Original Article

Effect of Adhesive Resin Cements on Bond Strength of Ceramic Core Materials to Dentin

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Abstract

Objectives: The aim of the present study was to evaluate the effects of self-etch and self-adhesive resin cements on the shear bond strength of ceramic core materials bonded to dentin. **Materials and Methods:** Extracted, caries-free, human central maxillary incisor teeth were selected, and the vestibule surfaces were cut flat to obtain dentin surfaces. Ceramic core materials (IPS e.max Press and Prettau Zirconia) were luted to the dentin surfaces using three self-etch adhesive systems (Duo-Link, Panavia F 2.0, and RelyX Ultimate Clicker) and two self-adhesive resin systems (RelyX U200 Automix and Maxcem Elite). A shear bond strength test was performed using a universal testing machine. Failure modes were observed under a stereomicroscope, and bonding interfaces between the adhesive resin cements and the teeth were evaluated with a scanning electron microscope. Data were analyzed with Student's *t*-test and one-way analysis of variance followed by Tukey's test ($\alpha = 0.05$). **Results:** The type of adhesive resin cement significantly affected the shear bond strengths of ceramic core materials bonded to dentin ($P < 0.05$). Significant differences were noted between the ceramic core materials when the specimens were luted with self-adhesive resin cements ($P < 0.05$). The specimens luted with RelyX Ultimate Clicker had the highest shear bond strengths. **Conclusion:** The self-etch adhesive resin cements exhibited better shear bond strength than the self-adhesive resin cements, except for Panavia cement in the IPS e.max Press group. However, shear bond strengths of the self-adhesive resin cements were dependent on the nature of the ceramic core materials.

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Introduction

Fundamentally, prosthetic rehabilitation includes the reestablishment of natural tooth appearance, a beautiful smile, masticatory function, and phonation, all of which should take place after an accurate diagnosis and proper treatment planning. The treatment of teeth that present with large dental caries, discoloration, malformation, midline diastemas, or other cosmetic problems, or that are simply unsightly, constitutes an esthetic challenge for the clinician and can also be socially and psychologically distressing for patients.[1-3]

Several treatment options have been described for the rehabilitation of unesthetic anterior teeth.[4] Metal-ceramic restorations have been used successfully in both anterior and posterior regions, due to the combination of the high fracture strength of the metal substructure and the esthetic properties of the ceramic veneer.[5] The carious lesions can be restored using amalgam and gold restorations; though these exhibit excellent long-term results, they are no longer preferred by patients.[3,6] An increased patient demand for tooth-colored restorations, improvements in adhesive dentistry and computer-aided design/computer-aided manufacturing technologies, the inevitable corrosion of metal restorations, and allergies to

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metal have increased the need for all-ceramic restorations such as crowns, laminate veneers, inlays, onlays, and other restorations. All-ceramic restorations are biocompatible and provide excellent aesthetic restoration with a lifelike appearance, as a wide range of colors can be prepared.[5,7-10]

The success and longevity of all-ceramic restorations are affected by their fracture strength and also by the adhesion between the restoration and the tooth.[11] Strong adhesion between the ceramic materials and the tooth structure can be achieved by adhesive cementation, which provides mechanical integration of the system, thus increasing the fracture strength. In addition, durable adhesion improves marginal adaptation and decreases microleakage and secondary caries.[12-14] In recent years, lithium-disilicate glass ceramics have widely been used to fabricate all-ceramic restorations because of their superior esthetic features and metal oxides (alumina and zirconia) as well as their superior mechanical features.[11]

The type of cement and the particular cementation technique used also play significant roles in the clinical success and longevity of all-ceramic restorations. Resin cements are recommended for cementation of all-ceramic restorations, especially those that use silica-based ceramics.[15-17] The resin cement penetrates into the irregularities of the restoration's internal surface, and the tooth/resin cement/ceramic interface allows an effective transfer of stress from the ceramic to the tooth, which will prevent crack initiation.[18-20] Additional advantages of resin cements are that they are more translucent than glass-ionomer and phosphate cements, and they are available in many different shades.[19]

The major disadvantage of resin cements is evident when they are used in conjunction with an adhesive system that is either total-etch or self-etch, and only clinically successful marginal sealing is achieved. Self-etch systems are accepted among clinicians because they are easy to use. However, they have shown a weaker bond strength to enamel than that of total-etch systems. Therefore, three-step total-etch systems are still considered the gold standard. Yet, pretreatments of the tooth structure – by etching, priming, and bonding during adhesive cementation – are complex, time-consuming, and technically sensitive. To overcome these problems, self-adhesive resin cements have been developed.[16,21,22] In addition, resin cements have disadvantages, such as color staining in the anterior region and hygroscopic expansion.[23]

Self-adhesive resin cements are hybrid materials that can integrate the properties of conventional cements and adhesive resin cements.[21,24] Self-adhesive resin cements adhere to tooth structures and restorative materials without requiring the application of a separate pretreatment or adhesive. Therefore, self-adhesive resin cements are also easy to use and can be applied in a single step. In addition, they release fluoride, tolerate moisture, and produce little to no postoperative sensitivity, because the smear layer is not removed.[24-26]

Limited information exists in the literature regarding the evaluation of the adhesive properties of self-adhesive resin cements. Although it is important to determine the adhesion between the ceramic materials and the tooth structure for better clinical significance, published studies on the bond strength of all-ceramic restorations have used stainless steel, composite resin blocks, or resin cement as the bonding substrates without considering the realities of teeth. This situation does not reflect normal clinical practice, in which the restorations are typically bonded to the tooth structure.[27,28] Therefore, the purpose of the present study was to evaluate the effects of self-etch and self-adhesive resin cements on the shear bond strength of ceramic core materials bonded to dentin. The hypotheses were that: (1) the adhesive resin cements would not affect the shear bond strength between the ceramic core materials and the dentin, and (2) the shear bond strengths of different ceramic core materials would be similar.

Materials and Methods

The present study was approved by the Ethics Committee at Atatürk University (Protocol No. 7, 27.09.2013). The shear bond strength test was carried out following the guidelines of ISO/TS 11405:2003.[29]

Eighty extracted, caries-free, human central maxillary incisors were selected, cleaned of both calculus deposits and soft tissues, and stored in 0.5% chloramine-T (Halamid, Axcentive, Bouc Bel Air, France) at 4°C for 1 week. The extracted teeth were then kept in distilled water at 4°C during the study. The distilled water was changed regularly each week. The tooth roots were cut with a diamond bur (Dia-Burs, SF-11C, Mani Inc., Japan) at high speed under water spray coolant. The crowns were embedded in autopolymerizing acrylic resin (Imıcryl, Konya, Turkey) using a plastic tube with a diameter and height of 20 mm and with the vestibule surfaces facing upward. To obtain perfectly parallel surfaces suited for bonding to the ceramic core materials, the dentin substrate was exposed with a diamond saw at low speed with water cooling (Isomet, Buehler, Lake Bluff, IL, USA). The dentin surfaces were finished with 600-grit abrasive paper to create a uniformly flat surface.

Two types of commercially available ceramic core materials were used in the present investigation: IPS e.max Press (Ivoclar Vivadent, Schaan, Liechtenstein) and Prettau Zirconia (Zirkonzahn GmbH, Gais, Italy). Forty ceramic core materials for each group were prepared according to the manufacturer's instructions; the core materials were 3 mm in diameter and 3 mm in thickness. After the ceramic core materials had been prepared, the bonding surfaces of all the specimens were finished with 600-grit abrasive paper to create a uniform surface.

Before cementation of the ceramic core materials to the dentin, the ceramic surfaces were treated as follows: the IPS e.max Press group was etched with 9.5% hydrofluoric acid (Porcelain Etchant, Bisco, Schaumburg, IL, USA) for 60 s, rinsed with water spray, and allowed to air dry; the Prettau Zirconia group was treated with airborne-particle abrasion with 50 µm Al_2O_3 (Airsonic, Hager and Werken, Duisburg, Germany) for 15 s at a pressure of 0.25 MPa from a distance of 10 mm, rinsed with water spray, and air dried. The dentin received no treatment.

Three self-etch adhesive systems and two self-adhesive resin systems [Table 1] were used to bond the ceramic core materials to the tooth structure and were handled according to the manufacturer's instructions, which are summarized in Table 2. The adhesive resin cements were placed on the ceramic core substrates, which were then bonded to the dentin under a pressure of 10 newtons (N). Light polymerization for 2–5 s with a light-emitting diode polymerizing unit (HS LED 1500, Henry Schein Inc., Melville, NY, USA) was performed to remove the excess cement. After the pressure and the excess cement had been removed, light polymerization was performed on each specimen's vestibule surfaces according to the manufacturer's instructions. The specimens were then stored in distilled water at 37°C ± 2°C for 24 h before the shear bond strength test.

The shear bond strength test was carried out on a universal testing machine (Model 2519-106, Instron Corp, Norwood, MA, USA). All specimens were loaded to failure at a crosshead speed of 0.75 mm/min with a knife-edge chisel placed in contact with and parallel to the vestibule surface of the specimens, and the maximum failure load was recorded in N. The shear bond strength values of each specimen were calculated in MPa by dividing the failure load (N) by the bonding area (mm^2).

The failure mode was assessed by inspecting the bonding surfaces of each specimen under a stereomicroscope (Olympus, Tokyo, Japan). The failure modes were classified as follows: (1) cohesive failure in the dentin; (2) adhesive failure between the dentin and the adhesive resin cement; (3) cohesive failure in the adhesive resin cement; (4) adhesive failure between the adhesive resin cement and the ceramic core material; and (5) cohesive failure in the ceramic core material.

An additional specimen from each test group was prepared to evaluate the bonded interface between the dentin and the ceramic core material for the scanning electron microscope (SEM) analysis. After these specimens had been stored in distilled water at 37°C ± 2°C for 24 h, the teeth were sectioned vestibulolingually through the restoration. The specimen interfaces were abraded with 400-, 600-, 800-, and 1000-grit abrasive papers. The bonded interfaces were etched with 35% phosphoric acid for 15 s, rinsed with water spray, and allowed to air dry. These specimens were then gold sputtered and examined with a field emission SEM (Inspect S50, Fei, Hillsboro, OR, USA) at ×2000 magnification.

Data from the shear bond strength tests were analyzed using a one-way analysis of variance and Tukey's honestly significant difference test with a confidence level of 0.05 to determine differences among the means. Student's *t*-test was used to compare differences between the bond strengths for the IPS e.max Press and Prettau Zirconia groups (α = 0.05). The statistical analyses were performed with Statistical Package for the Social Sciences (SPSS) statistical software (SPSS version 16.0, SPSS Inc., Chicago, IL, USA).

Results

Table 3 shows the mean and standard deviation of the shear bond strength for each of the experimental groups. The highest shear bond strength was observed in the Prettau Zirconia specimens luted with RU cement (17.44 ± 2.78 MPa), while the lowest was observed in the Prettau Zirconia specimens luted with ME cement (4.42 ± 1.53 MPa).

The shear bond strengths in the IPS e.max Press group were RU > D > RA > ME > P. The specimens luted with

Table 1: Adhesive resin cements used in the present study

Adhesive resin cements	Manufacturer	Group	Abbreviation
Duo-Link SE Kit	Bisco, Schaumburg, USA	Self-etch adhesive system	D
Panavia F 2.0 Complete Kit	Kuraray Noritake Dental Inc., Japan	Self-etch adhesive system	P
RelyX Ultimate Clicker	3M ESPE, 3M Deutschland GmbH, Germany	Self-etch adhesive system	RU
Maxcem Elite	Kerr, Salerno, Italy	Self-etch/self-adhesive system	ME
RelyX U200 Automix	3M ESPE, Neuss, USA	Self-adhesive system	RA

P cement had significantly lower shear bond strength than those luted with D and RU cements ($P < 0.05$). No significant differences were found among P, RA, and ME cements; D, RA, and ME cements; or D and RU cements ($P > 0.05$). However, the specimens luted with RU cement had significantly higher shear bond strength than those luted with P, RA, and ME cements ($P < 0.05$).

The shear bond strengths in the Prettau Zirconia group were RU > D > P > RA > ME. The specimens luted with RU and D cements had significantly higher shear bond strength than those luted with the other cements ($P < 0.05$). No significant differences were noted between RU and D cements; P and RA cements; or RA and ME cements ($P > 0.05$). In addition, P cement

Table 2: Bonding procedure

Material	Procedure
Duo-Link SE Kit	
All-Bond SE Parts I and II	Dispense Parts I and II in a ratio 1:1, mix until uniformly pink, apply 1-2 coats to the dentin, gently air dry for 5 s, light cure for 10 s
Porcelain Primer	Apply the bondable surface of I group for 30 s, dry with an air syringe
Z-Prime Plus	Apply the bondable surface of Z group, dry with an air syringe for 3-5 s
Panavia F 2.0 Complete Kit	
ED Primer II Liquid A and B	Dispense Liquid A and B in a ratio 1:1, apply to the dentin, leave it in place for 30 s, use a cotton pellet to carefully remove any excess ED Primer II, gently air dry
Clearfil Ceramic Primer	Apply the bondable surface of I group, gently air dry
Alloy Primer	Apply the bondable surface of Z group
RelyX Ultimate Clicker	
Single Bond Universal	Apply the dentin for 20 s, gently air dry for 5 s
	Apply the bondable surface of I and Z groups, gently air dry for 5 s
Maxcem Elite	
Silane Primer	Apply the bondable surface of I group, gently air dry for 5-10 s
OptiBond All-In-One	Apply the bondable surface of Z group for 20 s, apply a second application, air dry at least 5 s
RelyX U200 Automix	
RelyX Ceramic Primer	Apply the bondable surface of I group for 5 s, gently air dry
Alcohol	Clean the bondable surface of Z group, air dry

Table 3: Mean (standard deviation) shear bond strength values (MPa) of all experimental groups

Adhesive resin cements	IPS e.max press	Prettau Zirconia
Duo-Link SE	15.77 (3.63)^{b,c,A}	16.95 (3.55)^{a,A}
Panavia F 2.0	11.71 (1.73)^{a,A}	11.14 (2.69)^{b,A}
RelyX Ultimate Clicker	17.06 (2.43)^{c,A}	17.44 (2.78)^{a,A}
RelyX U200 Automix	13.33 (1.97)^{a,b,A}	7.68 (1.76)^{b,c,B}
Maxcem Elite	12.96 (1.85)^{a,b,A}	4.42 (1.53)^{c,B}

Vertically, significant difference between means are characterized by different lowercase letters; horizontally, significant difference between means are characterized by different uppercase letters

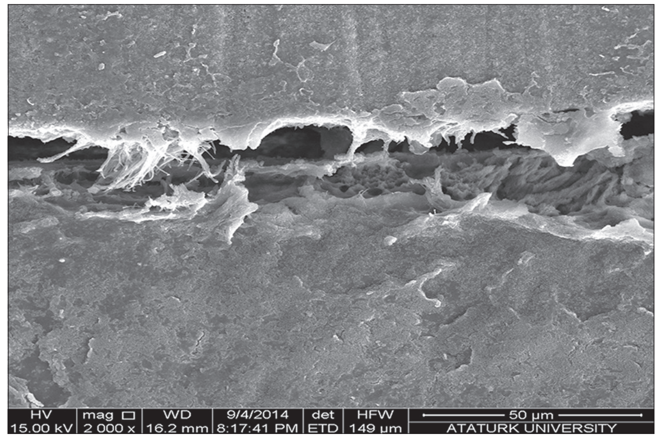

Figure 1: Scanning electron microscopy image of D cement-dentin interface

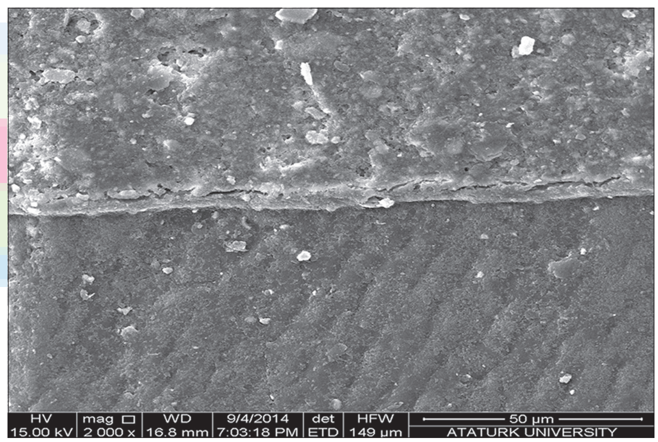

Figure 2: Scanning electron microscopy image of P cement-dentin interface

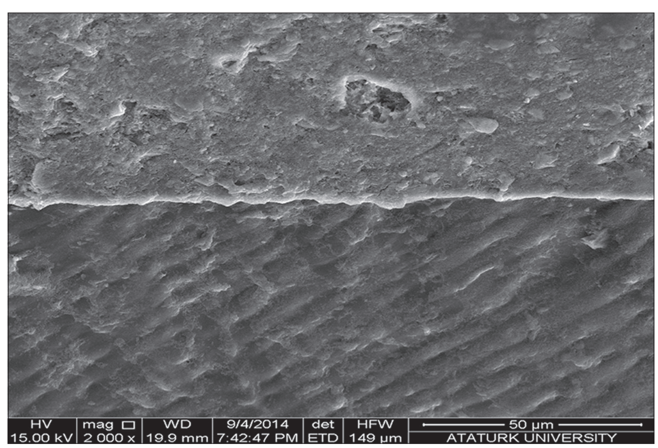

Figure 3: Scanning electron microscopy image of RU cement-dentin interface

Table 4: Failure mode distributions of experimental groups

Ceramic core materials	Adhesive resin cements	Cohesive: In dentin	Adhesive: Dentin-cement	Cohesive: In cement	Adhesive: Cement-ceramic	Cohesive: In ceramic
IPS e.max Press	Duo-Link SE Kit	0	3	4	1	0
	Panavia F 2.0 Complete Kit	0	4	4	0	0
	RelyX Ultimate Clicker	1	2	5	0	0
	RelyX U200 Automix	0	5	2	1	0
	Maxcem Elite	0	3	3	2	0
Prettau Zirconia	Duo-Link SE Kit	1	3	4	0	0
	Panavia F 2.0 Complete Kit	0	2	5	1	0
	RelyX Ultimate Clicker	2	2	2	2	0
	RelyX U200 Automix	0	8	0	0	0
	Maxcem Elite	0	0	6	2	0
Total (%)		5	40	43.75	11.25	0

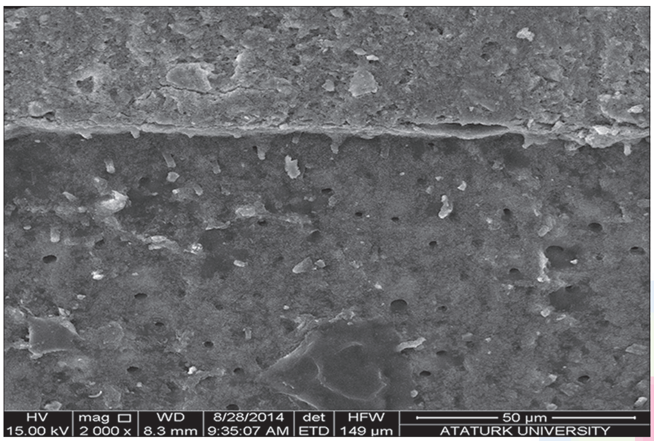

Figure 4: Scanning electron microscopy image of RA cement-dentin interface

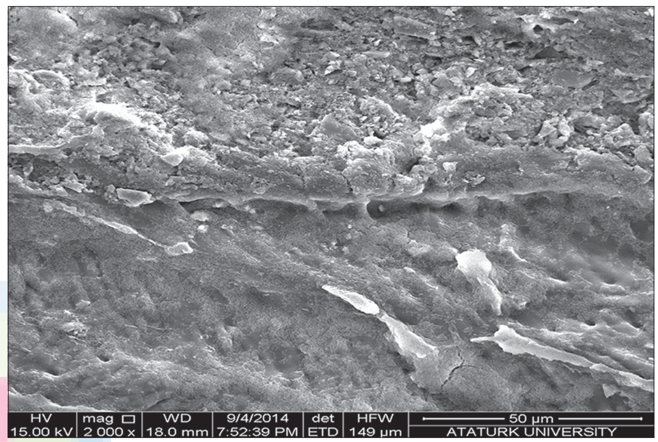

Figure 5: Scanning electron microscopy image of ME cement-dentin interface

had significantly higher shear bond strength than ME cement ($P < 0.05$).

No significant differences were observed between the IPS e.max Press and Prettau Zirconia specimens luted with D, P, and RU cements ($P > 0.05$). However, the IPS e.max Press specimens that were luted with RA and ME cements had significantly higher shear bond strength than those of the Prettau Zirconia specimens ($P < 0.05$).

Table 4 shows the failure mode distributions of the experimental groups. The failure modes were 43.75% cohesive failure for the adhesive resin cement, 40% adhesive failure between the dentin and the adhesive resin cement, 11.25% adhesive failure between the adhesive resin cement and the ceramic core material, and 5% cohesive failure in the dentin. No cohesive failures in the ceramic core material were observed.

Representative SEM images of the adhesive resin cement/dentin interface with D cement are shown in Figure 1, images of P cement are presented in Figure 2, RU cement is shown in Figure 3, RA cement is shown in Figure 4, and ME cement is shown in Figure 5. In each group, superficial demineralization and a thinner transitional layer without the formation of a thicker hybrid layer or longer resin tags can be seen. In addition, the resin cement/dentin interface had a superficial interaction with no strong micromechanical interlocking.

Discussion

The present study evaluated the effects of self-etch and self-adhesive resin cements on the shear bond strength of ceramic core materials bonded to dentin. The first null hypothesis was partially rejected because the data showed that the resin cement type significantly affected the shear bond strength of ceramic core materials ($P < 0.05$). Significant differences were observed for the ceramic core materials luted with self-adhesive resin cements ($P < 0.05$). Thus, the second null hypothesis was partially rejected.

Surface treatments are important in strengthening the bonding between adhesive resin cements and ceramic core materials, as well as in the preparation of enamel and dentin surfaces for bonding between tooth and adhesive resin cement in tooth/cement/ceramic combination.

A durable adhesive resin bond achieves micromechanical interlocking and chemical bonding to the ceramic's inner surface.[12,30] Silica-based ceramics have been successfully bonded to adhesive resin cements by etching the ceramic surface with hydrofluoric acid, then applying a silane coupling agent.[20,31] Airborne-particle abrasion or tribochemical silica coatings are recommended for alumina or zirconia ceramics.[12,32] In addition, primers can improve the bonding of adhesive resin cements to zirconia.[33,34] Therefore, in the present study, the IPS e.max Press specimens were etched with 9.5% hydrofluoric acid, and the Prettau Zirconia specimens were treated with airborne-particle abrasion using 50 µm Al_2O_3, in accordance with previous studies and the manufacturer's instructions.

Zhang and Degrange[35] evaluated the effects of total-etch (Variolink), self-etch (Multilink Automix), and self-adhesive (RelyX Unicem, Multilink Sprint, and Maxcem) resin cements on the shear bond strength of different restorative materials bonded to dentin. They reported that total-etch and self-etch adhesive resin cements had significantly higher bond strengths than self-adhesive resin cements, and Maxcem cement had the lowest shear bond strength in the glass ceramic group ($P < 0.05$). However, they found that the self-etch adhesive resin cement had significantly higher shear bond strength than the total-etch adhesive resin cement ($P < 0.05$), but the self-etch adhesive resin cement was not significantly different from the self-adhesive resin cements, except for Maxcem cement, in the zirconia ceramic group ($P > 0.05$). In contrast, in the present study, no significant differences were found between the self-adhesive resin cements (RA and ME; $P > 0.05$). In addition, the self-etch adhesive resin cements, which were RU cement in the IPS e.max Press group and RU and D cements in the Prettau Zirconia group, had significantly higher shear bond strength than the self-adhesive resin cements ($P < 0.05$). The lower shear bond strength of self-adhesive resin cements may be explained by the fact that these cements cannot completely remove the smear layer that can cause a weak hybrid layer between the resin cement and the dentin.[35,36]

The results of the present study demonstrated that the ceramic core materials luted to dentin with RU cement had the highest shear bond strength. This is because the resin cement is used with an adhesive system (Single Bond Universal) that includes 10-methacryloyloxydecyl dihydrogen phosphate (MDP). This adhesive system has been demonstrated to have a durable adhesion to dentin.[37] In the present study, the durable adhesion to dentin was also supported by the low cohesive failure in the dentin. However, the ceramic core materials luted to dentin with *P* cement, which is used with an adhesive system (ED Primer II) that also includes MDP, had significantly lower shear bond strength than those luted with D and RU cements ($P < 0.05$). One reason may be that higher filler content decreased the penetration of adhesive resin into the dentin.[38] Another reason may be that the self-etching ED Primer permits water-induced interfacial changes, resulting in lower cement/dentin bond strengths.[39]

In the present study, significant differences were noted between the IPS e.max Press and Prettau Zirconia specimens when these ceramic core materials were luted with RA and ME self-adhesive resin cements ($P < 0.05$). Similarly, Sabatini *et al.*[40] reported that the shear bond strength of the cements was dependent on the nature of the prosthodontic substrate; therefore, the cement should be chosen partially according to the substrate. These results indicate that self-adhesive resin cements may have specific adhesion to certain ceramic core materials, due to the chemical structure of these cements, which contain functional monomers.[35]

The current adhesive systems are easy to use, are not technically sensitive, and provide durable adhesion to dentin. The shear bond strength between adhesives and dentin has been estimated to range from 15 to 20 MPa.[41,42] The bond strength values were equal to or >20 MPa, which was acknowledged as clinically sufficient.[32] Besides, the bond strength of adhesives is associated with the mineral content in tooth structures.[43,44] In the present study, the experimental groups had bond strength values lower than 15 MPa, except for RU and D cements.

It should be noted that the shear bond strength was associated with two interfaces (dentin/resin cement and resin cement/ceramic) in the present study. The failure mode analysis provided important knowledge about the behavior of the system. In the shear test, the stress distribution on the bonding area was heterogeneous, especially when a knife-edge chisel was used. Therefore, the failure mode results may be questionable.[45] However, the shear stress is considered to be more representative of a clinical situation.[42] Theoretically, when there are two interfaces, the failure should begin from the weaker interface.[28] In the present study, the dentin/resin cement interface was weaker than the resin cement/ceramic interface for all experimental groups, except the ME group. Cohesive failure is more likely occur in the adhesive resin cement and tooth structure for shear bond strength values that are higher than 18–20 MPa.[46] In the present study, cohesive failure was seen most often when self-etch adhesive resin cements were used, while adhesive failure was more common when self-adhesive resin cements were employed. In other words, cohesive failure was more likely in the experimental groups that had a higher shear bond strength.

The bonding agents of adhesive resin cements that were used in the present study were mild and ultra-mild self-etch systems. Mild self-etch systems demineralize dentin superficially and keep residual hydroxyapatite attached to the collagen fibers. Thus, functional monomers in the mild self-etch adhesives may chemically bond to the residual hydroxyapatite.[46-48] Self-adhesive resin cements interact superficially with mineralized tissue and are not able to demineralize the smear layer completely; thus, the hybrid layer is not created. Because this interaction involves more chemical than micromechanical interlocking, resin tags cannot be seen in the hybrid layer.[35,36] This result clarified that the SEM images of smooth and superficial interacted with the adhesive resin cement/tooth structure interface without resin tags.

There are a few limitations of the present investigation when compared to a clinical situation. First, the specimens were stored in distilled water at 37°C ± 2°C for 24 h (short-term) before the shear bond strength test. Because the effects of long-term storage and thermocycling on bond strength were not assessed, the *in vitro* nature of this investigation may not simulate the changes in bond strength and degradation of adhesive resin cements after long periods of time under oral fluid conditions. In addition, it is not possible to imitate the static, chemical, and cyclic mechanical fatigue phenomena of the ceramic material-tooth structure interfaces with *in vitro* studies. These phenomena are important for simulating the clinical situation and should be investigated further in future *in vitro* studies. However, the results should also be verified by long-term clinical research.

Conclusion

Within the limitations of the present investigation, the following conclusions were drawn:

1. The self-etch adhesive resin cements exhibited better shear bond strength than the self-adhesive resin cements, except for P cement in the IPS e.max Press group
2. The shear bond strengths of self-adhesive resin cements were dependent on the nature of the ceramic core materials.

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Conflicts of interest

There are no conflicts of interest.

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