Sample preparation and diametral tensile strength test

Sixty specimens were created in a disk-shaped stainless-steel mold of two composite layers with a diameter of 6 mm and a height of 1.5 mm for each layer. Each layer's height was measured using a UNC-15 instrument (ASA Dental, Massarosa, Italy). Fifteen specimens of each composite resin type: packable (Filtek™ Z350 XT Universal Restorative, 3M ESPE), flowable (Filtek™ Z350 XT Flowable Restorative, 3M ESPE), HVBF (Filtek™ One Bulk Fill Restorative, 3M ESPE), and LVBF (Filtek™ Bulk Fill Flowable Restorative, 3M ESPE) were randomly divided into three groups ($n = 5$).

The specimens were made by inserting the first layer of composite resin into the mold using a plastic filling instrument (LM-ErgoSense, Parainen, Finland), and then irradiating them for 20 s at a distance of 1 mm using an LED light-curing unit with an intensity of 1200 mW/cm^2 (LY-B200). Afterward, the composite resin specimens were arbitrarily divided into three groups. Before polymerizing the first layer in Group I, the specimens were treated with an OIL formation inhibitor using a Mylar strip. The second layer of composite resin with a thickness of 1.5 mm was applied. Before the first layer was polymerized in Group II, the specimens were treated with an OIL formation inhibitor using glycerin and then polymerized. No OIL inhibitor was applied in the control group, so the specimens were polymerized immediately, and the next layer of composite resin was added and polymerized for 20 s at 25°C. The specimens were finally examined for DTS using a universal testing machine (AGS-X 5 kN, Shimadzu) at a 1 mm/min speed. The following formula computed the DTS value:[25]

$$T = \frac{2P}{\pi DT}$$

where T is the DTS (MPa); P is the load applied to the specimen (N); π = 3.1416; D is the specimen diameter (mm); and T is the specimen thickness (mm)

Statistical analysis

The normality of DTS value data was assessed using the Shapiro-Wilk test and Levene's test was utilized to assess data homogeneity ($P > 0.05$). The Shapiro–Wilk test was used to assess OIL thickness ($P > 0.05$) and Levene's test was utilized to assess data homogeneity ($P > 0.05$). A two-way ANOVA test ($P < 0.05$) and Tukey's *post hoc* test ($P < 0.05$) were used to analyze data on OIL thickness and DTS values. The connection between OIL thickness and DTS was evaluated using Pearson correlation ($P < 0.05$). The SPSS Statistical 26 software (SPSS Inc, Chicago, IL, USA) was used to analyze the data.

Results

Two-way ANOVA analysis demonstrated a significant effect of OIL inhibition techniques (Mylar strip and glycerin) on the formation and thickness of OIL in various nanofilled composite resins ($P < 0.001$). Tukey's *post hoc* test indicates that the OIL thickness of composite resin without OIL inhibitor (negative control) was significantly thicker than composite resin covered with Mylar strips($P < 0.001$) and composite resin applied with glycerin ($P < 0.001$). Furthermore, the OIL thickness of the composite resin covered with Mylar strips was significantly greater than the composite resin applied with glycerin ($P < 0.001$).

The OIL thickness of packable was more significant than flowable, HVBF, and LVBF, as determined by Tukey's *post hoc* test ($P < 0.001$). The OIL thickness of the flowable was significantly less than HVBF ($P < 0.001$). However, the difference was not of statistical significance from the

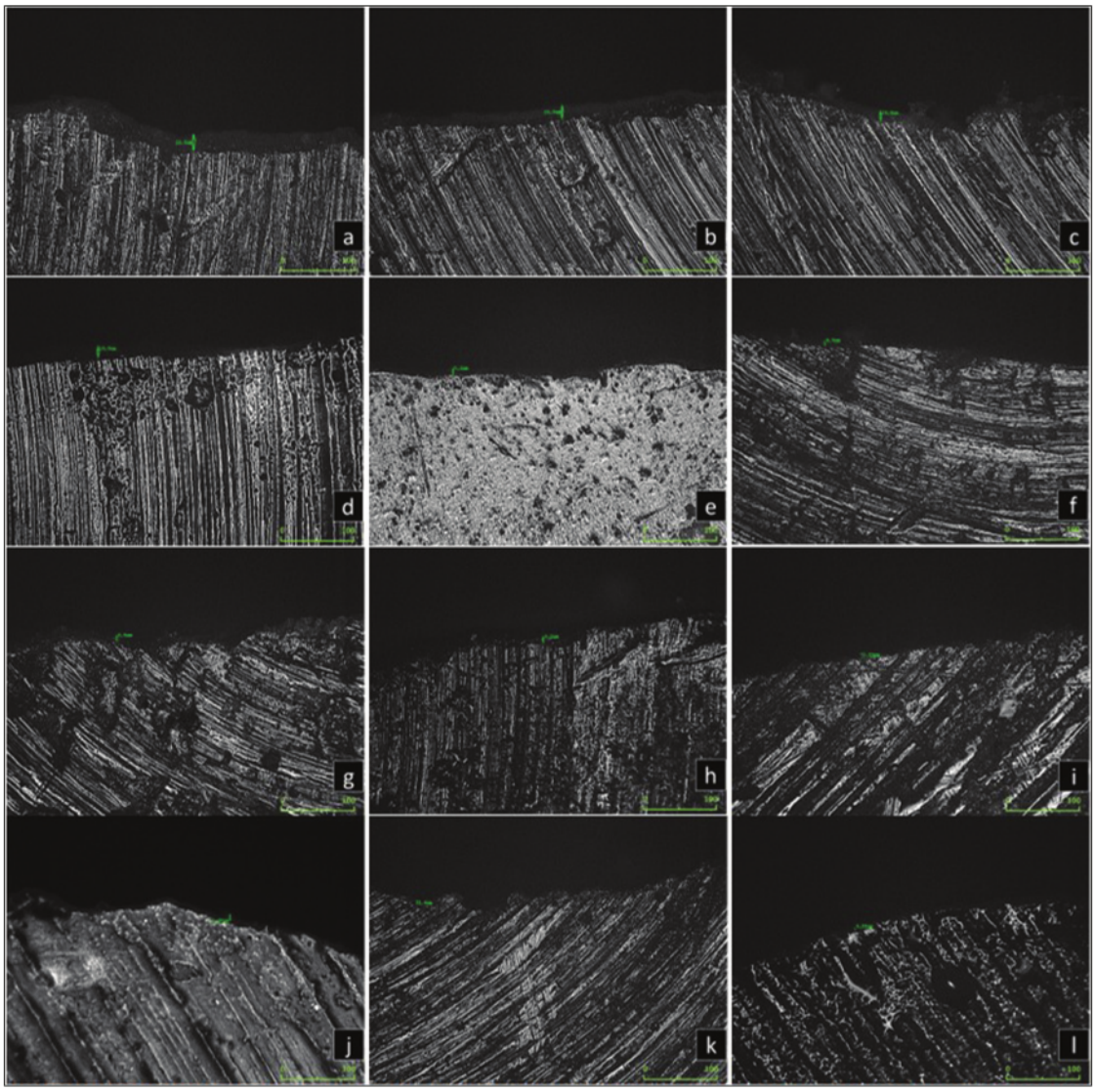

Figure 1: Evaluation of oxygen-inhibited layer (OIL) thickness (green arrows) using a microscope (10×) on different composite resin types with various OIL inhibition techniques. (A–D) OIL thickness of packable, flowable, high-viscosity bulk-fill (HVBF), and low-viscosity bulk-fill (LVBF) composite resin types, respectively, without OIL inhibitor. (E–H) OIL thickness of packable, flowable, HVBF, and LVBF composite resin types, respectively, coated with Mylar strip. (I–L) OIL thickness of packable, flowable, HVBF, and LVBF composite resin types, respectively, coated with glycerin

LVBF (P = 0.921). In addition, the OIL thickness of HVBF is significantly greater than LVBF [Table 1].

The results of OIL thickness observation under an optical microscope with 10× magnification are shown in Figures 1 and 2. Figure 2 shows the OIL thickness of various composite resins in each group. In the group treated with Mylar strip, HVBF composite resin had the thickest OIL (8.78 ± 0.60 μm), followed by LVBF (7.82 ± 1.18 μm), packable composite resin (7.02 ± 0.59 μm), and flowable (4.22 ± 0.66 μm). In the group treated with glycerin, HVBF composite resin had the thickest OIL (5.86 ± 0.76 μm), followed by LVBF (4.76 ± 0.49 μm), packable (4.33 ± 0.33 μm), and flowable (2.60 ± 0.28 μm). In the control group without OIL inhibitor, packable composite resin had the thickest OIL (21.76 ± 2.39 μm), followed by flowable composite resin (21.49 ± 2.99 μm), HVBF (15.49 ± 1.68 μm), and LVBF (14.85 ± 1.50 μm).

Two-way ANOVA analysis showed an effect of different composite resin types with various OIL inhibition techniques on DTS values (P = 0.002). Tukey's *post hoc* test indicates that the DTS test results for composite resins without OIL inhibitors were significantly higher than the

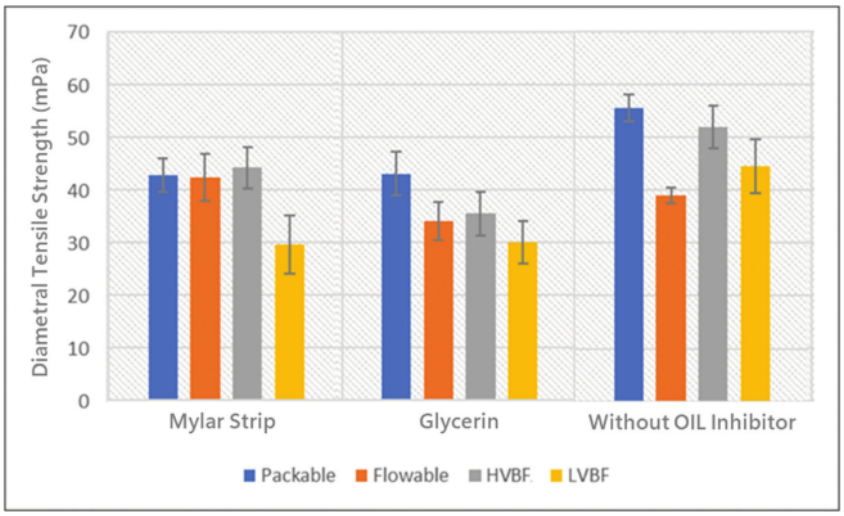

Figure 2: Mean values of oxygen-inhibited layer (OIL) thickness based on various types of composite resins in each group. Packable: Filtek Z350 XT Universal Restorative; Flowable: Filtek Z350 XT Flowable Restorative; HVBF: Filtek One Bulk Fill Restorative; and LVBF: Filtek Bulk Fill Flowable Restorative

Table 2: Difference in means and significance of DTS values between OIL's inhibition technique groups

OIL's Inhibition Technique	Coated with Mylar Strip		Coated with Glycerin	
	P value	Difference in means	*P* value	Difference in means
Without OIL inhibitor	<0.001*	7.9250	<0.001*	11.9063
Coated with Mylar Strip	-	-	0.021*	3.9813

DTS=diametrical tensile strength, OIL=oxygen-inhibited layer
*Tukey ($p < 0.05$)

Table 3: Difference in means and significance of DTS values between composite resin type groups

Nanofilled Composite Resin	Flowable		HVBF		LVBF	
	P value	Difference in means	*P* value	Difference in means	*P* value	Difference in means
Packable	<0.001*	9.4544	0.325	2.8280	<0.001*	12.6680
Flowable	-	-	<0.001*	-6.6264	0.221	3.2136
HVBF	-	-	-	-	<0.001*	9.8399

DTS=diametrical tensile strength, HVBF=high-viscosity bulk-fill, LVBF= low-viscosity bulk-fill
*Tukey ($p < 0.05$)

DTS test results for composite resins coated with Mylar strips and glycerin ($P < 0.001$). In addition, the results of the DTS test on composite resins coated with Mylar strips were significantly superior to those of composite resins containing glycerin [Table 2].

Tukey's *post hoc* test was performed between different groups of composite resin types on DTS, which revealed that the DTS test results for packable composite resins showed significantly greater than those for flowable and LVBF ($P < 0.001$). Simultaneously, there was no discernible difference compared to HVBF ($P = 0.325$). In addition, the DTS test result for flowable composite resin was substantially lower than that of HVBF ($P < 0.001$); however, did not differ significantly from that of LVBF. In addition, the DTS test results for HVBF were markedly superior to LVBF [Table 3].

DTS test data of each specimen group are demonstrated in Figure 3. In the Mylar strip group; it was found that HVBF composite resin had the highest DTS value (44.14 ± 3.97 MPa), followed by packable (42.83 ± 3.17 MPa), flowable (42.40 ± 4.44 MPa), and LVBF (29.55 ± 5.48 MPa) composite resins. In the glycerin group, it was found that the packable composite resin had the highest DTS (43.02 ± 4.18 MPa), followed by HVBF (35.48 ± 4.16 MPa), flowable (33.99 ± 3.63 MPa), and LVBF (30.03 ± 3.99 MPa). The negative control group showed that packable composite resin had the highest DTS value (55.53 ± 2.6 MPa), followed by HVBF

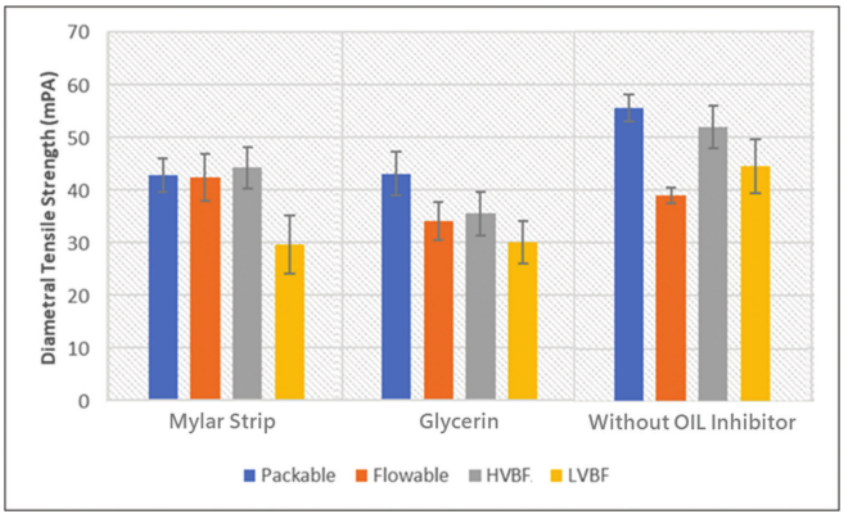

Figure 3: Mean diametral tensile strength values of composite resin types with various oxygen-inhibited layers (inhibition techniques

Table 4: Association between OIL thickness and the DTS value of composite resins

Source of variation	Correlation value	*P* value
OIL thickness × DTS value	0.559	<0.001*

DTS = diametral tensile strength, OIL = oxygen-inhibited layer
*Pearson ($P < 0.05$)

(51.97 ± 4.01 MPa), LVBF (44.49 ± 5.12 MPa), and flowable (38.93 ± 1.52 MPa).

The association that exists between OIL thickness and the DTS value of composite resins was determined using Pearson's correlation test [Table 4]. The findings of the Pearson's correlation test revealed a significantly positive relationship between OIL thickness and DTS value (r = 0.559; $P < 0.001$). The DTS value increases as the OIL layer thickness increases.

Discussion

OIL is a thick, viscous layer of unpolymerized resin that persists on the outermost layer after polymerization when a composite resin is exposed to oxygen in the air. Even though incremental techniques have been recommended to decrease polymerization shrinkage and increase the DC of composite resins; however, there is still no conclusive evidence regarding the effect of OIL and different types of composite resins on the bond strength of the composite resin increments themselves.[26]

It has been hypothesized that the formation of OIL on the outermost layer of the composite resin may increase the bonding strength between layers of the composite resin since OIL can disrupt the interfacial homogeneity to expand the contact area. As a result, upon the addition of another increment of composite resin, OIL will form an interdiffusion zone, which, when copolymerized, will form a chemical bond. These reactions are said to strengthen the bond between composite resin layers.[26]

This study demonstrates that in the absence of an OIL inhibitor, OIL tends to be more viscous than Mylar or glycerin. Before polymerization, Mylar strip and glycerin could be a physical barrier to OIL formation, as stated by other researchers. This study also demonstrates that composite resin coated with Mylar strip has a thicker OIL than glycerin.[6,8,14,15,26] This contradicts previous research that Mylar strips prevent atmospheric oxygen from reaching the restoration surface and that only oxygen within the composite resin contributes to OIL formation.[11,26] When glycerin is applied to the composite surface, the amount of oxygen in the glycerin, combined with the oxygen already present on the composite surface, can produce superior OIL formation compared with the Mylar strip.[27] The difference in results could be due to improper placement of the Mylar strip or ethanol to remove the glycerin. Although the authors have previously investigated the effect of various OIL inhibiting techniques on OIL thickness and found that ethanol has no significant effect, ethanol may eliminate some of the unpolymerized monomers.[28]

The results of this research indicate that composite resins with higher viscosities (packable and HVBF) have thicker OIL than lower viscosities (flowable and LVBF). This

result may be related to the composite resin material's DC. The DC of composite resin is significantly affected by the material's viscosity, which is proportional to the amount of matrix resin and inorganic filler. A material with lower viscosity contains a more significant proportion of resin matrix.[18,19,22] This investigation utilized composite resins with a DC of 52% (packable), 59% (flowable), 66% (HVBF), and 72% (LVBF).[27,29] The lower DC of the composite resins with higher viscosities (packable and HVBF) relative to the lower viscosities (flowable and LVBF) may lead to many unreacted monomers.[16,17]

The OIL thickness of conventional composite resins is thicker than that of bulk-fill whereas flowable composite resins are contrary. This is due to the elevated DC and photoreactivity of bulk-fill compared to conventional ones.[16,18] Higher DC results in less unreacted monomer and high polymerization stress; however, this does not occur in bulk-fill composite resins due to the chemically modified monomer structure due to changes in the composition of monomers and organic matrix of the composite resin, which reduces the shrinkage stress during polymerization by as much as 70%).[18,23,30]

This research also compared the DTS value of composite resins to various OIL inhibitor techniques. In contrast to the Mylar strip and glycerin groups, the composite resin group without OIL inhibitors had the highest DTS value. This result is consistent with other researchers' findings that composite resins without OIL inhibitors with thicker OIL have higher bond strength values than composite resins treated with OIL inhibitors.[28] In addition, it was also found that composite resins coated with Mylar strips had lower DTS values than glycerin. This result is consistent with the findings regarding OIL thickness, which indicate that the composite resin coated with Mylar strip has a greater OIL thickness than glycerin. Other studies have shown that glycerin could also function as a separating medium and have an impact on the micro tensile bond strength of composite resins.[31]

DTS values were also compared between composite resins. The results indicate that composite resins with higher viscosities (packable and HVBF) have higher DTS values than those with lower viscosities (flowable and LVBF). Monomer reactivity, material viscosity, and OIL affect the bonding strength between composite resin increments. Consistent with this research, other studies employing composites with triethylene glycol dimethacrylate diluent will produce higher viscosity, resulting in thicker OIL formation with greater bond strength values. On the other hand, composites containing low-viscosity resins will produce thinner OILs with weaker bond strengths.[14,32]

Recommendation

Furthermore, research is needed to compare OIL's thickness with the bond strength between composite resins using intervention techniques that can replace all oxygen (such as argon) and utilizing better OIL imaging techniques.

Conclusions

Mylar strip and glycerin could decrease the OIL thickness and DTS value of packable, flowable, HVBF, and LVBF nanofilled composite resins. The DTS value of nanofilled composite resin reflects that glycerin could reduce the OIL's thickness more than Mylar strips. The DTS value increases with the OIL layer's thickness on the outermost layer of the nanofilled composite resin.

Acknowledgements

This manuscript has not been published elsewhere and is not under consideration by another journal. We have approved the manuscript and agree with submission to the *Journal International of Oral Health*. All authors have made substantive contributions to this study and/or manuscript, and all have reviewed the final paper before its submission. There are no conflicts of interest to declare.

Financial support and sponsorship

Self-funded.

Conflicts of interest

There are no conflicts of interest.

Author contributions

S and EF contributed to concepts, design, definition of intellectual content, literature search, data acquisition, statistical analysis, article preparation, and article editing. RT contributed to concepts, design, definition of intellectual content, literature search, data acquisition, article preparation, and article review.

Ethical policy and Institutional Review Board statement

None.

Patient declaration of consent

None.

Data availability statement

Data are available on valid request by contacting the corresponding author.

List of Abbreviations

OIL: Oxygen-inhibited layer
DTS: Diametral tensile strength
DC: Degree of conversion
LED: Light-emitting diode
HVBF: High-viscosity bulk-fill
LVBF: Low-viscosity bulk-fill

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