Aarti C. Panchal, Geeta Asthana
Department of Conservative Dentistry and Endodontics, Government Dental College and Hospital, Ahmedabad, Gujarat, India

Abstract
Objective: This study aimed to evaluate the thickness of oxygen inhibition layer (OIL), produced on various composite materials, and to compare their interlayer shear bond strength (SBS), by treating the OIL with various agents.

Materials and Methods: The thickness of OIL of three different composite materials (120 specimens divided into three groups) Group 1 – Ivoclar Tetric N-Ceram (nanohybrid composite), Group 2 – Ivoclar Te Econom Plus (microhybrid composite), and Group 3 – GC EverX Posterior (a short glass fiber-reinforced composite) was evaluated. Each group was divided into four subgroups (A, B, C, and D) depending on the surface treatment given – no surface treatment (control group), ethanol, water spray, and grinding with SiC paper. This was followed by interlayer SBS testing.

Statistical Analysis: The data were statistically analyzed using ANOVA at a significance level of $P < 0.05$. Tukey’s post hoc analysis was performed following ANOVA to determine differences among the groups.

Results: The control group showed higher SBS irrespective of the type of composite material. The group treated with SiC paper resulted in the lowest interlayer SBS among all groups. Glass fiber composite showed higher interlayer SBS compared to both nanohybrid and microhybrid composites, irrespective of the surface treatment given.

Conclusion: The OIL, which acts as an intermediate layer, is retained on the surface of the composite even after treatment with ethanol and water spray. The presence of an OIL improved the interlayer SBS of two adjacent composite layers and led to more durable adhesion. Rather, the absence of an OIL adversely affected the bond strength and led to adhesive interfacial failures.

Keywords: Failure mode; oxygen inhibition layer; shear bond strength; surface treatment

INTRODUCTION

The composite resin material has become an integral part of modern restorative dentistry. Oxygen inhibition layer (OIL) is a sticky, resin-rich uncured layer and always present when a composite or bonding resin is polymerized in air. 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. 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.

Address for correspondence:
Dr. Aarti C. Panchal, 5370, Viththalwadi, Tarsadi, Kosamba, Surat - 394 120, Gujarat, India.
E-mail: draartipanchal@gmail.com

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Composites are becoming more durable with advances in the filler particles, monomer matrices, improved adhesive systems, and polymerization devices. Differences in the thickness of OIL have been observed among various dimethacrylate-based composites. This could be due to the filler content and its type and also network density of the resin composite. 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. For years, the dental community has held a common perception that an OIL is required before adding more layers of bonded composite. Reports on how the OIL affects bond strength have been inconsistent, despite many efforts dedicated to the subject.

The present study was conducted to evaluate OIL, produced on different composite material, and to compare their interlayer shear bond strength (SBS), by treating the OIL with various agents.

**MATERIALS AND METHODS**

**Evaluation of the thickness of oxygen inhibition layer**

An equal amount of each type of composite resin (Ivoclar Tetric N-Ceram [nanohybrid composite], Ivoclar Te Econom Plus [microhybrid composite], and GC EverX Posterior [a short glass fiber-reinforced composite]) was applied onto the middle of a horizontally placed glass microscope slides. A constant sample thickness of 0.1 mm was ensured by placing two glass plates (20 mm × 20 mm), as spacers, on each side of the composite resin, which was covered by another microscope slide as a coverslip. The specimens were polymerized through the coverslip for 40 s using a light-emitting diode light-curing unit. This method secured the formation of the OIL only at the outer sides of the specimens because air–resin contact was possible only at the resin boundary between the polymerized composite material and the spacers.

The depth of the OIL was measured along the periphery of each specimen using a stereomicroscope at a magnification of ×40, with a calibrated disk. Micrographs were taken of each specimen using the computer imaging program, to provide a visual record of the inhibition layer as shown in Figure 1.

**Preparation of the specimen for interlayer shear bond strength**

A total number of 120 specimens were prepared for this study, which were divided into three groups of 40 specimens each (Group-1 nanohybrid, Group-2 microhybrid, and Group-3 short glass fiber-reinforced) [Table 1].

Each of the three groups was further subdivided into four subgroups A, B, C, and D according to the surface treatments given to the OIL.

A. Control group – No surface treatment was given to OIL.

B. Ethanol group – OIL was treated by wiping it from the surface of the cured specimen using alcohol sponges soaked in 99 wt% ethanol for 20 s and then gently air dried for another 20 s.

C. Water spray group – OIL was treated with water applied as a water spray for 20 s from a distance of ~5 mm.
perpendicular to the specimen surface and then gently air dried for another 20 s
D. SiC paper – OIL was treated by grinding the surface with 1000-grit SiC paper.

OIL thickness was again measured in all the four subgroups – A, B, C, and D after the surface treatments were given to the OIL, and for the SBS testing, the specimens were further prepared.

For specimen preparation, acrylic resin blocks were made, in which round retentive cavity of 5 mm diameter and 3 mm depth was made. The same restorative material was used as the substrate and the adherent material.

Following polymerization, surface treatment with various agents was done. The adherent material was applied onto the substrate in an increment of 2 mm using a translucent polyethylene mold with an inner diameter of 3.5 mm and polymerized for 40 s.

Storage and thermocycling of specimens
Half the specimens were stored dry at 37°C for 7 days and then tested for interlayer SBS.

The other half were stored in distilled water for 48 h at room temperature (23 ± 1°C) and then thermocycled (alternating immersion of the samples in distilled water of a temperature of 5° and 55°C) for 6000 cycles, with a dwell time of the 30 s and a transfer time of 5 s. The specimens were then tested for interlayer SBS.

Testing of interlayer shear bond strength
The SBS test was performed using a universal testing machine at room temperature (23 ± 1°C). The specimens were mounted in a mounting jig and a circular edge blade created the shear type load positioned over the interface between the substrate and the adherent material at a crosshead speed of 1.0 mm/min until fracture. The shear load at failure was recorded as shown in Figure 2.

To analyze and to determine the fracture type, all fractured surfaces were visually examined under light microscopy at a magnification of ×40.

RESULTS

Oxygen inhibition layer thickness
The optically measurable thickness of the OIL was evaluated for different composites used in the study. The results are presented in Table 2. SGFR composite (Group 3) showed a maximum thickness of OIL, as compared to other composites (Group 1 and Group 2).

Two-way ANOVA showed that both the type of composite material and the surface treatment given, had significant effects (P < 0.001) on the thickness of the OIL.

Interlayer shear bond strength
The results of the SBS investigation between incrementally placed composite layers are presented in Graph 1.

A Three-way ANOVA (material type, surface treatment, and storage condition) revealed that both, type of composite material and the surface treatment given, had significant effects on the interlayer SBS (P < 0.001).

The difference between the dry and the thermocycled specimens was not statistically significant (P > 0.001).

DISCUSSION

Dental composites get cured by free-radical-induced polymerization reaction which is strongly inhibited by free-radical scavengers such as oxygen in the air. This free oxygen in contact with composite resin diffuses and inhibits polymerization reaction forming peroxide radicals that have low reactivity toward monomers.[1] This free monomer layer remaining on the surface after curing is known as the oxygen-inhibited layer.

\[ \text{R} + \text{O}_2 \rightarrow \text{R} – \text{OO} \text{ (stable radicals).} \]
The present study shows results in accordance with the study of Koga et al.,\textsuperscript{18} 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.

Water spray and ethanol treatments used in this present study are clinically more practical methods. They were applied for a controlled time of 20 s, and adverse bonding effects were not observed following either surface treatment.\textsuperscript{19}

The OIL thickness of SiC paper-treated groups was assumed to be zero, as the grinding procedure removed the OIL as confirmed microscopically. The mechanical polishing process not only removes the original surface of the composites but also removes the silane layer on the filler surface of composites which reduces the bond strength between increments due to inadequate wetting of resin to the fillers as discussed by Vankerckhoven et al.\textsuperscript{20}

The composite material and the surface layer treatment, both, affected the interlayer SBSSs. The outcome for both dimethacrylate-based composites (nanohybrid and microhybrid) was improved interlayer SBS when the OIL was present. This finding supports the influence of the physical surface properties of the OIL on the bond strength between incrementally placed composite layers and also its influence on the failure mode. Therefore, it can be suggested that the surface wettability provided by the OIL is crucial for the adhesion of the adherent surface. Factors such as the surface free energy of the solid and the surface tension of the liquid influence wettability.\textsuperscript{18}

Among the three groups, Group 3 showed a higher interlayer bond compared to both nanohybrid and microhybrid composites irrespective of any surface treatment and storage condition. The above result can be explained in the context of the chemical composition of each composite [Table 1].

The type of composite material and surface treatment given had significant effects on failure mode too. Surface treatment of OIL with ethanol and water spray may have extracted some unpolymerized monomers, which, in turn, influence the bond strength, which is evident as cohesive breaks, observed among all groups. Predominantly, cohesive fractures were observed in SGFR composites, where surface treatment was done with SiC, possibly due to micromechanical interlock between monomer from overlaying composite and fibers of underlying composite exposed during the grinding procedure. The SiC treated all groups resulted in the lowest interlayer bond strengths.

As multilayer techniques are recommended to minimize polymerization shrinkage as well as to increase the degree of conversion of dental composites, therefore, the interlayer bond strength achieved becomes an important consideration. Some studies on how the OIL affects bond strength described a positive correlation, indicating that the OIL increased bond strength.\textsuperscript{16} Kim et al. in 2006 concluded that when the OIL was missing or too thin, the absence or shortage of the unreacted monomers could have failed to connect both sides chemically and also failed to resist the shrinkage stress.\textsuperscript{17}

Among the three groups, SGFR composite (Group 3) showed a maximum thickness of OIL, followed by nanohybrid and least thickness of OIL in microhybrid. The plausible reason is, composites impregnated with glass fiber may contain some voids between the fibers due to their orientation, and these voids between the single fibers are assumed to be oxygen reservoir which allows oxygen to inhibit polymerization deeper in the structure of the SGFR composite.\textsuperscript{9,12}

### Table 2: Oxygen inhibition layer thickness value of all materials seen by stereomicroscope

| Group – Nanohybrid composite | Thickness of oxygen inhibition layer (micron) | Subgroup 1A (untreated surface) - 19.8 (5.5)* | Subgroup 1B (ethanol-treated surface) - 19.0 (5.7)* | Subgroup 1C (water spray-treated surface) - 19.4 (4.7)* | Subgroup 1D (SiC treated) - Nil |
|-----------------------------|---------------------------------------------|---------------------------------------------|---------------------------------------------|---------------------------------------------|---------------------------------------------|
| Group – Microhybrid composite | Thickness of oxygen inhibition layer (micron) | Subgroup 2A (untreated surface) - 17.78 (5.5)* | Subgroup 2B (ethanol-treated surface) - 17.19 (5.7)* | Subgroup 2C (water spray-treated surface) - 17.4 (4.7)* | Subgroup 2D (SiC treated) - Nil |
| Group – Short glass fiber composite | Thickness of oxygen inhibition layer (micron) | Subgroup 3A (untreated surface) - 20.8 (5.5)* | Subgroup 3B (ethanol-treated surface) - 20.3 (5.7)* | Subgroup 3C (water spray-treated surface) - 20.5 (4.7)* | Subgroup 3D (SiC treated) - Nil |

Values are given as mean (SD). The superscript symbols (*) within a value represent a homogenous subset \((P>0.05)\) among the groups for each material individually. SD: Standard deviation.

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In this study, a high frequency of cohesive failure was seen with SGFR composite (three-dimensional group) (40%) as shown in Graph 2, followed by one-dimensional group and two-dimensional group showing 30% and 10% failure, respectively.

Adhesive failures and mixed failures were seen in particulate filler composites with ground surfaces (SiC treated).[19]

**CONCLUSION**

Within the limitations of this study, the following conclusions were drawn:
- The OIL, which acts as an intermediate layer, is retained on the surface of the composite even after treatment with ethanol and water spray
- The presence of an OIL improved the interlayer SBS of adjacent composite layers and led to more durable adhesion, whereas the absence of an OIL adversely affected the bond strength and led to adhesive interfacial failures.

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**Conflicts of interest**
There are no conflicts of interest.

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