DOI: http://doi.org/10.54617/adoklinikbilimler.1568236

Original Research Article

The Effect of Different Multimode Adhesives on Microleakage of Class V Composite Restorations in Three Etching Modes

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ABSTRACT

Aim: The aim of this *in vitro* study was to evaluate the effects of different multimode adhesives in three etching modes on occlusal and gingival microleakage of class V composite restorations.

Materials & Methods: One hundred and twenty human molars were randomly assigned to four groups (G2-Bond Universal [GBU], Clearfil Tri-S Bond Universal [CTU], OptiBond Universal [OBU], and Tokuyama Universal Bond [TUB]), and then three etching subgroups (total etch, self etch, and selective etch) (n=10 each). Standard Class V cavities were prepared and restored with a microhybrid resin composite. All teeth were exposed to a 30-second thermal cycle for 10,000 times at 5–55°C and then kept in 0.5% basic fuchsine solution for 24 hours. After the teeth were buccolingually cut, dye penetration was evaluated under a light microscope. Scanning electron microscopy analysis was also performed. Kruskal–Wallis and Mann–Whitney U tests were used for statistical evaluation (p=0.05).

Results: GBU, CTU, OBU, and TUB showed the least microleakage score with self etch, selective etch, total etch, and self etch modes, respectively. In self etch mode, GBU had the least and CTU had the most microleakage on occlusal margin ($p<0.05$), while there was no difference among adhesives on gingival margin. In total etch mode, GBU had the most microleakage on gingival margin ($p<0.05$), while there was no difference on occlusal margin among adhesives.

Conclusion: Occlusal or gingival microleakages of Class V composite restorations vary depending on the adhesives and etching modes.

Keywords: Acid etching; Composite resin; Dental adhesives; Dental leakage

Citation: Yilmaz F., Kursun S., Ozturk Z. The Effect of Different Multimode Adhesives on Microleakage of Class V Composite Restorations in Three Etching Modes. ADO Klinik Bilimler Dergisi 2025;14(3):177-188

Editor: Sinem Akgül, Gazi University, Ankara, Türkiye

INTRODUCTION

Microleakage is the subtle, often unnoticed transfer of bacteria, liquids, molecules, and ions at the interface between a cavity and the restorative material used.[1] Microleakage is a complex dental problem caused by many factors, such as weak adhesion of the restorative material to the dental tissues, polymerization shrinkage of composites, and difference in the coefficient of thermal expansion between the tooth and the restorative material.[1,2] The effectiveness of the adhesive agent used to ensure complete sealing by micromechanically and chemically adhesion of composite restorations to dental tissues remains extremely important. The effectiveness of adhesive systems in reducing microleakage at the tooth/restoration junction is a key factor in determining clinical success.[3] Obtaining a leak-proof restoration largely depends on the bond strength of the adhesive systems and strong adhesion.[4]

Received: 17.10.2024; Accepted: 22.07.2025
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Recent advances in adhesive systems have focused on simplifying application procedures to reduce technical precision and shorten application time. Depending on the adhesive mode, they are basically classified as total etch (TE) and self etch (SE) adhesives.[5] Their interactions with the smear layer—a uniform residual layer formed after preparation, which blocks the entrance of hydroxyapatite and dentin tubules, hence reducing their permeability—are also effective in the classification of adhesives. In TE adhesives, etching and then rinsing the phosphoric acid completely dissolve smear layer and remove it from the environment. In this system, a hybrid layer is formed by infiltration of the adhesive agent to the demineralization areas/microporosities created by phosphoric acid and subsequent polymerization (macrotags and microtags formations). However, the smear layer is made permeable without being completely removed and is included in the hybrid layer in SE adhesives, in which demineralization and infiltration are achieved simultaneously.[6] Although three-staged TE adhesives are accepted as a gold standard,[6] many clinicians request for simpler and less technique-sensitive materials or strategies.[7] This demand has encouraged manufacturers to develop easier-to-use adhesive systems.

The latest developments in adhesive systems comprise “universal” or “multimode” adhesives that claim to allow clinicians to choose their adhesion strategy. These new types of adhesives allow application with SE mode for dentin tissue and TE or the selective etch (SEE) modes for enamel tissue. In addition, these adhesives offer versatile uses for physically and chemically different dental tissues, such as enamel, dentin, and various restorative materials, such as composite resins, glass matrix ceramics, zirconia, and metals.[7] They are referred to as “universal” adhesives because they can bond to a wide variety of surfaces, and “multimode” adhesives because they can be used with different bonding techniques.

Multimode adhesives are similar to traditional SE adhesives; however, they contain specific functional monomers, such as carboxylate monomers (4-methacryloyloxyethyl trimellitate anhydride (4-META)) and/or phosphate monomers (10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) or glycerol phosphate dimethacrylate (GPDM), or a combination of an aryl borate catalyst and an acidic three-dimensional self-reinforcing monomer (3D-SR)). These functional monomers also ensure wetting and/or demineralization and chemical bonding to tooth substances.[8] The stability of bond strength is dependent on the material used and is subject to hydrolytic degradation, although these types of adhesives can chemically bond to various dental tissues.[9] Therefore, *in vitro* and clinical studies regarding the effectiveness of multimode adhesives are required.

This study aimed to evaluate the effects of different multimode adhesives in three etching modes on occlusal and gingival microleakage of Class V composite resin restorations using the dye penetration test method. The null hypotheses tested were as follows: (i) No difference was found among adhesive groups in gingival and occlusal microleakage scores of each etching mode. (ii) No differences exist among etching modes in gingival and occlusal microleakage scores of each adhesive.

MATERIALS AND METHODS

Ethics approval

This study was approved by the Ethics Committee of the Mugla Sitki Kocman University under protocol number 230038/68-07/10/2023. Patients who were indicated for tooth extraction for orthodontic or periodontal treatment at Mugla Sitki Kocman University were informed about the study. After written and signed consent forms were obtained from the volunteer participants, the extracted teeth were stored for use in the study.

Sample preparation

A power analysis was conducted using the G*Power package program with $\alpha = 0.05$, power = 80%, df = 3, and a medium effect size (f = 0.25, $\eta^2 = 0.06$. The required sample size was 10 per group. Therefore, a total of 120 human caries-free molars stored in distilled water containing 0.1% thymol solution at room temperature no longer than 6 months were used. Before use, the calculus, residual soft tissues, and periodontal fibers on the root surface were carefully eliminated using a scaler (Hu-Friedy Mfg. Co., Chicago, USA), and the teeth were subsequently polished with a pumice slurry.

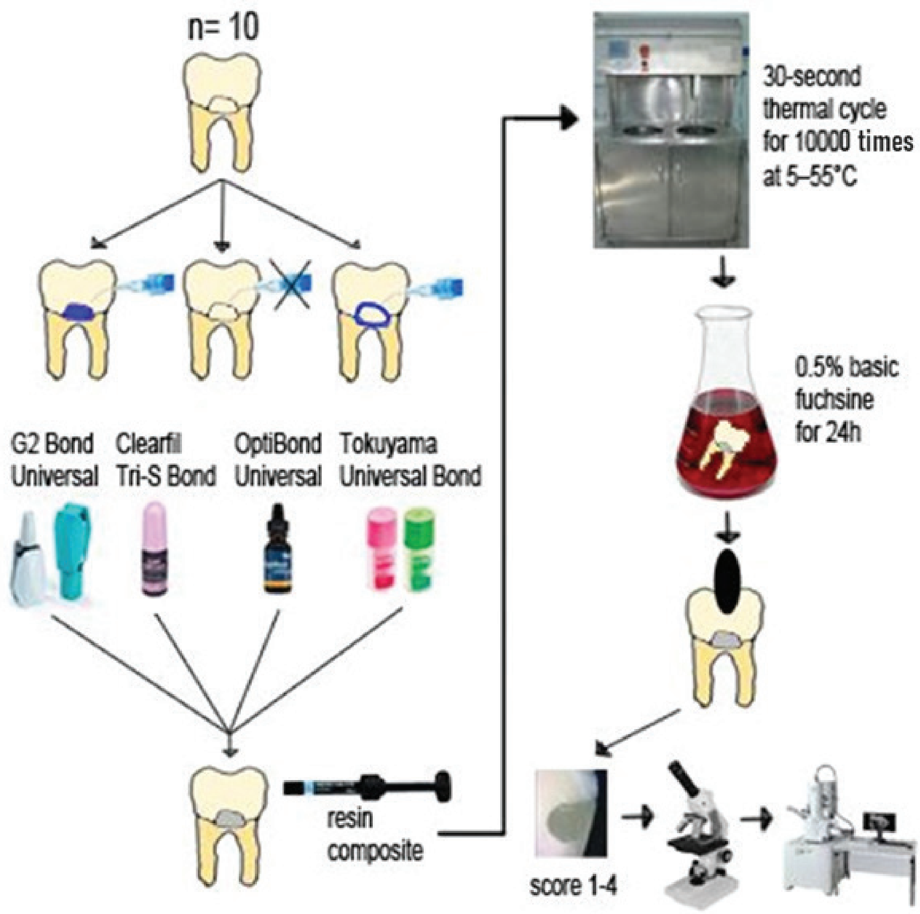

Figure 1. Flow chart of the study design.

Standard (mesiodistally 3 mm, occlusocervically 2 mm, and 1.5 mm depth, not beveled, rounded corners) Class V cavities located in the gingival third (gingival step 1 mm apical to the cementum–enamel border) were prepared on the buccal surface of the crown.[10] When opening the cavities, 801/016 mL diamond burs (Diatech, USA) were used. Each drill was replaced with a new one after used four times.

Experimental groups and restorative procedures

The teeth were randomly divided into four adhesive groups (G2-Bond Universal [GBU], Clearfil Tri-S Bond Universal [CTU], OptiBond Universal [OBU], and Tokuyama Universal Bond [TUB]). Then, the teeth in each adhesive group were further divided into three etching subgroups (TE, SE, and selective etch (SEE)) (n = 10). The research design is presented in Figure 1. Table 1 presents the materials used in the study, their contents, and the manufacturer's instructions.

Following the adhesive system protocols, the cavities were restored with a microhybrid composite resin (Herculite Classic, Kerr Corporation, Orange, CA, USA) in a single layer. The restoration was light-cured for 20 seconds using an monowave LED unit (Elipar™ Deepcure-S, 3M ESPE St Paul, MN, USA) was used in standard mode at 1470 mW/cm^2. All procedures and curing durations adhered to the manufacturer's instructions. The restorations were finished with fine-grit finishing diamond burs (Komet Dental, Brasseler GmbH and Co., Lemgo, Germany) and aluminum oxide-coated fexible discs (Sof-Lex, 3M ESPE) with coarse grit at 10,000 rpm for 30 s and then polished using the aluminum oxide-coated discs (Sof-Lex) with medium grit at 10,000 rpm and with fine and super-fine grits at 30,000 rpm for 30 s by the same operator.

Evaluation of microleakage

After completion of the restorations, the tooth samples were kept in distilled water for 7 days.[11] Then, they were exposed to a 30-second thermal cycle at 5–55°C for 10,000 times. The apexes of all samples were covered with two layers of wax to prevent dye penetration. The surfaces of the samples were covered with two coats of nail polish so that the restoration surface and the 1 mm area from the edges were exposed. Then, they were kept in 0.5% basic fuchsine solution for 24 hours.[11] Then, each tooth was embedded in standard transparent cold cure acrylic blocks, and three parallel vertical (longitudinal) sections were taken from the samples in the buccolingual direction with a low-speed, water-cooled diamond separator (Isomet, Buehler, Lake Bluff, IL, USA). Dye penetration along the occlusal and gingival margins in each section was examined under a stereo-light microscope (Olympus SZ61, Tokyo, Japan) at 20× magnification (1280 × 1024 resolution).

Table 1. The materials used in the study.

	Manufacturer	Content	Instructions
G2-Bond Universal (pH: 1.5; Lot number: 2210071)	GC, USA	**Primer:** 4-MET, 10-MDP, 10-MDTP, dimethacrylate monomer, acetone, water, initiators, and fillers **Adhesive:** dimethacrylate monomer, Bis-GMA, filler, photo-initiator	**Total etch mode:** Apply phosphoric acid on enamel for 30 s and on dentin for 15 s. Then, apply the adhesive, as shown in the self etch mode. **Selective etch mode:** Apply phosphoric acid on enamel only for 15 s. Then, apply the adhesive, as shown in the self etch mode. **Self etch mode:** Do not apply acid on anything. 1-Primer: Leave undisturbed for 10 s after application. Dry thoroughly for 5 s with oil-free air under maximum air pressure. 2-Bond: Apply the bond and gently apply air to evenly disperse the material into a uniform film thickness. Light curing with halogen/LED (700–1200 mW/cm²) 10 s.
Clearfil Tri-S Bond Universal (Ph: 2.3; Lot Number: 000104)	Kuraray Medical Inc., Japan	10-MDP, Bis-GMA, HEMA, colloidal silica, ethanol, saline, sodium fluoride, camphoquinone, ethanol, water	**Total etch mode:** Apply phosphoric acid on enamel for 30 s and on dentin for 15 s. Then, apply the adhesive, as shown in the self etch mode. **Selective etch mode:** Apply phosphoric acid on enamel only for 15 s. Then, apply the adhesive, as shown in the self etch mode. **Self etch mode:** Do not apply acid on anything. Apply bond and rub for 10 s. Dry by blowing in mild air for 5 s. Light cure.
OptiBond Universal (pH: 1.9; Lot number: 9740868)	Kerr Dental, USA	GPDM, GDM, HEMA, dimethacrylate, acetone, ethanol, water, (CQ)-based photo-initiator system, three nano-sized fillers, fluoride-releasing fillers, sodium exafluorosilicate and ytterbium fluoride	**Total etch mode:** Apply phosphoric acid on enamel for 30 s and on dentin for 15 s. Then, apply the adhesive, as shown in the self etch mode. **Selective etch mode:** Apply phosphoric acid on enamel only for 15 s. Then, apply the adhesive, as shown in the self etch mode. **Self etch mode:** Do not apply acid on anything. Apply bond and rub for 10 s. Dry by blowing mild air for 5 s. Light cure.
Tokuyama Universal Bond (Ph: 2.2; Lot Number: 001e12)	Tokuyama Dental Corp., USA	**Liquid A:** Phosphoric acid monomer (3D-SR monomer), MTU-6, HEMA, Bis-GEMA, TEGDMA, and acetone **Liquid B:** γ-MPTES, borate, peroxide, acetone, isopropyl alcohol, water	**Total etch mode:** Apply phosphoric acid on enamel for 30 s and on dentin for 15 s. Then, apply the adhesive, as shown in the self etch mode. **Selective etch mode:** Apply phosphoric acid on enamel only for 15 s. Then, apply the adhesive, as shown in the self etch mode. **Self etch mode:** Do not apply acid on anything. Dispense one drop each of A and B into the mixing well or disposable mixing well and mix. Apply the mixed bond. No need to wait. Apply weak air continuously to the surface until the runny bond stays in the same position without any movement, and then mild air to the surface. No need to light cure.
Herculite Classic (Lot number: 9726917)	Kerr Dental, USA	10-MDP, Bis-EMA, Bis-GMA, GDM; GPDM, HEMA, PENTA, TEGDMA, TMPTMA, UDMA, VBATDT.	
Gel Etchant (Lot number: V9425812)	Kerr Dental, USA	37.5% phosphoric acid gel	

4-MET, 4-[2-(methacryloyloxy)ethoxycarbonyl]phthalic acid; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; 10-MDTP, 10-methacryloyloxydecyl dihydrogen thiophosphate; bis-GMA, bisphenol A di (2-hydroxy propoxy) dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; TEGDMA, triethylene glycol methacrylate; MTU-6, 6-methacryloyloxyhexyl 2-thiouracil 5-carboxylateİ γ; MPTES, 3-meth-acryloxypropyl trimethoxy silane; GPDM, glycerol phosphate dimethacrylate; GDM, glycerol 1,3-dimethacrylate; CQ, camphoroquine; bis-EMA, bisphenol A ethoxylate dimethacrylate; PENTA, dipentaerythritol penttacrylate monophosphate; TMPTMA, trimethylolpropane trimethacrylate; UDMA, urethane dimethacrylate; VBATDT, 6-(4-vinylbenzyl-n-propyl) amino-1,3,5-triazine-2,4-dithione

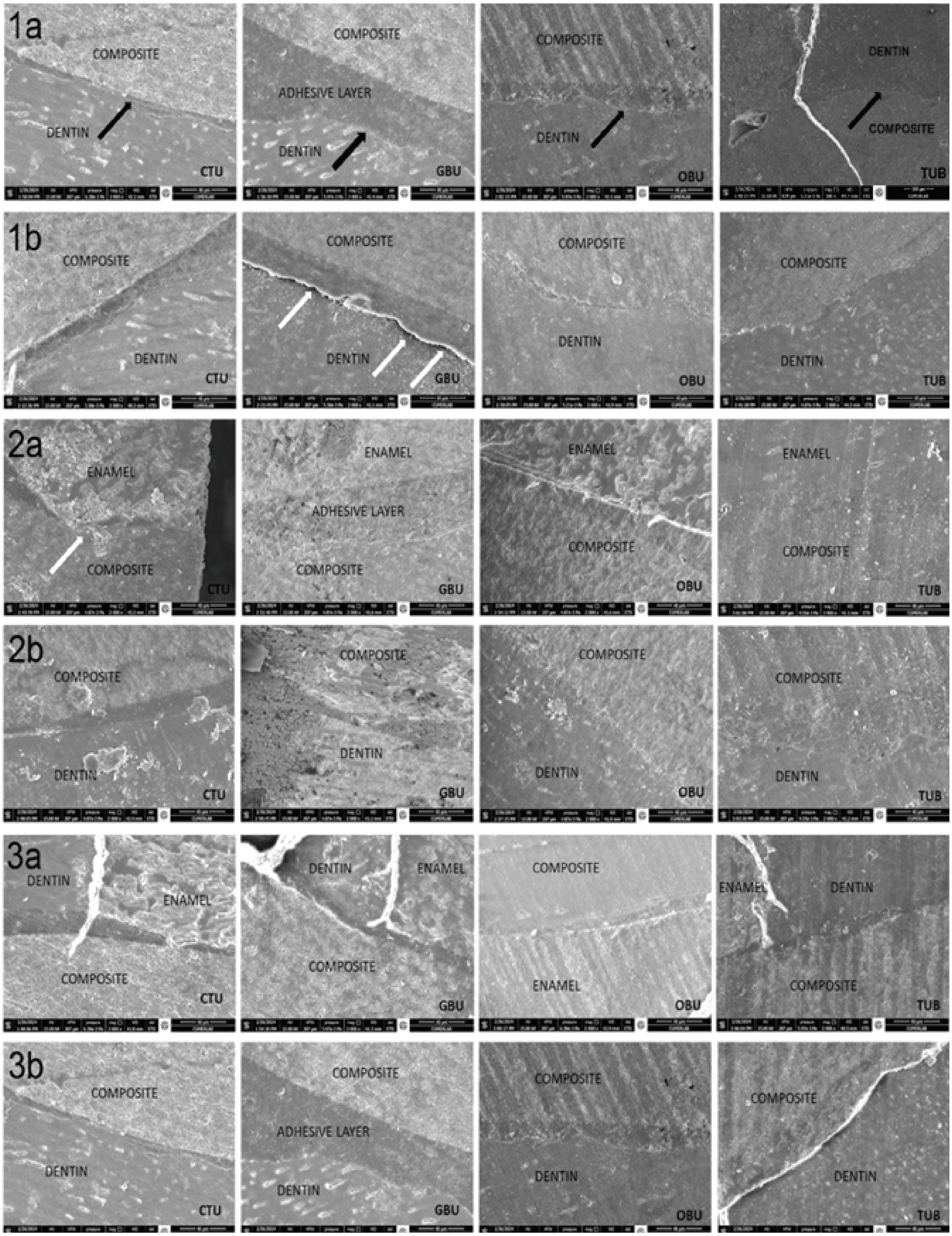

Figure 2. SEM images (x2000) of each adhesive group and etching subgroups (1a: Occlusal margin in total etch (TE) mode, 1b: Gingival margin in total etch (TE) mode; 2a: Occlusal margin in self etch (SE) mode, 2b: Gingival margin in self etch (SE) mode; 3a: Occlusal margin in selective etch (SEE) mode, 3b: Gingival margin in selective etch (SEE) mode). The tips of the white arrows on the images indicate microgaps and irregularities at the adhesive–dentin interface, while the black arrows indicate excellent adaptation of the adhesives.

Digital photographs of the images were taken and transferred to an IBM-compatible computer and evaluated using Touptek Toupview software program. Each surface was scored separately, including the enamel (occlusal) and dentin (gingival) margins, by two examiners in a double-blind manner. The highest leakage score for each margin was included in the statistical analysis. Microleakage values in Class V cavities were evaluated according to the following scoring criteria[11] by scoring the sections from 1 to 4:0. No dye penetration, 1. Dye penetration is equal or less than one-third of the gingival/occlusal wall length. 2. Dye penetration is up to a maximum of two-thirds of the gingival / occlusal wall length. 3. Dye penetration is throughout the gingival/occlusal wall. 4. Dye penetration spreads to the axial wall.

One sample randomly selected from each group was examined using scanning electron microscopy (SEM). Following dehydration in an aqueous ethanol solution, the specimens were coated with palladium using an ion plating device (Polaron SC500 sputter coater, FISONS Instrument, UK). The specimens were then carefully observed with a SEM (JSM-5600LV, JEOL, Tokyo, Japan) at 2000x magnification.

Statistical analysis

The data were analyzed using IBM SPSS V23. Kruskal–Wallis test was used for evaluating the differences between etching subgroups of each adhesive group, and differences between adhesive groups in each etching mode. Mann–Whitney U test was used in pairwise comparisons. The statistical significance threshold was $p < 0.05$.

Table 2. Comparison of etching modes of each adhesive group on gingival and occlusal margins and their descriptive statistics.

Adhesive	Location	Etching mode	Mean	SD	Median	95% Confidence interval		p
						Lower limit	Upper limit	
GBU	Gingival microleakage score	TE	1.80^{a}	1.55	1	0.69	2.91	0.014
		SE	0.40^{b}	1.27	0	−0.50	1.30	
		SEE	$0.60^{a,b}$	0.70	0.5	0.10	1.10	
	Occlusal microleakage score	TE	$0.30^{a,b}$	0.68	0	−0.18	0.78	0.033
		SE	0.10^{a}	0.32	0	−0.13	0.33	
		SEE	1.00^{b}	1.05	1	0.25	1.75	
CTU	Gingival microleakage score	TE	0.00^{a}	0.00	0	0.00	0.00	0.166
		SE	1.00^{a}	1.63	0	−0.17	2.17	
		SEE	0.60^{a}	1.27	0	−0.30	1.50	
	Occlusal microleakage score	TE	0.10^{a}	0.32	0	−0.13	0.33	0.001
		SE	1.70^{b}	1.42	2	0.69	2.71	
		SEE	0.00^{a}	0.00	0	0.00	0.00	
OBU	Gingival microleakage score	TE	0.10^{a}	0.32	0	−0.13	0.33	0.038
		SE	1.20^{b}	1.62	1	0.04	2.36	
		SEE	0.10^{a}	0.32	0	−0.13	0.33	
	Occlusal microleakage score	TE	0.70^{a}	0.68	1	0.22	1.18	0.286
		SE	0.90^{a}	0.99	1.5	0.19	1.61	
		SEE	0.40^{a}	0.97	0	−0.29	1.09	
TUB	Gingival microleakage score	TE	0.40^{a}	0.52	0.5	0.03	0.77	0.314
		SE	0.70^{a}	0.82	1	0.11	1.29	
		SEE	1.10^{a}	1.10	1.5	0.31	1.89	
	Occlusal microleakage score	TE	0.50^{a}	0.71	0	−0.01	1.01	0.130
		SE	0.80^{a}	0.79	1	0.24	1.36	
		SEE	0.40^{a}	1.27	0	−0.50	1.30	

Different superscripts represent statistical difference ($p < 0.05$). The Kruskal–Wallis test was used in the evaluations between the different etching subgroups of each adhesive group, and the Mann–Whitney U test was used in pairwise comparisons.

RESULTS

The mean microleakage score values of all tested groups were lower than 2 as shown in Tables 2 and 3 according to the scoring system in which score 0 and 1 were clinically acceptable while score 2, 3 and 4 showing dye penetration is two or more than-thirds of the gingival / occlusal wall length were not acceptable.[11, 12]

Comparison of microleakage scores according to etching modes within each adhesive group

Table 2 shows the microleakage scores of etching modes of each adhesive. Only TUB showed statistically similar microleakage in both the occlusal and gingival margins at all etching modes. In GBU group, GBU-SE showed statistically less microleakage than GBU-TE in the gingival margin (p=0.014) and then GBU-SEE in the occlusal margin (p=0.033). OBU-SE showed significantly more microleakage than OBU-TE and OBU-SEE in the gingival margin (p=0.038). CTU-SE showed more microleakage than CTU-SEE and CTU-TE in the occlusal margin (p=0.001).

Comparison of microleakage scores according to adhesives within each etching mode

Table 3 shows the microleakage scores of the different adhesives within each etching mode. In TE mode, GBU showed more microleakage than each other adhesives in gingival margin (p<0.01), while no difference was found among adhesives in occlusal margin. In the SE mode, no difference was found among adhesives in gingival margin, while GBU showed the least microleakage in occlusal margin (p=0.019). In SEE mode, no differences were found among adhesives in gingival margin, while CTU showed the least microleakage in occlusal margin (p=0.016).

Table 3. Comparison of adhesives according to the etching modes on gingival and occlusal margins and their descriptive statistics.

Etching mode	Location	Adhesive	Mean	S.D.	Median	95% Confidence interval		p
						Lower limit	Upper limit	
TE	Gingival microleakage score	GBU	1.80[a]	1.55	1	0.69	2.91	< 0.01
		CTU	0.00[b]	0.00	0	0.00	0.00	
		OBU	0.10[b]	0.32	0	−0.13	0.33	
		TUB	0.40[b]	0.52	0.5	0.03	0.77	
	Occlusal microleakage score	GBU	0.30[a]	0.68	0	−0.18	0.78	0.108
		CTU	0.10[a]	0.32	0	−0.13	0.33	
		OBU	0.70[a]	0.68	1	0.22	1.18	
		TUB	0.50[a]	0.71	0	−0.01	1.01	
SE	Gingival microleakage score	GBU	0.40[a]	1.27	0	−0.50	1.30	0.356
		CTU	1.00[a]	1.63	0	−0.17	2.17	
		OBU	1.20[a]	1.62	1	0.04	2.36	
		TUB	0.70[a]	0.82	1	0.11	1.29	
	Occlusal microleakage score	GBU	0.10[a]	0.32	0	−0.13	0.33	0.019
		CTU	1.70[b]	1.42	2	0.69	2.71	
		OBU	0.90[a,b]	0.99	1.5	0.19	1.61	
		TUB	0.80[a,b]	0.79	1	0.24	1.36	
SEE	Gingival microleakage score	GBU	0.60[a]	0.70	0.5	0.10	1.10	0.079
		CTU	0.60[a]	1.27	0	−0.30	1.50	
		OBU	0.10[a]	0.32	1	−0.13	0.33	
		TUB	1.10[a]	1.10	1	0.31	1.89	
	Occlusal microleakage score	GBU	1.00[a]	1.05	1	0.25	1.75	0.016
		CTU	0.00[b]	0.00	0	0.00	0.00	
		OBU	0.40[a,b]	0.97	0	−0.29	1.09	
		TUB	0.40[a,b]	1.27	0	−0.50	1.30	

Different superscripts represent statistical difference (p < 0.05). The Kruskal–Wallis test was used in the evaluations between different adhesive groups in each etching mode, and the Mann–Whitney U test was used in pairwise comparisons.

SEM observations

Representative SEM images of each adhesive according to the etching modes on gingival and occlusal margins are presented in Figure 2. In Figure 2/1a, excellent adaptation of all adhesives in TE mode in the occlusal margin can be seen. In Figure 2/1b, GBU demonstrated microgaps at adhesive-dentin interface in TE mode in the gingival margin. In Figure 2/2a, CTU had irregular adhesive layer in SE mode in the occlusal margin. The adhesive layer thickness of GBU in the occlusal margins was remarkably higher than the other groups in all etching modes.

DISCUSSION

The null hypothesis of the study had to be rejected because the occlusal and gingival microleakages of Class V composite restorations varied depending on the adhesives and etching modes. The functional monomers of tested adhesives in the study were MDP in the CTU and GBU adhesive groups, GPDM, GDM, and HEMA in the OBU group, and the 3D-SR phosphoric acid monomer in the TUB group.

In the present study, the GBU adhesive group containing MDP as functional monomer showed good marginal coverage and the least microleakage on both the occlusal and gingival areas when used in the SE mode. GBU has both an MDP monomer that provides a chemical connection to dental tissues creating hydrolytically stable calcium salts when interacting with hydroxyapatite,[13] as a functional monomer, and 4-MET, as an acidic monomer. Some investigations have reported that MDP-containing adhesives cause fewer secondary caries, better marginal sealing and long-lasting restorations clinically.[14] The 4-MET dissolves smear layer and demineralizes, and wetting agents infiltrate the demineralize surface while promoting chemical adhesion between tooth and monomers. In addition, the pH of the GBU adhesive was 1.5. Most multimode adhesives fall into the categories of weak (pH ≥ 2.5), mild (pH ≈ 2), and moderate (pH = 1 to 2).[15] Since the GBU adhesive contains both 4-MET monomers and the lowest pH among the adhesives tested, the GBU-SE group is thought to show less microleakage than the other groups used in SE mode, especially at the occlusal margin in which the enamel tissue is dense. The combination of MDP and 4-MET functional monomers enables simultaneous demineralization and resin infiltration, thus reducing the risk of leakage from the dentin/bond interface and increasing the long-term durability of the adhesive. Moreover, the GBU exhibited less microleakage on the occlusal margin than on the gingival when applied in the TE mode. The low microleakage of the GBU adhesive group on the gingival and occlusal margins, especially in the SE mode, is thought to occur due to the functional monomers it has and because it does not contain the HEMA (2-hydroxyethyl methacrylate) monomer in its formulation. It has been reported that HEMA has multiple disadvantages[16]: (i) Because HEMA, a hydrophilic monomer, has high water absorption, degradation occurs at the adhesive interface over time. (ii) The degradation occurring after hydrolysis of HEMA leads to the release of some small alcohol molecules, such as ethylene glycol, that have substantial water solubility. (iii) The polymerization efficiency of HEMA is weak because of having only one polymerizable group. (iv) It has been reported that HEMA reduces the effectiveness of MDP chemical interaction with hydroxyapatite. (v) HEMA has been showed to induce contact allergic reactions.[17] When all adhesives used in the SE mode were compared, especially in terms of occlusal microleakage, the lowest microleakage scores were obtained from the GBU adhesive group. In a recent study, HEMA-free adhesives had higher or equal enamel and higher dentin fatigue bond strength in the TE and SE mode.[18] Although the bond strength results obtained from the SE mode in a previous study[18] were compatible with the microleakage results of present study, the results obtained from the TE mode were found to be different. Although no differences were found between adhesives in terms of occlusal microleakage when the TE mode was applied in present study, the highest scores among gingival microleakage were obtained from the GBU-TE group. The microgaps at adhesive-dentin interface were also seen in the SEM images (Figure 2/1b). The increased microleakage is thought to be due to the acid causing the release and activation of endogenic enzymes (MMP) in the dentin tissue. Similarly, it was reported that MMPs can cause a loss of bond strength on dentin.[19] It is also very difficult to maintain acidified dentin moisture under optimum conditions. Improper and severe drying following the pickling and washing process causes collagen fibrils to precipitate, resulting in insufficient infiltration, remaining impenetrable gaps, and, ultimately, the formation of voids in the hybrid layer (hybridoid layer), bringing about a loss of bonding. In contrast, it has been reported that the adhesion of multimode adhesives to dentin is not negatively affected when they are used in TE modes, and adhesion is even better than conventional SE adhesives.[20, 21] As a result of the present study, GBU adhesive group is considered to cause the least microleakage on the occlusal and gingival margins when used in SE mode, while its use in TE mode causes more microleakage, considering that it reduces the bonding to dentin.

When the effects of different etching methods on gingival and occlusal microleakage were evaluated, different results were obtained. The CTU group containing MDP and HEMA were observed to have high occlusal microleakage scores in the SE mode. This result was also supported by the SEM image where the irregular adhesive layer was observed (Figure 2/2a). This situation can be attributed to many reasons, such as: HEMA disrupts the chemical bond of MDP to hydroxyapatite, acidic monomers are needed to provide a micromechanical connection, especially for enamel tissue, and they are in the weak pH range (pH: 2.3). In fact, CTU-SEE showed the least microleakage in the occlusal compared to all other adhesives used in the SEE mode. The fact that CTU-TE exhibits minimal microleakage on the gingival margin shows that the application of additional acid increases the bond of this adhesive. Similarly, it was reported that the use of multimode adhesives containing MDP in TE mode positively affected the bond to dentin.[20] It was also reported that multimode adhesives have good bonds to dentin, regardless of etching mode.[21]

Similarly, the OBU group containing GPDM, GDM, and HEMA showed better coverage and less microleakage after etching on the gingival margin, while OBU-SE showed the highest microleakage. This result is thought to be because the functional monomers did not fully cover the dentin; that is, the chemical bond to the dentin was weak. In addition, extra etching of the dentin tissue appeared to reduce microleakage by increasing the connection, similar to the CTU group. However, no difference in occlusal margin between all etching modes in the OBU adhesive group indicates that the bond of functional monomers to the enamel is better compared to dentin, and that the micromechanical adhesion to the enamel is better due to the medium pH of the OBU adhesive.[22] Similarly, Nascimento Foly *et al*.[23] recently reported that OptiBond Universal exhibited the highest resin-enamel bonds.

In the TUB group with the 3D-SR phosphoric acid monomer no difference was found among the etching subgroups on both the gingival and occlusal margins. TUB is a multimode adhesive that can be chemically polymerized and comes in two bottles. The first bottle has an aryl borate catalyst, while the second bottle had an acidic three-dimensional self-reinforcing (3D-SR) monomer. When the two solutions are mixed together, a borane compound forms. Borane is then oxidized by the peroxide it contains and acts as the initiator of chemical polymerization.[24] In this way, the manufacturer claims that TUB polymerizes chemically without the need for light application. However, there is not enough evidence in the literature on this subject. This study determined that the TUB applied with SE and self-cure exhibited microleakage scores similar to those of other adhesives used in the SE mode. These results are thought to be able to contribute to the literature. On the other hand, a study evaluating the effect of the curing mode on the bonding performance of multimode adhesives reported that light polymerization is necessary for the bond of TUB adhesive to hydrophilic dentin.[24]

When the tested adhesives were compared, the gingival microleakage scores of all adhesives used in the SE mode were found to be similar, showing that the abilities of covering the gingival margin and chemical bonding to dentin are close to each other. Since all adhesives exhibited similar gingival microleakage in SE mode, it can also be said that the pH level of the adhesive and the presence of HEMA in it have a greater effect on gingival microleakage than the main functional monomer. In contrast, a previous study comparing the microleakage of different multimode adhesives reported that the adhesive group with GPDM monomer showed less microleakage than the MDP and 3D-SR monomer groups.[25]

When all adhesives were applied in SE mode, and the resulting occlusal microleakage scores were compared, the least leakage was obtained in the GBU group as mentioned above. GBU had the lowest pH of the adhesives tested and was the only formulation that did not contain HEMA. Providing a micromechanical connection is more important and effective in adhesion to enamel, and a chemical connection comes second.[26] A study[27] which tested two weak pH-universal adhesives reported that applying three coats of universal adhesive in self-etch mode may improve the bonding performance and etching pattern on enamel, while the chemical interaction with enamel calcium remains unaffected by either the number of layers applied or any prior phosphoric acid etching.

When all adhesives were applied in the TE mode, no difference was found between the occlusal microleakage scores of the tested adhesives, while the gingival microleakage score of the GBU group was found to be higher than that of the others. According to the findings of this study, when all adhesives were applied in SEE mode, no difference was found on gingival microleakage as in SE mode, while the occlusal microleakage of GBU was found to be significantly higher than that of CTU. In a recent study in which the bond durability of a two-stage MDP-containing HEMA-free adhesive was tested, results supported the microleakage results of the present study in TE applications but differed in SE application.[18] GBU was found to exhibit greater or comparable fatigue bond strength for enamel and, at the very least, equal or greater fatigue bond strength for dentin when compared to other selected adhesive systems in TE mode.[18] Additionally, it also demonstrated that equal or superior bond strength for enamel and higher bond strength for dentin compared to adhesive systems in SE mode.[18]

Chemical formulations of adhesive systems can be discussed as described by the brands. Each adhesive system has different primers, solvents, organic/inorganic monomers, and pH. All these monomers have an effect on adhesion, microleakage and the physical properties of the adhesive. Although different adhesive systems were discussed in terms of their general content in our study, more specific material studies are needed to see the effect of each functional/non-functional monomer on microleakage. In the present study, the emphasis was on monomers in chemical formulations of adhesives. Many factors influence the results of microleakage studies, including the source and type of teeth or tooth specimens, as well as the selected storage medium and duration.[1] The way the restorative material is handled, placed, and polished also affects the level of detectable microleakage. There is currently no consensus on how to standardize various influential elements, such as aging methods like thermocycling, pH cycling, or repetitive mechanical loading.[1] These can be considered as limitations of this *in vitro* study.

CONCLUSION

Within the limitation of the current study, it was concluded that the microleakage scores of the gingival margin were statistically similar in SE mode, regardless of the adhesive system. HEMA-free adhesive showed the best microleakage scores at occlusal margin in SE mode. It can be concluded that rather than the effects of the functional monomers contained in different adhesives produced with commercial formulations, other factors accompanying the basic functional monomer, such as HEMA content and acidity, have a greater effect on microleakage.

However, the effects of all monomers in the chemical formulation of multimode adhesives should be considered as a whole. Furthermore, it was observed that different etching modes directly affected the degree of occlusal or gingival microleakages of the tested adhesives except for TUB. As a result of the study, GBU, CTU, OBU and TUB exhibited acceptable microleakage results under *in vitro* conditions in SE, SEE, TE and SE modes, respectively.

ACKNOWLEDGMENTS

The authors would like to acknowledge Nevin Guler Dincer for statistical analysis.

Farklı Çok Modlu Adezivlerin Üç Aşındırma Modunda Sınıf V Kompozit Restorasyonların Mikrosızıntısına Etkisi

ÖZET

Amaç: Bu in *vitro* çalışmanın amacı, farklı çok modlu adezivlerin üç asitleme modunda Sınıf V kompozit restorasyonların oklüzal ve gingival mikrosızıntısı üzerindeki etkilerini değerlendirmektir.

Gereç ve Yöntem: Yüz yirmi insan molar diş rastgele dört gruba (G2-Bond Universal [GBU], Clearfil Tri-S Bond Universal [CTU], OptiBond Universal [OBU] ve Tokuyama Universal Bond [TUB]) ve ardından üç asitleme alt grubuna (total asitleme, kendinden asitli ve selektif asitleme) (n = 10) ayrıldı. Standart Sınıf V kaviteler hazırlandı ve mikrohibrit bir kompozit rezin ile restore edildi. Tüm dişler, 5–55°C'de 30 saniye boyunca 10000 kez termal siklusa maruz bırakıldı ve ardından 24 saat boyunca % 0,5 bazik fuksin çözeltisinde bekletildi. Dişler bukkolingual olarak kesildikten sonra, boya penetrasyonu ışık mikroskobu altında değerlendirildi. Ayrıca taramalı elektron mikroskobu analizi yapıldı. İstatistiksel değerlendirme için Kruskal-Wallis ve Mann-Whitney U testleri kullanıldı ($p=0.05$).

Bulgular: GBU, CTU, OBU ve TUB sırasıyla kendinden asitli, selektif asitleme, total asitleme ve kendinden asitli modlarında en az mikrosızıntı skorunu göstermiştir. Kendinden asitli modunda, oklüzal kenarda GBU anlamlı derecede en az, CTU en çok mikrosızıntı gösterirken ($p<0.05$), gingival kenarda adezivler arasında fark yoktu. Total asitleme modunda, GBU gingival kenarda en çok mikrosızıntı gösterirken ($p<0.05$), oklüzal kenarda adezivler arasında anlamlı bir fark çıkmadı.

Sonuç: Sınıf V kompozit restorasyonların oklüzal veya gingival mikrosızıntıları, adezivler ve asitleme modlarına bağlı olarak değişiklik göstermektedir.

Anahtar Kelimeler: Asitleme; Dental adezivler; Dental sızıntı; Kompozit rezin

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