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Shear Bond Strength of Chairside CAD-CAM Blocks to Eroded Dentin

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Research Article	ABSTRACT
	Objectives: To assess the shear bond strength (SBS) of chairside computer-aided design and computer-aided
History	manufacturing (CAD-CAM) blocks to dentin subjected to simulated gastric erosion when cemented with self-
	etch and self-adhesive resin cements.
Received: 28/03/2022	Materials and Methods: One hundred eighty dentin samples were assigned to two groups: sound and eroded
Accepted: 14/06/2022	dentin. In the eroded dentin group, samples were eroded with HCI (0.01 M, pH 2, 2 min) and stored in artificial
	saliva for 60 min then, brushed using a power brush (2N, 15 s). This cycle was repeated three times. CAD-CAM
	blocks (3x3x3 mm ³ , n=15/group) of Lava Ultimate (LU), Vita Enamic (VE), and Vita Suprinity (VS) were cemented
	to sound and eroded dentin with self-etch Multilink N (MN) and self-adhesive RelyX U200 Automix (RU) resin
	cements. SBS was measured after 24 hours. The failure mode was assessed by using a stereomicroscope. Data
	was analyzed with 3-way ANOVA and Bonferroni correction.
	Results: The SBS was significantly affected by the main factors: tooth structure, resin cements, and CAD-CAM
	blocks. When LU was cemented with RU to sound dentin, a higher SBS was obtained compared to eroded dentin.
	MN revealed significantly higher SBS than RU. When using MN in sound dentin LU showed lower bond strength
	than VE and VS. The predominant failure mode was mixed for all groups.
	Conclusions: It was determined that the bond strength of dentin was affected by simulated gastric erosion. The
	use of Multilink N resin cement in both sound and eroded dentin can be recommended. For a reliable bond to
	eroded dentin, selection of the proper cement system and material type are necessary.

Keywords: Dentin; CAD-CAM blocks; Shear Bond Strength; Gastric Erosion; Resin Cement.

Hastabaşı CAD-CAM Blokların Eroze Dentine Makaslama Bağlanma Dayanımı

e	ÖZ			
Süreç	Amaç: Bu çalışmada hastabaşı bilgisayar destekli tasarım-bilgisayar destekli üretim (CAD-CAM) bloklarının self-			
Calic: 28/02/2022	etch ve self-adeziv rezin simanlar kullanılarak gastrik erozyona uğratılmış dentine olan makaslama bağlanma			
Geliş, 28/05/2022 Kabul: 14/06/2022	dayanımlarının değerlendirilmesi amaçlanmıştır.			
Kabul: 14/06/2022	Gereç ve Yöntem: Yüz seksen dentin örneği sağlam dentin ve eroze dentin olmak üzere iki gruba ayrılmıştır.			
	Eroze dentin grubunda örnekler HCI (0,01 M, pH 2, 2 dk) ile erozyona uğratılmış ve 60 dk yapay tükrükte			
	bekletilmiş, sonrasında, elektrikli diş fırçası kullanılarak (2N, 15 s) fırçalanmıştır. Bu döngü üç kere tekrar			
	edilmiştir. Lava Ultimate (LU), Vita Enamic (VE) ve Vita Suprinity (VS) CAD-CAM blokları (3x3x3 mm3, n=15/grup)			
	self-etch Multilink N (MN) ve self-adeziv RelyX U200 Automix (RU) rezin simanları kullanılarak sağlam ve eroze			
	dentin örneklerine simante edilmiştir. Makaslama bağlanma dayanımı 24 saat sonra ölçülmüştür. Başarısızlık tipi			
	stereomikroskop kullanılarak degerlendirilmiştir. Veriler üç-yonlu ANOVA ve Bonferroni düzeltmesi kullanılarak			
	analiz edilmiştir. Bulayılar Malazlara bağlaran dayanan ana falti alardan ilandi. İlaüda atlikanıştırı da yaşarı (ö. 0.011)			
	Bulgular: Makaslama baglanma dayanimi ana taktorierden onemii olçude etkilenmiştir: diş yapısı (p=u,011),			
rezin simanlar ve CAU-CAM bloklar LU saglam dentine RU ile simante edildiginde, ei karşılaştırıldığında daha yüksek bağlanma dayanımı elde edilmiştir. MN kullanıldığında, RU'dar bağlanma dayanımı değarları elde edilmiştir. Sağlam dentinde MN kullanıldığında ILU KE ve VS'da				
	belirlenmistir.			
	Sonuclar: Dentinin bağlanma davanımının gastrik erozvondan etkilendiği belirlenmistir. Hem sağlam hem de			
	eroze dentinde Multilink N rezin siman kullanımı önerilebilir. Eroze dentinde güvenilir bir bağlanma için uygun			
License	siman ve restoratif materyal seçimi gerekmektedir.			
This work is licensed under				
Creative Commons Attribution 4.0				
International License	Anantar Kelimeler: Dentin; CAD-CAIVI Blok; Makaslama Baglanma Dayanimi; Gastrik Erozyon; Rezin Siman.			
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Introduction

Dental erosion is the devastation of dental hard tissue caused by extrinsic (environment, diet and drugs) and intrinsic (gastric juice) acids without the influence of bacteria.¹ Tooth surfaces can be affected by acidic gastric content as a result of chronic vomiting, regurgitation, rumination, or gastroesophageal reflux. In these patients, loss of the mineral by intrinsic acid on tooth structure can be explained by the erosive potential of hydrochloric acid (HCl). HCl in gastric juice is a strong acid with high erosive potential.²⁻⁴

Erosive challenges to tooth structure lead to loss of structural integrity, greater wear and loss of microhardness.⁴⁻⁶ The opening of the dentin tubules, the removal of the organic part of the intertubular dentin and the dentin plugs, increasing the diameter of the tubule, and causing collagen exposure have been reported when erosive lesions reached dentin.⁷ If dentin is chronically exposed to acids, mineral loss increases and the organic dentin matrix is gradually exposed. If demineralization proceeds, mineral loss decreases over time with the protective effect of the developing organic dentin matrix.⁸

Restorative procedures are essential to recover function and esthetics, maintaining tooth structure and preventing dentin hypersensitivity in the teeth where erosive tooth wear occurs.9 Computer-aided design and computer-aided manufacturing (CAD-CAM) system allows clinicians to independently design and process high quality, highly esthetic dental restorations, allowing the procedure to be completed in a single visit.¹⁰ Chairside CAD/CAM blocks can be used to make inlays, onlays, veneers and crowns. Within chairside CAD/CAM blocks, Lava Ultimate (3M ESPE) is a resin nano-ceramic material, that contains a polymer network (20% wt) strengthened by 80% wt zirconia-silica nanofillers.¹¹ Vita Enamic (Vita Zahnfabrik) is a polymer-infiltrated ceramic network material that contains strengthened polymer network (14% wt) and ceramic network (86% wt). The ceramic and polymer networks completely penetrated each other.¹² Vita Suprinity (Vita Zahnfabrik) is a zirconia-reinforced lithium silicate ceramic. The incorporation of zirconia particles strengthens the ceramic structure and helps prevent crack progression.13,14

It is recommended to use adhesive luting to bond CAD-CAM blocks to tooth structure.^{15,16} Self-etch cements are used with self-etch primer on prepared tooth surfaces. Self-adhesive resin cements eliminate adhesive or acid application steps and offer a simplified application

procedure with one-step use. They provide chemical bonding to the tooth surface due to the acidic monomers in the structure.^{17,18} Several studies have evaluated the changes that occur on the dentin bond strength after erosive challenge.¹⁹⁻²⁴ In this studies, the extrinsic erosion was simulated by using citric acid^{19,20} or acidic drinks²¹⁻²³ and intrinsic erosion was simulated by using HCI-pepsin solution.²⁴ To our knowledge, no previous study has investigated the bond strength of chairside CAD-CAM blocks to dentin exposed to intrinsic erosion. Therefore, the present study aimed to evaluate the shear bond strength (SBS) of current chairside CAD-CAM blocks to dentin exposed to simulated gastric erosion when cemented with self-etch and self-adhesive resin cements. The null hypothesis was that there would be no effect of intrinsic dental erosion, of resin cements, or of CAD-CAM blocks on the SBS to dentin.

Materials and Methods

In this in vitro study, three types CAD-CAM blocks-Lava Ultimate (LU), Vita Enamic (VE), and Vita Suprinity (VS)were cemented to sound and eroded dentin with self-etch Multilink N and self-adhesive Rely X U200 Automix resin cements to evaluate the SBS (Table 1).

Ethical approval was received from the Ethical Research Committee of the Karadeniz Technical University in Trabzon, Türkiye (ID: 2018/71 and decision date 07.05.2018).

Preparation of Dentin Samples

Human third molars without cracks, fractures, or defects were used. After extraction, ninety teeth were kept in +4 °C 0.5% chloramine-T aqueous solution for 1 week and then kept in +4 °C distilled water for a maximum of one month. The buccal and lingual enamel was removed using a slow-speed diamond saw (Microcut 150; Metkon Instruments), and the flat dentin surfaces were exposed. One hundred eighty dentin samples were embedded in autopolymerizing acrylic resin (SC; Imicryl Dental, Türkiye) in cylindrical silicone molds. After removal mold, grinding was performed under running water with silicon carbide abrasive paper up to 1200-grit in a polishing machine (Beta Grinder-Polisher, Buehler) to create standardized smear layers. Dentin samples were assigned to two groups: sound dentin and eroded dentin. A schematic flow chart of the experimental procedure is shown in (Figure 1).

Table 1. CAD-CAM blocks used in this study					
Materials	Batch Numbers	Composition			
Lava Ultimate	N804706	80% ceramic (69% SiO ₂ , 31% ZrO ₂), 20% polymer (Bis-GMA, UDMA,			
(3M ESPE, USA)	11094700	Bis-EMA, TEGDMA)			
Vita Enamic	50711	86% ceramic (58-63% SiO ₂ , 20-23% Al ₂ O ₃ , 9-11% Na ₂ O, 4-6% K ₂ O, 0-			
(VitaZahnfabrik, Germany)	55/11	1% ZrO ₂), 14% polymer (UDMA, TEGDMA)			
Vita Suprinity (VitaZahnfabrik, Germany)	63323	56-64% SiO ₂ , 1-4% Al ₂ O ₃ , 15-21% Li ₂ O, 8-12% ZrO ₂ , 1-4% K ₂ O			

SiO₂: silicon dioxide, ZrO₂: zirconium dioxide, Bis-GMA: bisphenol-A-glycidylmethacrylate, UDMA: urethane dimethacrylate, Bis-EMA: bisphenol-Aethoxylate glycidyl methacrylate, TEGDMA: triethylene glycol dimethacrylate, Al₂O₃: aluminium trioxide, Na₂O: sodium oxide, K₂O: potassium oxide, Li₂O: lithium oxide



Figure 1. Shematic flow chart of the experimental procedure.



Figure 2. Distribution of failure modes after shear bond strength test.

LU.RU: Lava Ultimate + RelyX U200 Automix; LU.MN: Lava Ultimate + Multilink N; VE.RU: Vita Enamic + RelyX U200 Automix; VE.MN: Vita Enamic + Multilink N; VS.RU: Vita Suprinity + RelyX U200 Automix; VS.MN: Vita Suprinity + Multilink N.

Erosion-Abrasion Procedure

Samples in eroded dentin group were subjected to three cycles of erosion-abrasion procedure. One cycle consisted of erosion and brushing.²⁵ To simulate gastric erosion, 0.01 M HCI (Noratex Kimya) with a pH of 2 applied to the dentin samples with 2 minutes (min).²⁶ Samples were rinsed in distilled water and kept in artificial saliva for 60 min for remineralization then samples were brushed using a power brush (Triumph Professional Care, Oral B Braun GmbH) which was settled on a custom-made holder and a brushing force of 2 N⁸ for 15 seconds. Softbristles toothbrush head (Oral-B Sensitive, Braun) was selected. Slurry was prepared with toothpaste (ProNamel, Sensodyne; RDA value 34) and artificial saliva (3:1). After toothbrushing, samples were rinsed in distilled water for 1 min, then the other cycle was carried out. Three cycles were performed, one after the other. ^{25,26}

Preparation of CAD-CAM Blocks

The blocks were cut with a slow-speed, water-cooled diamond saw (Microcut 150; Metkon Instruments). A total of 180 samples, 60 samples from each block type, of $3 \times 3 \times 3 \text{ mm}^3$ dimensions were obtained. LU, VE, and VS CAD-CAM blocks were cemented with RelyX U200 Automix (RU) and Multilink N (MN) resin cements to sound and eroded dentin surfaces (n=15).

Pretreatment of CAD-CAM blocks and application procedures of the resin cements were used according to the corresponding manufacturer's instructions (Tables 2, 3). Resin cements were applied to the surface of the blocks and seated to the dentin with finger pressure then polymerized with light (Elipar S10, 3M ESPE, 1200 mW/cm²). After cementation, samples were kept in 37 °C, 100% humidity for 24 hours (h), then shear bond strength (SBS) was tested by using a universal testing machine (Instron 3382) with 0.5 mm/min crosshead speed. The maximum force (N) was recorded at the time the fracture occurred, then N divided by the surface area of the sample to calculate SBS values (MPa). The fractured surface of each sample was examined by stereomicroscopy (Leica MZ16) at 40x magnification, and the failure modes were categorized as cohesive in dentin, cohesive in cement, adhesive in dentin-cement interface, adhesive in cement-CAD-CAM block interface, and mixed.

Statistical Analysis

The data was analyzed by SPSS for Windows 17.0 (SPSS Inc.). The Shapiro–Wilk test was used for testing normality. SBS data was evaluated by three-way ANOVA. The Bonferroni correction was used for multiple comparisons. Statistical significance was considered at p<0.05.

Table 2. Pre-treatment of CAD-CAM blocks				
CAD-CAM material	Pretreatment steps			
	Sandblasting with Cojet sand, at 2 bars			
Lava Ultimate	Removing sand with alcohol, air-drying			
	Applying Single Bond Universal Adhesive (scrubbing 20s)			
Vita Enamic	Conditioning (HF 5%, 60 s), rinsing (60 s), drying (20 s), silanization			
Vita Suprinity	Cleaning ultrasonic bath with distilled water			
	Crystallized at 840 °C for 8 min in ceramic furnace (Programat P300),			
	Conditioning (HF 5%, 20 s), rinsing with water, cleaning with 98% alcohol (1–3 min), drying, silanization			

*HF: Hydrofluoric acid.

Table 3. Application procedures of resin cements

Resin cement	Application procedure
Multilink N (Ivoclar Vivadent Liechtenstein)	Tooth surface: Clean, rinse, dry with air that is free of water and oil.
	Mix Multilink N Primer A and B in a 1:1 ratio. Apply the mixed Multilink N Primer A/B with disposable
	microbrush to the entire bonding surfaces, scrub (30 s), and disperse excess with blown air until the
	mobile liquid film is no longer visible.
	Dispense Multilink N catalyst and base from the double-push syringe and mix in a 1:1 ratio. Remove
	the excess cement.Light-polymerize (all margins 20 s).
RelyX U200 Automix (3M ESPE, Germany)	Tooth surface: Clean, rinse, lightly air dry in only 2–3 bursts of water-free and oil-free air. Dispense
	RelyX U200 Automix catalyst and base from the double-push syringe and mix in a 1:1 ratio. Remove
	the excess cement. Light-polymerize (single surface 20 s; any other surface, additional 20 s).

Table 4. Results of three-way ANOVA

Source	Type III sum of squares	df	Mean square	F	p value
Tooth structure (A)	87.340	1	87.340	6.649	.011
Resin cements (B)	1411.635	1	1411.635	107.464	.000
CAD-CAM blocks (C)	191.300	2	95.650	7.282	.001
A×B	0.278	1	0.278	0.021	.885
A×C	9.914	2	4.957	0.377	.686
B × C	33.771	2	16.886	1.285	.279
$A \times B \times C$	38.427	2	19.213	1.463	.235

Table 5. SBS (MPa) means and standard deviations for all experimental groups

			<u> </u>		
	Sound dentin		Eroded dentin		
	Multilink N	RelyX U200 Automix	Multilink N	RelyX U200 Automix	
Lava Ultimate	11.06±2.33 ^{Aa}	7.73±1.59 ^{Ab*}	11.35±2.61 ^{Aa}	5.90±1.04 ^{Ab*}	
Vita Enamic	15.86±4.62 ^{Ba}	8.27±2.32 ^{Ab}	13.16±5.28 ^{Aa}	7.97±2.97 ^{Ab}	
Vita Suprinity	15.06±3.31 ^{Ba}	8.95±3.24 ^{Ab}	13.06±7.09 ^{Aa}	7.14±2.49 ^{Ab}	

Different uppercase letters indicate a statistically significant difference among CAD-CAM blocks (p<0.05). Different lowercase letters indicate a statistically significant difference between resin cements (p<0.05). * indicates a statistically significant difference between sound and eroded dentin (p<0.05).

Results

The analysis of variance and significant differences for different factors and interactions are presented in Table 4. The 3-way ANOVA showed that the three main factors, tooth structure (sound dentin or eroded dentin) (p=0.011), resin cements (RU or MN) (p<0.001), and CAD-CAM blocks (LU or VE or VS) (p=0.001) significantly affected the SBS.

SBS means and standard deviations for all groups are presented in Table 5. The highest values were obtained when VE was used with MN (15.86 ± 4.62 MPa) in sound dentin and (13.16 ± 5.28 MPa) in eroded dentin. The lowest values were obtained when LU was used with RU (7.73 ± 1.59 MPa) in sound dentin and (5.90 ± 1.04 MPa) in eroded dentin. Generally, higher SBS values were obtained in sound dentin than in eroded dentin, but the difference was significantly only when the LU was used with RU in sound dentin(p=0.001). MN showed higher SBS than RU. The difference was significantly for all groups (p<0.05). When LU, VE, and VS were used with MN in sound dentin, LU showed lower SBS than VE (p=0.002) and VS (p=0.011). When LU, VE, and VS were used with MN in eroded dentin, LU showed lower SBS than VE and VS, but the difference was not significantly. When LU, VE, and VS were used with RU in sound and eroded dentin, LU showed lower SBS than VE and VS, but the difference was not significantly. There was not significantly difference between the SBS of VE and VS in all groups. The failure modes are shown in Figure 2. For LU, VE, and VS, the predominant failure mode was mixed.

Discussion

In the present study, it was aimed to assess the shear bond strength of three chairside CAD-CAM blocks to dentin subjected to simulated gastric erosion when cemented two resin cements. The null hypothesis that there would be no effect of intrinsic dental erosion, resin cements, and CAD-CAM blocks on SBS to dentin was rejected. Significant differences were found for the three main factors.

Erosion, abrasion, and attrition are not usually seen alone but interact with each other. Abrasion of dental hard tissues affected by erosion is considered the most important interaction.²⁷ Therefore, in this in vitro study, the erosion protocol of Hove et al. was used to simulate intrinsic gastric erosion²⁶, and erosion-abrasion protocol was applied together to simulate better conditions in daily life.²⁷

In this study for erosion procedure HCI (0.01 M, pH 2, 2 min) was applied to dentin samples, then samples were stored in artificial saliva for 60 min. For abrasion procedure a power brush was used for 15 s. A demineralization period of 2 min shows the duration of the pH drop in saliva after the acid attack.³ Although the pH of pure gastric acid is between 0.9 and 1.5, it is rarely lower than 1.5 due to the buffering effect in the esophagus and the dilution effect of food and drinks. It is more convenient to use 0.01 M HCl than 0.1 M HCl for clinical conditions.²⁶ Not to brush immediately after contact with acid may represent better conditions in daily life, because people are unlikely to brush their teeth immediately after every erosive attack, so in this study, after-erosion dentin samples were stored in artificial saliva for 60 min.²⁷ In this study, a brushing force of 2N was used in accordance with ISO 14569-1 820075.5

Bond strength to eroded dentin has been evaluated in few studies.¹⁹⁻²⁴ It has been shown that the bond strength of adhesive systems to eroded dentin is lower than sound dentin.^{20,21,23} In some studies, no significantly difference was found in the bond strength of sound and eroded dentin.^{19,22} There is no consensus among studies regarding the erosion procedures used. In these studies, citric acid^{19,20}, acidic drinks²¹⁻²³ and HCl-pepsin solution²⁴ were used while performing the extrinsic erosion procedure. In this study, unlike existing studies, HCl was used to simulate intrinsic erosion, then abrasion procedure was performed. According to the findings of this study, eroded dentin exhibited lower bond strength values than sound dentin. When dentin is eroded, changes such as opening of dentin tubules, removal of the organic part of intertubular dentin and dentin plugs, increased tubule diameter, and collagen exposure may occur.⁷ It becomes difficult for the adhesive to infiltrate into exposed collagen.²⁰ In addition, when dentin is eroded, the mineral components dissolve and the organic dentin matrix is released.^{7,8} The presence of a thickened superficial organic layer could lead to the assumption that adhesive penetration would be impaired, producing lower bond strengths in eroded dentin.¹⁹

In this study, as reported by previous studies, self-etch resin cement showed higher bond strength in all groups than self-adhesive resin cement.²⁸⁻³⁰ The reasons the bond strength of self-adhesive resin cements is lower than conventional cements are that the acidic monomers in resin have limited etching potential for demineralization, insufficient pH neutralization after curing¹⁸, the higher viscosity of the cement prevents deeper resin

penetration¹⁷, and non-removal or incomplete removal of the smear layer creates a weaker bond at the interface.³⁰ In addition, self-adhesive resin cement's increased hydrophilicity can compromise mechanical strength¹⁷, which may be one reason for reduced bond strength. Furthermore, chemical composition differences, surface wetting properties, viscosity and mechanical properties of resin cements may be among the factors affecting bond strength.³¹

Flury et al. cemented LU and VE to dentin with five resin cements and reported that after 24-h storage, SBS did not significantly differ between LU and VE.³² A previous study stated that the bond strength of LU was lower than that of VE.³³ In this study, LU showed lower SBS in all groups than VE and VS. The difference was significantly only when used with MN in sound dentin. This finding can be explained by the differences in the microstructural properties such as filler type and concentration, material composition and mechanical properties of the LU, VE, and VS. It has been reported that hydrofluoric acid (HF) etching and silane application is an effective surface preparation protocol for VE and VS.^{14,33} HF enhances bond strength due to the glass matrix structure in VE and VS.^{33,34} Bellan et al. evaluated the dentin bond strength of LU, VE, and VS and reported that LU and VE showed significantly higher µTBS than did VS, which may be due to the differences in the modulus of elasticity of restorative materials.¹⁵ The difference from the present study is that different surface preparation protocols were applied to the blocks. LU, VE, and VS were sandblasted with 50 μ m aluminum-oxide (Al₂O₃) particles. In the present study, only LU was sandblasted with Cojet. VE and VS was etched with hydrofluoric acid, then silane was applied to the blocks surfaces. According to the manufacturer's instructions, the Cojet sandblasting application was recommended in the surface preparation protocol of LU (Table 2), but there was no clear indication about the application time. The difference made at this stage may cause a decrease in the surface energy of the LU. These reasons can explain lower the SBS when LU was used in this study.

According to Elsaka, mixed failure is associated with increased bond strength, whereas adhesive failure indicates low bond strength.³⁵ In this study for all groups, the most commonly observed mode of failure was mixed. Increased adhesive failure when LU, VE, and VS with RU were used in eroded dentin can be expressed by the lower bond strength that self-adhesive cement exhibits.

In the present study, SBS after 24 h was examined. The main limitation of this study was the lack of aging procedures. Therefore, clinical and laboratory studies are needed to assess the long-term effect of erosion on the SBS of CAD-CAM blocks to dentin.

Conclusions

Within the limitation of this in vitro study, it can be concluded that dental erosion affected the SBS to dentin, and eroded dentin showed generally lower SBS. The use of self-etch resin cement system, Multilink N can be recommended due to higher SBS values compared to selfadhesive cement. In terms of material type, Vita Enamic and Vita Suprinity showed higher SBS in comparison to Lava Ultimate. The type of material and cement system chosen by the clinician is important to provide better bond strength to the eroded dentin structure.

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Conflict of Interest

The authors declare that they have no conflict of interest.

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