Original Article

The *in vitro* Study of the Effect of Three Oxygen-Inhibited Layer Removal Methods on the Micro-Shear bond Strength of Light-Cure, Dual-Cure and Self-Cure Composite Resins to Dentin

Emad Pourhassan [1]; Reza Tayefeh Davalloo [2]; Fereshteh Naser Alavi [2];

[1] Postgraduate, Dept. of Operative Dentistry, Dentistry School, Guilan University of Medical Sciences, Rasht, Iran.
[2] Dept. of Operative Dentistry, Dental school, Guilan University of Medical Sciences, Rasht, Iran.

KEY WORDS
Composite Resins;
Adhesives;
Polymerization;

Received: 7 July 2025;
Revised: 8 December 2025;
Accepted: 5 May 2026;

ABSTRACT

Background: High concentrations of acidic monomers in adhesives can inhibit proper polymerization at the interface with self-curing composites.

Purpose: This study aimed to evaluate the effect of three oxygen-inhibited layer (OIL) removal methods on the micro-shear bond strength (μSBS) of dual-cure and self-cure composites to dentin using a two-step self-etch adhesive.

Materials and Method: In this *in vitro* study, forty bovine incisor crowns were mounted in acrylic resin. Dentin surfaces were abraded with 600-grit silicon carbide paper to create a standardized smear layer. Experimental groups included using a two-step self-etch adhesive (Clearfil SE Bond), curing it, and then dividing it into four groups: (1) control (no preparation after adhesive application) and subsequent groups including removal of the OIL with (2) cotton wool soaked in ethanol, (3) cotton wool soaked in pumice, and (4) blocking air with glycerin and re-irradiation. Light-cure (Clearfil APX), self-cure (Alpha-Dent Self Cure Composite), and dual-cure resin cement (Duo-Link Universal Adhesive) were placed in cylindrical molds made of transparent polyethylene tubes (120 total, 30 per group) on dentin. The μSBS of the samples was measured after thermocycling.

Results: In the light-cure composite, μSBS did not differ significantly in any group compared to the control. In the dual-cure composite, only the alcohol group showed a significant decrease in bond strength compared to the control. In the self-cure composite, all OIL removal methods significantly improved bond strength compared to the control.

Conclusion: Although removing the OIL improved bond strength in self-cure composites, it failed to achieve values comparable to light-cure groups. For dual-cure composites, OIL removal is unnecessary and potentially detrimental.

Corresponding Author: Pourhassan E, Dept. of Operative Dentistry, Dental School, Guilan University of Medical Sciences, Dental School, Rasht, Iran. Tel: +98-9113349268 Fax: +98-131-3263622
Email: emadphk@gmail.com

Cite this article as:

Introduction

The oxygen-inhibited layer (OIL) present in acidic self-etch adhesives can severely compromise bond strength when used in conjunction with self-cured resin composites.

Uncured acidic surface monomers infiltrate the curing resin and react in a reaction with the aromatic tertiary amine present in the composite material, thereby inhibiting adequate polymerization at the interface [1]. Compatibility issues between adhesives with acidic resin monomers and self-curing composites were first noted for certain two-step etch-and-rinse adhesives [2-3]. With the increase in acidic monomers in the adhesive, their tendency to undergo acid-base reactions with the amine component of self-cure composites increases. From a clinical perspective, this means that these types

of composites do not polymerize properly at their interface with these adhesives [2].

However, it seems that these negative interactions between self-cure resin composites and self-etch adhesives can be prevented by sequentially using an acidic primer and then a bonding resin free of acidic monomers [4]. The study conducted by Moll *et al.* [5] found that using self-cure resin composites in combination with certain adhesive systems, like Clearfil SE Bond and AdheSE (both two-step self-etch adhesives), resulted in a significant reduction in bond strength.

Certain manufacturers incorporate an additional activator into both multi-step and single-step adhesives to optimize their bonding effectiveness with dual-cure or self-cure composites. This approach ensures adequate polymerization occurs in deeper areas where light exposure may be considerably insufficient. However, several studies indicate that the introduction of a self-cure activator does not necessarily address potential compatibility issues [3, 6].

A study demonstrated that the application of an additional layer of adhesive resin, free from acidic monomers, or the placement of a low-viscosity composite layer on bonded dentin, can effectively resolve compatibility issues. This method significantly enhanced the bond strength between the adhesive and the composite material that incorporates a self-curing component [7].

Efforts to address this issue can be categorized at three different levels including (1) elimination of the acid-base reaction, (2) reduction of the effects of the acid-base reaction, and (3) removal of the OIL [8].

The OIL can be managed by either preventing its formation or removing it after it has formed. Prevention methods include polymerization in a nitrogen environment or coating the adhesive with glycerin gel, although the first method is not practical for clinical use. Removal methods, such as cleaning with alcohol or using pumice, have been discussed previously. However, these methods may not be suitable for dentin adhesives, as their thin layers can lead to the removal of the adhesive itself [2].

The aim of this study was to evaluate the effect of three distinct OIL removal methods on the micro-shear bond strength (μSBS) of light-cure, self-cure, and dual-cure composites bonded to dentin using a two-step self-etch adhesive.

Materials and Method

Sample Preparation and Study Design

This experimental *in vitro* study was conducted following approval by the Ethics Committee of Guilan University of Medical Sciences (Ethical Code: IR.GUMS.REC.1403.443).

Forty freshly extracted bovine incisors, free of caries, cracks, and defects, were selected using a convenience sampling method. While this sampling method is acknowledged as a limitation, strict inclusion criteria were applied to minimize potential selection bias. The teeth were cleaned of soft tissue residues using a scaler and stored in a 1% chloramine solution for one week, followed by storage in distilled water at 4°C for less than one month. The roots were removed using a diamond disc (Isomet, Buehler, Evanstone, IL, USA) under water cooling, and the pulp tissue was extracted. Ideally, the pulp chamber was sealed with cotton to prevent acrylic penetration. The crowns were then mounted in acrylic resin molds (Acropars, Karaj, Iran), with the labial surface exposed. The dentin surfaces were ground using wet silicon carbide papers (60, 80, 240, and 320 grit) to expose flat mid-coronal dentin. Finally, the surfaces were polished with 600-grit paper to create a standardized smear layer.

Grouping and Surface Treatments

Thirty-six of the mounted teeth were randomly divided into four main groups (n=9 teeth per group) based on the surface treatment applied to the OIL. On each tooth, approximately 3 to 4 composite cylinders were bonded to obtain a total of 30 samples per main group (Total N=120).

The two-step self-etch adhesive, Clearfil SE Bond (Kuraray, Osaka, Japan), was applied and cured according to the manufacturer's instructions in all groups. The specimens were then subjected to one of the following surface treatments as:

Group 1 (Control): No surface preparation was performed after the application and curing of the adhesive.

Group 2 (Ethanol): The OIL was removed by rubbing the surface with a cotton ball soaked in 70%

ethanol for 10 s applying gentle manual pressure, followed by rinsing with an air-water syringe.

Group 3 (Pumice): The surface was scrubbed with a mixture of fine-grained pumice powder and water using a cotton ball for 10 s with gentle manual pressure, follo-

wed by rinsing with an air-water syringe.

Group 4 (Glycerin): The adhesive surface was covered with a thin layer of glycerin gel (Ultradent Deox, Ultradent Products, Inc, USA) and re-irradiated with a light-curing device for 20 seconds. The distance between the light tip and the surface was maintained at 1 mm. The gel was then rinsed off.

Bonding Procedure

Following surface treatment, each main group was further subdivided into three subgroups based on the restorative material used: (1) light-cure composite (Clearfil APX, Kuraray, Osaka, Japan, Shade A2), (2) self-cure composite (Alpha-Dent Self Cure Composite, Dental Tech Inc, USA), and (3) dual-cure resin cement (Duo-Link Universal Adhesive Resin Cement, BISCO, Inc., Schaumburg, IL, USA).

Transparent polyethylene tubes (internal diameter: 1 mm, height: 2 mm) were placed on the treated dentin surfaces. To ensure stability and prevent marginal leakage, the tubes were peripherally sealed to the tooth surface using sticky wax.

The restorative materials were inserted into the molds according to their viscosity. For light-cure and self-cure composites (paste consistency), the materials were carefully inserted into the tubes using a condenser to minimize air entrapment. For the dual-cure resin cement, the material was injected directly into the tubes using the auto-mix syringe with a needle tip, ensuring complete filling from the bottom up to avoid void formation. In the light-cure group, the material was irradiated for 20 seconds from the top (distance: 2 mm). For the self-cure group, the material was allowed to set in a light-proof container for 15 minutes. In the dual-cure group, the material was light-cured for 40 seconds to simulate clinical light attenuation.

All light-curing procedures were performed using a Woodpecker O-Light device (Woodpecker Medical Instrument Co., Ltd, China) with an intensity of 1200 mW/cm². The intensity was verified using a radiometer.

To ensure standardization and consistency of the applied pressure and technique, all surface preparation and bonding procedures were performed by a single calibrated operator.

µSBS Test

After bonding, the polyethylene tubes were carefully removed using a scalpel blade. The specimens were examined under a light microscope (30×) to exclude any samples with interfacial defects or bubbles. The samples were stored in distilled water at 37°C for 24 hours and then subjected to 5,000 thermal cycles (5°C–55°C, dwell time: 30 s). The µSBS was measured using a Universal Testing Machine (SANTAM-20, Tehran, Iran) at a crosshead speed of 1 mm/min using a wire loop (thickness: 0.2mm). The bond strength (MPa) was calculated by dividing the failure load (N) by the bonding area (mm²).

Degree of Polymerization

Four additional teeth (one representing each surface treatment group) were prepared to evaluate the degree of conversion (DC) of the adhesive layer using **(**Fourier-transform infrared spectroscopy (FTIR). The degree of polymerization was calculated by analyzing the peak area ratios of the cured and uncured adhesive. The peak at 1608 cm^{-1} (aromatic C=C double bonds) was used as an internal standard reference as it remains unchanged during polymerization, while the peak at 1638 cm^{-1} (aliphatic C=C double bonds) was monitored to determine the changes in the degree of conversion.

$$\%DC = \left(1 - \frac{\left[\frac{abs(aliphatic{:}C{=}C)_{1,638\ cm^{-1}}}{abs(aromatic{:}C{\ldots}C)_{1,608\ cm^{-1}}}\right]_{cured}}{\left[\frac{abs(aliphatic{:}C{=}C)_{1,638\ cm^{-1}}}{abs(aromatic{:}C{\ldots}C)_{1,608\ cm^{-1}}}\right]_{uncured}}\right) \times 100$$

Data Analysis

Data were analyzed using SPSS software (version 23). The normality of distribution was assessed using the Shapiro-Wilk and Levene's tests. Data were analyzed using Two-way analysis of variance and Tukey's post hoc tests (for normally distributed data) or the Kruskal-Wallis test (for non-normal data). The significance level was set at $\alpha = 0.05$.

Results

µSBS

Table 1 presents the mean and standard deviations of the µSBS for all experimental groups.

In the light-cure composite, statistical analysis revealed no significant difference in bond strength among the experimental groups compared to the control (25.05 ±7.97 MPa).

For the dual-cure composite, the control group exhibited a mean bond strength of 25.38±5.70 MPa.

The ethanol-treated group showed a significant red-

Table 1: Mean and standard deviation of μSBS (MPa) for experimental groups

Group	Light-cure composite	Dual-cure composite	Self-cure composite
Control	25.05 ± 7.97 [a]	25.38 ± 5.70 [a]	0.00 ± 0.00 [c]
Alcohol	19.70 ± 8.26 [a]	13.51 ± 7.80 [b]	4.77 ± 3.13 [b]
Glycerin	24.69 ± 5.78 [a]	19.26 ± 7.89 [ab]	3.84 ± 1.95 [b]
Pumice	25.56 ± 7.90 [a]	23.09 ± 7.25 [a]	8.30 ± 3.01 [a]

Different superscript lowercase letters within the same column indicate statistically significant differences (p Value < 0.05).
Note: (μSBS: micro-shear bond strength)

uction in bond strength compared to the control,

whereas the pumice and glycerin groups showed no statistically significant differences.

In the self-cure composite groups, the control specimens exhibited complete adhesive failure prior to testing (pre-test failures), resulting in a bond strength of 0 MPa. However, all three surface treatment methods (ethanol, pumice, and glycerin) successfully facilitated bonding. Among these, the glycerin re-irradiation group demonstrated the highest mean bond strength (8.30± 3.01 MPa), followed by the ethanol (4.77±3.13 MPa) and pumice (3.84±1.95 MPa) groups.

Degree of Polymerization (FTIR Analysis)

Figure 1 illustrates the FTIR spectra of the adhesive surfaces following different treatments. The degree of conversion (DC) was calculated based on the ratio of the aliphatic C=C peak (1638 cm^{-1}) to the aromatic C=C internal standard peak (1608 cm^{-1}). The calculated surface polymerization rates were as follows:

* Glycerin group: 74%
* Pumice group: 72%
* Control group: 52%
* Ethanol group: 41%

These findings indicate that surface treatment with glycerin and pumice effectively increased the degree of polymerization compared to the control group. Conversely, the ethanol treatment resulted in a reduction in the degree of surface polymerization (41%), suggesting a potential degradation of the adhesive layer.

Discussion

Achieving optimal dentin bonding relies on the effective separation of hydrophilic primers and hydrophobic adhesives, a standard feature of gold-standard etch-and-rinse systems [9]. However, in clinical scenarios requiring bulk-fill restorations or core build-ups, practitioners often combine simplified self-etch adhesives with chemical-cured (self-cure or dual-cure) composites. This combination presents a well-documented incompatibility in which the acidic monomers present in the OIL of the adhesive interface react with and deactivate the tertiary amines required for the chemical polymerization of the composite [1-2]. While some manufacturers provide specific activators to mitigate this issue, evidence suggests these are often ineffective or may even compromise bond strength by diluting photoinitiators [6].

The primary objective of this study was to evaluate

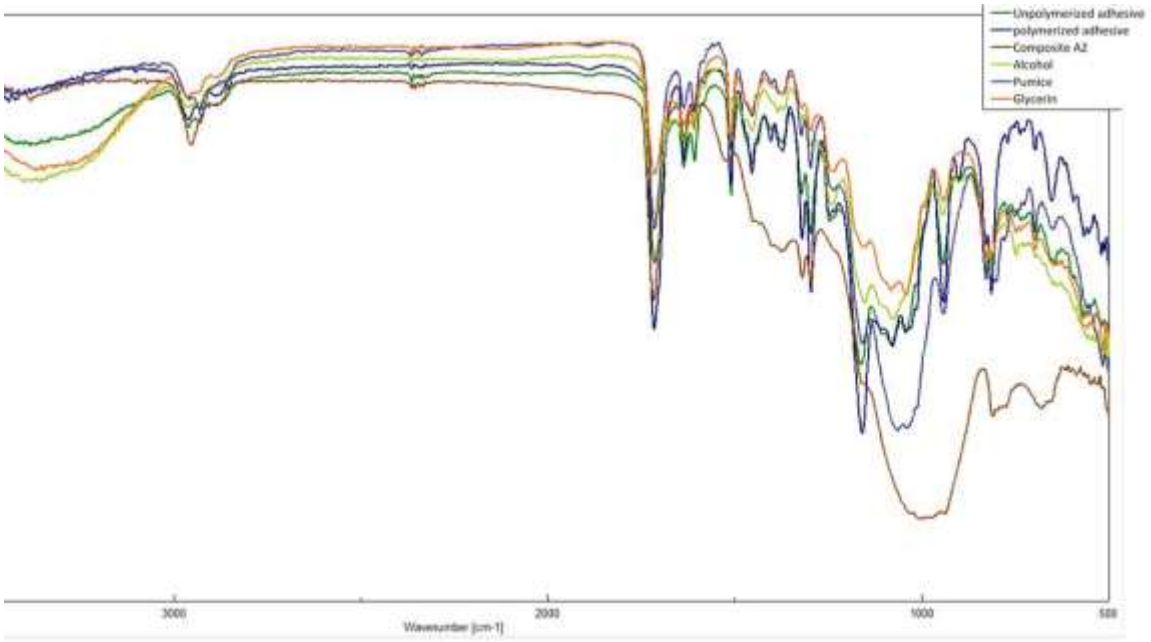

Figure 1: Fourier-transform infrared spectroscopy (FTIR) analysis of samples

whether removing or preventing the OIL could resolve this incompatibility. Our findings confirmed the severity of the issue; in the control group (no treatment), the self-cure composite failed to bond entirely (0 MPa), demonstrating a complete inhibition of polymerization at the interface [10].

Regarding the efficacy of OIL removal methods, all three tested methods- ethanol wiping, pumice scrubbing, and glycerin re-irradiation-successfully facilitated bonding for the self-cure composite, proving that neutralizing the OIL is a viable strategy. However, the effectiveness varied significantly among the methods.

The self-cure composite exhibited the most striking changes. In the control condition, no measurable bond strength was obtained (0 MPa), confirming severe incompatibility between chemically cured composites and an oxygen-rich, under-polymerized adhesive surface. The mechanism likely involves a competition for free radicals between the acidic monomers and the composite's initiator system, leading to deactivation of tertiary amines and incomplete curing at the interface [10].

Among the tested methods, glycerin gel re-irradiation produced the highest bond strength. This is due to the oxygen-blocking effect of glycerin during light activation, which promotes complete polymerization of the superficial adhesive layer [11].

Ethanol and pumice also significantly increased self-cure μSBS, but to a lesser extent than glycerin.

Regarding Pumice Scrubbing, while abrasion removes the inhibited layer and enhances micromechanical interlocking, excessive abrasion may overly thin the adhesive layer, creating stress-concentrating areas that compromise bond strength. The limitation of this mechanical approach lies in the delicate nature of the adhesive layer [8, 12].

Regarding ethanol cleaning, although it allowed the self-cure composite to bond, it negatively impacted the other groups. Ethanol is known to soften and partially dissolve cross-linked resin matrices, decrease cross-link density, and increase susceptibility to degradation [13]. Such effects can remove or plasticize the superficial portion of the adhesive, reduce the number of available double bonds, and create a porous, less homogeneous interface. This mechanism is strongly supported by the lower degree of conversion (41%) observed for the ethanol group in our FTIR analysis.

However, it is important to note that even after treatment, μSBS values for the self-cure composite remained below commonly suggested thresholds for durable dentin bonding. This aligns with earlier reports stating that incompatibility problems cannot always be fully resolved by surface treatments alone [11, 14].

These findings partially contrast with those of Tsujimoto *et al.* [15] who suggested that removing the OIL with ethanol is comparable to preventing its formation using nitrogen gas. However, their study focused primarily on surface free energy and did not fully account for the chemical degradation caused by prolonged exposure to alcohol on specific two-step self-etch adhesives. Our study demonstrates that for dual-cure and light-cure resins, the collateral damage caused by ethanol scrubbing (solvent-induced surface degradation) outweighs the benefits of removing the OIL [15-16].

The dual-cure composite showed a unique behavior in which only ethanol scrubbing significantly reduced μSBS compared with the control. This suggests that the dual-cure system is more sensitive to chemical alteration of the adhesive surface than to oxygen inhibition alone. As long as the adhesive surface remains structurally intact, the chemical curing component can evidently overcome moderate oxygen inhibition [16]. Interestingly, for dual-cure composites, the OIL did not appear to hinder bonding in the control group. In fact, removing this layer (especially with ethanol) proved detrimental [8]. This suggests that the light-activation component of dual-cure resins provides sufficient initial polymerization to overcome the acidic inhibition, making OIL removal steps unnecessary and potentially harmful for these materials. Therefore, aggressive chemical cleaning methods like ethanol wiping should be avoided for these materials, as solvent-induced surface damage appears to be more detrimental than the OIL itself.

Considering the relationship between FTIR and bond strength, the FTIR measurements showed that glycerin and pumice yielded the highest degrees of conversion at the adhesive surface, while ethanol produced the lowest values, with the control group in an intermediate position. This pattern closely paralleled the mechanical results, where glycerin and pumice generally supported higher μSBS, particularly in self-cure specimens, and ethanol was associated with the weakest bonds in dual-cure and self-cure groups.

The concordance between degree of conversion and bond strength observed here is in agreement with previous evidence indicating that higher conversion improves cohesive properties, interfacial integrity, and long-term durability of bonded interfaces [15]. Conversely, inadequate surface polymerization

may lead to plasticization, hydrolytic degradation, and early failure of the adhesive layer.

From a clinical standpoint, the present findings suggest that preventing oxygen inhibition during curing is more effective than mechanically or chemically removing the OIL after it has formed. Using glycerin gel over the adhesive before final light exposure represents a simple, inexpensive, and non-destructive method to improve surface conversion and bond strength, especially when bonding dual-cure or self-cure composites.

Pumice abrasion may provide some benefit, particularly for self-cure materials, but should be used cautiously because of the risk of thinning the adhesive layer and causing unnecessary surface damage. Ethanol scrubbing, based on the current and previous findings, should be applied with care, as it can soften polymerized resin matrices and reduce bond strength, particularly in dual-cure systems [13].

This study had several limitations. It was conducted *in vitro* on bovine teeth obtained by convenience sampling, which may not perfectly represent human clinical conditions. Only a single adhesive system and limited composite formulations were evaluated, and aging was restricted to thermocycling without long-term water storage or mechanical fatigue. Future investigations should assess the durability of bonds formed after different OIL-modification strategies, explore additional oxygen-blocking materials, and examine whether similar trends are observed with other adhesive chemistries and universal adhesives.

Conclusion

In summary, while removing the OIL is essential for establishing a bond with strictly self-curing composites, it does not restore bond strength to the levels achieved with light-cured materials. For dual-cure composites, OIL removal is unnecessary and potentially detrimental.

Acknowledgment

None.

Conflict of Interests

The authors declare no conflict of interest

References

[1] Koga K, Tsujimoto A, Ishii R, Iino M, Kotaku M, Takamizawa T, et al. Influence of oxygen inhibition on the surface free-energy and dentin bond strength of self-etch adhesives. Eur J Oral Sci. 2011; 119: 395-400.

[2] Tay FR, King N, Suh B, Pashley DH. Effect of delayed activation of light-cured resin composites on bonding of all-in-one adhesives. J Adhes Dent. 2001; 3: 207-225.

[3] Malaquias P, Gutiérrez MF, Sutil E, Matos TdP, Hanzen TA, Reis A, et al. Universal adhesives and dual-cured core buildup composite material: adhesive properties. J Appl Oral Sci. 2020; 28: e20200121.

[4] Panchal AC, Asthana G. Oxygen inhibition layer: A dilemma to be solved. J Conserv Dent. 2020; 23: 254-258.

[5] Moll K, Schuster B, Hailer B. Dentin bonding of light- and self-curing resin composites using simplified total- and self-etch adhesives. Quintessence Int. 2007; 38: e27-e35.

[6] Rathke A, Balz U, Muche R, Haller B. Effects of self-curing activator and curing protocol on the bond strength of composite core buildups. J Adhes Dent. 2012; 14: 39-46.

[7] Giannini M, De Goes MF, Nikaido T, Shimada Y, Tagami J. Influence of activation mode of dual-cured resin composite cores and low-viscosity composite liners on bond strength to dentin treated with self-etching adhesives. J Adhes Dent. 2004; 6: 301-306.

[8] Shade A, Wajdowicz M, Bailey C, Vandewalle K. The effect of simplified adhesives on the bond strength to dentin of dual-cure resin cements. Oper Dent. 2014; 39: 627-636.

[9] Haller B. Which self-etch bonding systems are suitable for which clinical indications? Quintessence Int. 2013; 44: 645-661.

[10] Sanares AME, Itthagarun A, King NM, Tay FR, Pashley DH. Adverse surface interactions between one-bottle light-cured adhesives and chemical-cured composites. Dent Mater. 2001; 17: 542-556.

[11] Yamaji A, Koga K, Tsujimoto A, Shimizu Y, Tsubota K, Takamizawa T, et al. Influence of oxygen-inhibited layer on dentin bond strength of chemical-cured resin composite. Eur J Oral Sci. 2013; 121: 497-503.

[12] Nowak AJ, Christensen JR, Mabry TR, Townsend JA,

Wells M. Pediatric dentistry: infancy through adolescence. 6th ed. Elsevier: St. Louis (MO); 2020. p.

[13] Moraes RRd, Schneider LFJ, Correr-Sobrinho L, Consani S, Sinhoreti MAC. Influence of ethanol concentration on softening tests for cross-link density evaluation of dental composites. Materials Research. 2007; 10: 79-81.

[14] Ritter A, Boushell L, Walter R. Sturdevant's Art and Science of Operative Dentistry. 7th ed. Missouri Elsevier: St. Louis; 2019. p.

[15] Tsujimoto A, Barkmeier WW, Takamizawa T, Latta MA, Miyazaki M. Influence of the oxygen-inhibited layer on bonding performance of dental adhesive systems: Surface free energy perspectives. J Adhes Dent. 2016; 18: 82-88.

[16] Ueta H, Tsujimoto A, Barkmeier WW, Oouchi H, Sai K, Takamizawa T, et al. Influence of an oxygen-inhibited layer on enamel bonding of dental adhesive systems: surface free-energy perspectives. Eur J Oral Sci. 2016; 124: 82-88.