The Effect of Universal Adhesives on Bond Strength of Two Different Resin Cement to Dentine and Enamel

Üniversal Adezivlerin İki Farklı Rezin Simanın Dentine ve Mineye Bağlanma Mukavemeti Üzerine Etkisi

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ABSTRACT

INTRODUCTION: The purpose of this study was to investigate the effect of universal adhesives on the bond strength of resin cements to enamel-dentine.

METHODS: In this study, facial-enamel surface of 60 freshly extracted human upper-incisors and middle-coronal dentine surface of 60 freshly extracted human lower third-molars were used. According to applied adhesive and cement, 20 groups (n=12) were randomly formed, with 2 samples each tooth. Phosphoric acid (37%) was applied to enamel surface 30 seconds, universal adhesive was applied to the dentine surface without applying acid, and resin cement samples were bonded with Tygon tubes. RelyX U200 (3M ESPE, Germany) and Variolink N (Ivoclar Vivadent, Liechtenstein) resin cement were used with Single Bond Universal (3M ESPE, Germany), All Bond Universal (Bisco, USA), Clearfil Universal (Kuraray, Japan) and Futurabond U (Voco, Germany) adhesives were used. After the samples were subjected 5000 thermal-cycles, micro-shear bond test was performed on universal testing machine. Fracture types were determined by Scanning Electron Microscopy (SEM). Obtained data were evaluated with One-Way ANOVA and Tukey-Post-Hoc tests (p<0.05).

RESULTS: Universal adhesives increased bond strength of resin cement statistically significantly. In addition, bond strength enamel groups was higher than dentine groups. The lowest bond strength in enamel (13.5±2.8) and dentine (4.9±1.6) was observed group where RelyX U200 resin cement was used without applying adhesive (p<0.05).

DISCUSSION AND CONCLUSION: In clinical applications, universal adhesives can be preferred before resin cementation in order to increase bond strength to enamel-dentine.

Keywords: *Dentine, enamel, resine cement, universal adhesives*

ÖZ

GİRİŞ ve AMAÇ: Bu çalışmanın amacı, üniversal adezivlerin rezin simanların dentin ve mineye bağlanma mukavemetine etkisini araştırmaktır.

YÖNTEM ve GEREÇLER: Bu çalışmada 60 adet yeni çekilmiş insan üst kesici dişinin fasiyal mine yüzeyi ve 60 adet yeni çekilmiş insan alt üçüncü azı dişinin orta koronal dentin yüzeyi kullanıldı. Uygulanan adeziv ve simana göre her dişte 2 numune olacak şekilde rastgele 20 grup (n=12) oluşturuldu. Mine yüzeyine %37 fosforik asit 30 saniye uygulandı, dentin yüzeyine asit uygulanmadan üniversal adeziv uygulanarak rezin siman örnekleri Tygon tüpleri ile bağlandı. Çalışmada RelyX U200 (3M ESPE, Almanya) ve Variolink N (Ivoclar Vivadent, Liechtenstein) rezin simanlar ile Single Bond Universal (3M ESPE, Almanya), All Bond Universal (Bisco, ABD), Clearfil Universal (Kuraray, Japonya) ve Futurabond U (Voco, Almanya) adezivler kullanıldı. Numuneler 5000 termal döngüye tabi tutularak üniversal test makinesinde mikro-makaslama bağlanma testi gerçekleştirildi. Kopma tipleri Taramalı Elektron Mikroskobu (SEM) ile belirlendi. Elde edilen veriler Tek Yönlü ANOVA ve Tukey Post-Hoc testleri ile değerlendirildi (p<0.05).

BULGULAR: Üniversal adezivler rezin simanların bağlanma dayanımını istatistiksel anlamlı şekilde artırdı. Ayrıca mine gruplarındaki bağlanma dayanımı dentin gruplarından daha yüksekti. Mine (13.5±2.8) ve dentin de (4.9±1.6) en düşük bağlanma dayanımı RelyX U200 rezin simanın adeziv uygulanmadan kullanıldığı grupta görüldü (p<0.05).

TARTIŞMA ve SONUÇ: Klinik uygulamalarda mineye ve dentine bağlanma dayanımını artırmak amacıyla rezin simantasyon öncesi üniversal adezivler tercih edilebilir.

Anahtar Kelimeler: *Dentin, mine, rezin simanlar, üniversal adezivler*

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INTRODUCTION

Today, resin cement are often used for bonding indirect restorations (crown, inlay, onlay, overlay, laminate veneer) and posts.[1] The ability of resin cement to adhere to dental tissues and dental materials affects the survival of the restoration. The restoration survival is influenced by the physicochemical and mechanical properties of the resin cement, as well as the bond strength between the resin cement and the dental tissues/materials.[2]

The manufacturers introduced resin cement with different adhesion strategies. Some of these require extra acid-etching, rinsing, and adhesive application steps, while others require only adhesive resin application.[2] Three-step resin cement with acid-etch and bonding steps were defined as the best bonding systems by the researchers.[3] These systems are especially preferred for laminate veneers where bonding is very technique sensitive. However, clinicians are looking for a resin cement option with fewer steps to replace these systems because of their time-consuming steps.

In recent years, self-adhesive resin cement, which can bond to tooth structures without any adhesive steps, have been introduced.[4] They are widely accepted by clinicians because they are less time-consuming and technique sensitive. Manufacturers have claimed that these products contain a functional acidic monomer, methacrylate phosphoric acid that performs acid-etching and bonding on dental tissues simultaneously.[5] However, there are not enough studies in the literature regarding the potential of self-adhesive resin cement to bond to enamel and dentine tissue. In a study[6] related to the bonding of self-adhesive resins to fiber posts, it was noted that the bond strength was significantly reduced when applied without an additional silane or sandblasting step. Thus, contrary to the manufacturer's claim, a similar effect can be observed in dental hard tissues.[7]

Universal adhesives have been defined as adhesive systems that have been introduced by manufacturers in recent years and can be applied to different dental tissues or materials with or without acid-etching. In previous studies, investigators have studied the bonding performance of universal adhesives to dental tissues such as enamel, dentine, and different dental materials, and have received satisfactory results *in vivo* and *in vitro*.[8-11] However, the number of studies on whether universal adhesive resins affect the bond strength of resin cement is insufficient. This study aimed to investigate the effect of four different universal adhesives on the bond strength of resin cement to enamel and dentine tissues. The hypotheses tested in the study are as follows:

1. Universal adhesive application increases the bonding strength of the RelyX U200 and Variolink N resin cement to enamel and dentine.
2. There is a difference in the bond strength of the four universal adhesives used in the study to resin cement enamel and dentine.

MATERIALS AND METHODS

This study was approved by Nuh Naci Yazgan University Clinical Researches, Ethics Committee with the protocol number: 2022/9394.

Preparation of teeth

For the study, caries-free and freshly extracted 60 upper incisors and 60 lower third molars were used. Irregular areas on the enamel surface of sixty human upper incisors were flattened with a red belt diamond bur. Then, it was polished with 600-800-1000 grit silicon carbide (SiC) abrasives for 30 seconds (sec) to form a standard smear layer on the enamel surface. Sixty human lower third molars were trimmed 1/3 of the crown length to obtain middle coronal dentine. Then, the exposed dentine surface was smoothed with a red belt diamond bur and polished for 30 s with 600-800-1000 grit SiC abrasives to form a standard smear layer.

Study groups

Materials used in the study, manufacturers, contents, and application procedures are given in Table 1.

In both studies, two resin cement (RelyX U200 (3M ESPE, Neuss, Germany), Variolink N (Ivoclar Vivadent, Schaan, Liechtenstein)) and four Universal adhesives (Single Bond Universal (3M ESPE, Neuss, Germany), All Bond Universal (Bisco, Schaumburg, USA), Clearfil Universal (Kuraray, Okayama, Japan), Futurabond U (Voco, Cuxhaven, Germany)) were used. Finally, 20 groups were created for dentine and enamel studies (Figure 1).

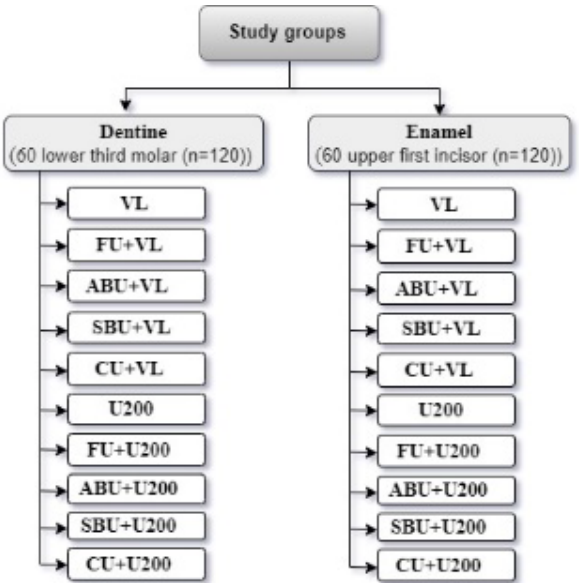

Figure 1. Study groups and systematic representation. VL: Variolink, ABU: All Bond Universal, SBU: Single Bond Universal, CU: Clearfil Universal, FU: Futurabond Universal, U200: RelyX U200.

Application of adhesive systems

In the enamel study, 37% phosphoric acid was applied for 30 s, rinsed for 10 s and dried for 10 s with air spray before applying adhesive. Each adhesive system was then applied according to the manufacturer's instructions (Table 1), polymerized with an LED light device (Valo, 1200 mW/cm^2, Ultradent Products Inc., South Jordan, USA) for 10 s.

Table 1. Materials, manufacturers, compositions, and application procedures used in the study.

Materials	Composition	Application mode
Relyx U200, 3M ESPE, Neuss, Germany. **Batch # 619698**	Glass powder treated with silane, ester dimethacrylate, TEGDMA, silica treated silane, glass fiber, sodium persulfate, sodium p-toluenesulfonate, calcium hydroxide, titanium dioxide.	Base and catalyst paste are applied with an automix syringe without pretreatment of dentin/enamel and light cured for 40 seconds.
Variolink N, Ivoclar Vivadent, AG, Schaan, Liechtenstein. **Batch # W36476**	Bis-GMA, UDMA, TEGDMA, barium glass, ytterbium trifluoride, Ba-Al fluorosilicate glass, spheroid mixed oxide, initiators, stabilizers and pigments.	Syntax Primer on the tooth and rub it gently using a brush for 15 seconds on the dentin. Then disperse the primer. Apply Syntax Adhesive using a new brush and rub it for 10 seconds. Disperse the excess adhesive and dry the tooth. Apply Heliobond and blow it to a thin layer. Mix Variolink N base and catalyst in 1:1 ratio for 10 seconds immediately before application.
Futura Bond Universal, Voco, Cuxhaven, Germany. **Batch # 1415274**	Liquid 1: Asidic adhesive monomer, HEMA, Bis-GMA, HEDMA, UDMA, catalyst. Liquid 2: Ethanol, initiator, catalyst.	Mix and stir thoroughly both liquids with the single tim applicator. Apply the adhesive homogenously to the surface and rub 20 seconds using the single tim. Dry off the adhesive layer with dry, oil-free air for at least 5 seconds. Light cure the adhesive layer for 10 seconds.
Clearfil Universal Bond, Kuraray, Okayama, Japan. **Batch # 2B0005**	MDP, Bis-GMA, HEMA, hydrophilic aliphatic dimethacrylate, colloidal silica, silane coupling agent, CQ, ethanol, water.	Apply the adhesive with the applicator to the entire surface and rub for 10 seconds. Dry gently for about 5 s until it no longer moves and the solvent has evaporated completely. Light-cure for 10 seconds.
Single Bond Universal Adhesive 3M ESPE, Neuss, Germany. **Batch # 623863**	10-MDP phosphate monomer, Vitrebond, Copolymer, HEMA, Bis-GMA, dimethacrylate resins, Filler, silane, initiators, Ethanol, water.	Apply the adhesive with the applicator to the entire surface and rub for 20 seconds. Dry gently for about 5 s until it no longer moves and the solvent has evaporated completely. Harden the adhesive with a curing light for 10 seconds.
All-Bond Universal Bisco, Schaumburg, USA **Batch # 1700007282**	10-MDP phosphate monomer, HEMA, Bis-GMA, ethanol, water, initiators.	Dispense 1–2 drops of ABU into a clean well. Apply two separate coats, scrubbing the preparation with a micro brush for 10–15 seconds percoat. Evaporate excess solvent by thoroughly air-drying for at least 10 seconds. Surface should have a uniform glossy appearance. Light cure for 10 seconds.

Bis-GMA, bis-phenol A diglycidylmethacrylate; HEMA, 2-hydroxyethyl methacrylate; TEGDMA, triethyleneglycol-dimethacrylate; MDP, 10- methacryloyloxydecyl dihydrogen phosphat; UDMA, urethane dimethacrylate; CQ, camphorquinone.

No additional acid-etching was performed in the dentine study. However, in the group where Variolink N was used alone without universal adhesives, the dentine-enamel adhesive system (Syntac® Primer and Syntac® Adhesive, Ivoclar Vivadent) and Heliobond (Ivoclar Vivadent) in the set were applied according to the manufacturer's instructions (primer 15 seconds, adhesive 10 seconds).

Application of resin cement

For each of the enamel and dentine studies, two resin samples (6 teeth x 2 samples) were obtained in each group, using Tygon tubes (0.75 mm inner diameter, 1 mm height) (Tygon, Norton Performance Plastic Co.). The cement was polymerized from two sides for 20 s each (40 s in total) with the LED (light emitting diode) light device (Valo, 1200 mW/cm^2, Ultradent Products Inc, South Jordan, ABD).

Micro-shear bond strength test

After thermal cycling of the prepared samples (5000×, 5 °C and 55 °C, 20 s dwell time), the Tygon tubes were removed using a scalpel and the samples were placed in a universal testing machine (Instron, Model 4444). Micro-shear bonding test was performed at a speed of 0.5 mm/min. The measured forces were recorded as Newtons (N) and calculated as megapascals (MPa) by dividing the bonding surface area (mm^2).

SEM analysis and examination of fracture surfaces

All fractured surfaces of the debonded samples were coated with gold-palladium and examined under scanning electron microscopy (SEM) (Leo-440, Cambridge, England) at ×100 and ×1000 magnification (Fig. 1). Failure modes were classified as follows: adhesive failure (A), at the resin-dentine interface; mixed failure (M), in which adhesive failure occurred with a thin layer of composite material remaining on the dentine surface; cohesive failure inside the resin composite material or dentine/enamel.

Statistical analysis

Statistical analysis was performed using SPSS 20.0 software (SPSS Inc., Chicago, IL, USA). Kolmogorov-Smirnov analysis was used to test the normality and it was decided to perform a parametric test for the analysis of the data. For this purpose, one-way ANOVA and Tukey Post-Hoc tests were used ($p<0.05$). Pre-testing failures were not included in the statistical analysis.

RESULTS

The mean, lowest, and highest bonding values, standard deviations, fracture types, and statistical differences of the groups and subgroups are given in Table 2.

Table 2. Mean, minimum, and maximum bond strength values, standard deviation, and fracture types of resin cement in dentine and enamel tissues. The different letters in the D column indicate the statistical difference between the groups.

			Bond strength (MPa)			Failure Modes (n)		
	Groups (n=12)	D	Mean±SD	Minimum	Maximum	Adhesive	Cohesive	Mixed
DENTINE	**VL**	Y	10.2 ± 3.1	6.1	13.9	9	0	3
	FU+VL	Y	9.9 ± 2.8	6.2	15.1	6	0	6
	ABU+VL	Y,Z	10.4 ± 2.8	7.4	16.4	5	1	6
	SBU+VL	Y,Z	10.9 ± 2.9	7.1	15.7	5	1	6
	CU+VL	Y	9.4 ± 2.5	5.5	14.4	7	0	5
	U200	X	4.9 ± 1.6	3.6	8.4	10	0	2
	FU+U200	Y,Z	12.1 ± 3.1	7.3	18.8	5	0	7
	ABU+U200	Y,Z	10.5 ± 3.1	5.1	15.6	6	0	6
	SBU+U200	Z	14.1 ± 3.6	7.6	20.6	5	1	6
	CU+U200	Y,Z	10.9 ± 2.9	7.7	15.3	7	0	5
ENAMEL	**VL**	B	18 ± 3.8	12.1	27.3	4	0	8
	FU+VL	E	26.5 ± 4.6	14.5	32.9	3	1	8
	ABU+VL	B,C	19.1 ± 3.5	13.2	24.1	5	0	7
	SBU+VL	D,E	25.9 ± 4.7	19.7	36.3	3	1	8
	CU+VL	A,B	17.1 ± 3.7	12.6	25.5	5	0	7
	U200	A	13.5 ± 2.8	10.3	19.5	6	0	6
	FU+U200	B,C,D	21.1 ± 4.3	13.4	29.3	4	0	8
	ABU+U200	A,B	16.8 ± 3.2	12.3	22.4	3	0	9
	SBU+U200	D,E	26.3 ± 5.1	19.3	33.7	2	1	9
	CU+U200	C,D,E	24 ± 4.9	10.9	36.5	4	1	7

D: Statistical difference, SD: Standard deviation, VL: Variolink, ABU: All Bond Universal, SBU: Single Bond Universal, CU: Clearfil Universal, FU: Futurabond Universal, U200: RelyX U200.

Dentine-bond strength

Bond strength values were lower in the RelyX U200 resin cement group (U200) without universal adhesive compared to the other groups. A statistically significant difference was found in the paired comparisons of RelyX U200 with all other groups ($p<0.05$). The four universal adhesive systems used in the study increased the bonding

values of the RelyX U200 ($p<0.05$). No statistical difference was observed between these four adhesives ($p>0.05$)). Variolink N resin cement showed similar bond strength values in the control group (VL) to the other groups ($p>0.05$). There was no statistical difference between the groups in which universal adhesive was applied together with Variolink N resin cement ($p>0.05$).

Enamel-bond strength

The results obtained in the enamel study showed variability according to the adhesive system used. RelyX U200 cement also showed the lowest bonding values in enamel (13.5 ± 2.8 MPa). All universal adhesives except All Bond Universal increased the bond strength of the RelyX U200 ($p<0.05$). All Bond Universal and Clearfil Universal did not alter the bond strength of Variolink N ($p>0.05$) while Futurabond U and Single Bond increased it ($p<0.05$).

Fracture types

The most common type of fracture in the dentine study was 'adhesive' (52%), followed by 'mixed' (46%) and 'cohesive' (2%) respectively. Fracture types did not show a statistical difference between groups ($p>0.05$).

The most common type of fracture observed in the enamel study was 'mixed' (64%), followed by 'adhesive' (33%) and 'cohesive' (3%) respectively. Fracture types did not show a statistical difference between groups ($p>0.05$).

An example SEM observation of the debonded surfaces from the dentine and enamel studies is given in Figure 2 and Figure 3.

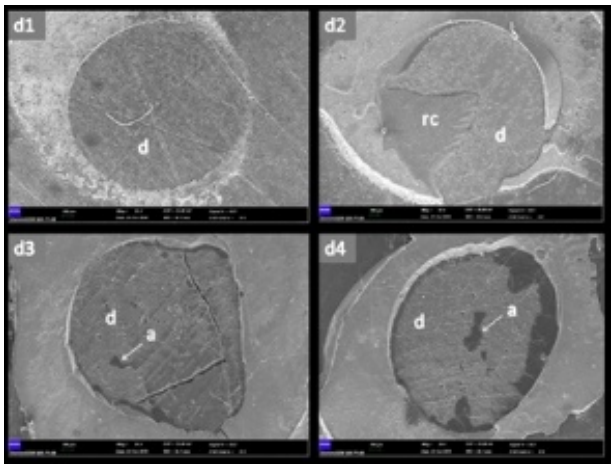

Figure 2. SEM observation of debonded surfaces from dentine study. **d1,** an adhesive failure from RelyX U200 resin cement group; **d2,** a mixed failure from Single Bond Universal + RelyX U200 resin group; **d3** and **d4**, adhesive failures from Variolink N resin cement and All Bond Universal + Variolink N resin group respectively. **d,** dentin; **a,** adhesive resin remnant; **rc,** resin cement remnant.

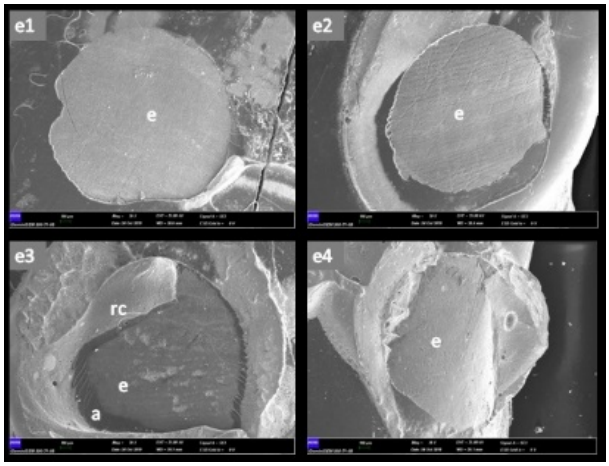

Figure 3. SEM observation of debonded surfaces from enamel study. **e1** and **e2,** adhesive failure from RelyX U200 resin cement and Variolink N resin cement groups respectively; **e3,** a mixed failure from Futurabond Universal + RelyX U200 resin group; **e4,** a cohesive failure from Single Bond Universal + Variolink N resin group. **e,** enamel; **a,** adhesive resin remnant; **rc,** resin cement remnant.

DISCUSSION

Self-adhesive systems can be preferred by clinicians because of their time savings and low sensitivity properties. Self-adhesive cement contains acrylic or diacrylate monomers and acidic adhesive monomers that form self-adhesive properties. Bonding to dental tissues occurs by chelation reaction with acidic monomers.[2,12] For adhesion, factors that are important in bonding to enamel and dentine such as the complex inorganic-organic structure of the tissues, the presence of moisture, possible chemical interactions, and the smear layer should be considered.[13] Many adhesive system manufacturers are working to ensure that more bonding agents penetrate these complex structures sufficiently, be chemically bonded, and form an ideal hybrid layer. Therefore, these cement, which are completely resin based, may be considered insufficient to bond to restorative materials and dental tissues. Not enough studies have been found in the relevant literature on that subject.

In this study, the effect of the use of universal adhesives on the application of two different resin cement to enamel and dentine tissues was investigated. RelyX U200 is a self-adhesive resin cement. The manufacturer claims that RelyX U200 resin cement can bond to enamel and dentine without any additional adhesive system. However, according to the results of the present study, the bond strength values of this cement alone were found to be very low. All universal adhesives used in this study increased the bond strength of RelyX U200 to dentine. The dentine bond strength values of Variolink N resin cement, which include acid etching, rinsing, and adhesive

application procedures in its application procedure, were not increased with the use of universal adhesives. In enamel, only Single Bond and Futurabond U increased the bond strength value of cement. Therefore, the first hypothesis of the study was partially accepted.

According to the results of the study, when the universal adhesive was not used, Variolink N showed higher bond strength to enamel and dentine than RelyX U200 resin cement. This may be because Variolink N resin cement includes etching and bonding steps in its application procedure.

All of the universal adhesives used in the study generally increased or did not change the bond strength of resin cement. Single Bond Universal adhesive was highest in dentine and Clearfil Universal was the lowest. Single Bond Universal and FuturaBond Universal adhesives were highest in enamel, while Clearfil Universal and All Bond Universal were the lowest. Briefly, different bonding results have occurred according to the applied tissue or adhesive system. Therefore, the second hypothesis of the study was accepted.

Pamato et al.[14] used Single Bond Universal adhesive before the application of RelyX U200 resin cement to dentine in a study. In this study, universal adhesives were tested with resin cement. Researchers compared Single Bond Universal to other bonding systems with different strategies and observed it to provide the highest bond strength values. They attributed this to the advantage of the Vitrebond copolymer contained in Single Bond Universal. Similarly, in this study, Single Bond Universal adhesive increased the bond strength values of cement on both enamel and dentine.

Prado et al.[6] reported that the bond strength of self-adhesive resin cement decreased significantly when applied without an additional silane or sandblasting step. A similar result was found in the presented study. The possible reasons for this are; the acidic monomer may have insufficient acid-etching capacity[2], tissue minerals buffering the low pH of the monomer[15], insufficient penetration[5] due to the higher viscosity of cement, compared to bonding systems[5], and inability to completely remove the smear layer.[16]

Temel et al.[17] reported that multi-step (etch and rinse) resin cement has higher bond strength on both enamel and dentine than self-adhesive resin cement. In a similar study, Cerqueira et al.[18] concluded that multistage resin cement offers a better hybrid layer than self-adhesive resins. The results of these studies are consistent with the present study.

In the study, while the most common type of fracture observed in enamel was found to be a ‘mixed’ fracture, the ‘adhesive’ fracture type was commonly observed in dentine. This is a natural result of lower mean bond strength values in the dentine study. Similarly, the bonding to dentine was found to be weaker in self-adhesive resin cement groups with ‘adhesive’ type fracture. Therefore, according to the results of this study, it is not recommended to use self-adhesive resin cement, especially in dentine without any bonding agent.

In this study, the micro-shear test was used to evaluate the bond strength of materials. Especially in the self-adhesive cement groups, very low bond strength and numerous pretesting failures after the thermal cycle confirmed this decision. The micro-shear test used in previous similar studies was preferred for this study.[19, 20]

The present study is an *in vitro* study and various factors in oral conditions such as oral stresses, pH changes, occlusal loads, enzymatic changes, and temperature changes have been ignored. Therefore, long-term *in vivo* and *in vitro* studies are needed.

CONCLUSION

According to the results obtained in the limitations of this study, universal adhesives increased the bond strength of resin cement to enamel and dentine. However, bond strength in enamel groups was higher than in dentine groups. For this reason, universal adhesives may be preferred before resin cementation in clinical applications. Universal adhesives can increase bond strength, especially in applications limited to the enamel.

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