





Shear Bond Strength of E. Max Ceramic Restoration to Hydraulic Calcium Silicate Based Cement (Biodentine): An *In Vitro* Study

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ABSTRACT

Objective: The purpose of this study was to evaluate the shear bond strength (SBS) of hydraulic calcium silicate (Biodentine) as a core material to the e.max ceramic restoration.

Methods: Forty discs (6 mm diameter; 2 mm thickness) were fabricated from each core material, Hydraulic calcium silicate [Biodentine™, Septodont], resin composite [Filtek™Z250 XT, 3M ESPE], and resin-modified glass ionomer cement (RMGIC) [GC Fuji II LC, GC Corporation]. Dentine surfaces of 40 extracted human permanent molars were exposed and used as a control group. All specimens were mounted in self-curing acrylic resin. One hundred sixty IPS e.max discs were fabricated (4 mm diameter; 2 mm thickness) and cemented to the core specimens with Variolink N (IvoclarVivadent). After storage in distilled water (37°C; 24h), the specimens were thermocycled 1.500 times. SBS was tested using a universal testing machine at 0.05 mm/min crosshead speed. The fracture modes were determined by a stereomicroscope at ×20 magnification. Data were analyzed using one-way analysis of variance followed by Tukey's test (P=0.05).

Results: The mean SBS values of four tested groups showed statistically significant differences (P<0.05). The resin composite group exhibited the highest SBS value (36.17±6.08 MPa), while the Biodentine had the lowest SBS value (21.86±3.18 MPa). Mixed failure mode was the most common failure type in all tested groups except in the Biodentine group, which had a predominantly cohesive failure.

Conclusion: The SBS of e.max ceramic restorations cemented with resin is affected by the type of core material. Biodentine core material had the lowest SBS to e.max restoration. However, when Biodentine is indicated to be used as core material for pulp preservation, it is recommended to be covered with a layer of resin composite material to enhance its bonding strength to the e.max restoration.

Keywords: Bond strength, calcium silicate, Filtek Z250, Fuji II LC cement, IPS e.max, variolink

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HIGHLIGHTS

- The success of e.max ceramic restoration cemented with resin luting cement depends on the type of core material.
- The resin composite core material has the strongest bond to the e.max ceramic restoration with resin-luting cement.
- Hydraulic calcium silicate core (Biodentine) is not advocated to be used as core material for e.max restoration because of its low bond strength.
- If Biodentine material is indicated to be used in vital pulp therapy cases, it should be covered by a layer of a resin composite material as a core for e.max ceramic restoration.

INTRODUCTION

Maintaining dental pulp vitality is one of the ultimate objectives of endodontic therapy (1). When the vitality of the pulp is preserved, the aesthetic outcome of the permeant restoration is enhanced alongside patient satisfaction (2, 3). With development in ceramic restorations and adhesive systems, preservation of the remaining tooth structure of vital teeth can be attained with an optimal clinical performance and aesthetic results (4). Lithium disilicate ceramic is an example of the ceramic restorative materials and has been introduced in the markets under the brand name of

IPS e.max Press. This aesthetic restoration is commonly fabricated as inlays, onlays, and crowns, especially in restoring the posterior teeth (5).

One important factor that affects the success of ceramic restorations is the bonding of the luting agent to dentine or core materials (6, 7). Core materials are commonly used to replace the lost coronal tooth structure, maintain pulp vitality, and bond to the coronal restorations and crowns (8). The core materials must have enough strength to withstand chewing forces (9). Various restorative materials are available to build up the missing tooth structure such as amalgams, resin composites, glass ionomers, and resin-modified glass ionomers. Each of these materials possesses advantages and disadvantages, depending on the patient need's (8, 9).

It has been long acknowledged that the bonding strength of both luting cement and tooth substance is typically affected by the type of core material (10). Resin composite is an aesthetic restorative material that bonds to the tooth structure and does not require retentive features in the tooth preparation. The resin composite core has also higher flexural and compressive strengths when compared to amalgam and resin-modified glass ionomer restorations (9). In a previous study, the resin composite core showed the highest bonding strength to the ceramic restoration amongst different tested core materials including glass ionomers and ceramic-based materials (11). However, the use of resin composite material is considered a technique sensitive which requires adequate moisture control (8). Animal studies have also reported the hypersensitivity and cytotoxicity of the resin composite core to the pulpal and subcutaneous tissues (12, 13). Conversely, resin-modified glass ionomer cements (RMGICs) cause less pulpal inflammation when compared to resin composite restorations (12). The RMGICs exhibit higher mechanical properties compared to regular glass ionomers; however, both ionomer types are weaker than amalgam and composite cores (8). A study done by Jayanthi et al. (9) found a lower compressive strength for glass ionomer than amalgam and resin composite restorations. Several studies have demonstrated a lower bonding strength of glass ionomer to the ceramic restoration and core material as compared to resin composite (11, 14). Therefore, the glass ionomer has not been recommended as an alternative to the composite core build-up material (9).

Hydraulic calcium silicate cement (HCSC), including mineral trioxide aggregate (MTA), is highly biocompatible and non-cytotoxic material. Calcium silicate cement maintains pulp vitality and stimulates the formation of reparative dentine (4). It is used frequently in reparative pulp procedures and hard tissue repairs, such as pulp capping, pulpotomy, apexogenesis, apexification, perforation repair, and root-end filling (15). Biodentine is a second-generation hydraulic calcium silicate material used as a dentine replacement material. It is developed to overcome the disadvantages of mineral trioxide aggregate (MTA), such as low compressive strength and long setting time (16, 17).

Biodentine is commonly used in posterior tooth restoration, particularly when the pulp situation needs to be monitored. Besides its role in postoperative pain reduction, it exhibits minimum rough surfaces and good marginal integrity which eventually yields in clinically sound restoration (18). Bioden-

tine is also considered an efficient dentine substitute material, especially when it is covered by the resin composite restoration (18). Cantekin et al. (19) compared the shear bond strength (SBS) of MTA and Biodentine to a methacrylate-based composite, and found a higher SBS of Biodentine over MTA material. To the best of our knowledge, no study has yet assessed the bonding strength of hydraulic calcium silicate cement (Biodentine) to e.max ceramic restoration. Therefore, the present study aimed to evaluate the SBS of hydraulic calcium silicate cement (Biodentine) as a core material to the e.max ceramic discs using Variolink N (IvoclarVivadent, Schaan, Liechtenstein, Germany) as a luting agent.

MATERIALS AND METHODS

Specimen preparation

The current study was registered and approved by the Ethical Committee of Research Center (CDRC), College of Dentistry, King Saud University, Riyadh, Saudi Arabia (CDRC No. IR 0257). Three core materials were used in this study: hydraulic calcium silicate cement (Biodentine™, Septodont, Saint-Maur-des-Fossés, Creteil, France), resin composite (Filtek™Z250 XT, 3M ESPE, St. Paul, MN, USA), and resin-modified glass ionomer (RMGIC) (GC Corporation, Tokyo, Japan) (Table 1).

Forty discs (6 mm diameter and 2 mm thickness) were fabricated from each core material using a metallic mold. All core material discs were embedded in self-curing acrylic resin (Vertex Orthoplast, Vertex-Dental B.V. Asia Ptd. Ltd., Singapore) using a polyvinyl chloride tube.

For the control group, 40 extracted human permanent molar teeth were cleansed of gross debris and stored in distilled water. The teeth were mounted in self-curing acrylic resin (Vertex Orthoplast, Vertex-Dental B.V. Asia Ptd Ltd, Singapore). Dentine surfaces were exposed using 320-, 400-, and 600-gritsilicon carbide abrasive paper under water lubrication. All specimens were ultrasonically cleaned (Branson CPX1800H Ultrasonic Cleaner/Branson Inc., USA) in distilled water for 15 minutes.

In total, 160 IPS e.max discs (4 mm diameter; 2 mm thickness) were made by the lost wax technique and the IPS e.max Press ingot (IPS e.max Press, IvoclarVivadent, Schaan, Liechtenstein, Germany).

Bonding procedure

The IPS e.max discs were cemented to the core specimens using Variolink N (IvoclarVivadent, Schaan, Liechtenstein, Germany) luting agent (Fig. 1). The ceramic discs were treated with 5% hydrofluoric acid for 20 seconds (s), then thoroughly rinsed with water spray and dried with air. Primer agent (Monobond N) was applied with a micro-brush to ceramic discs for 60 s and subsequently dispersed with a strong stream of air. All core surfaces were etched with N-Etch (37% phosphoric acid) for 15 s, then the surfaces were cleaned with vigorous water spray for five s and dried. The adhesive agent (Excite F DSC, Ivoclar Vivadent) was applied and thinned with air. Cement (Variolink N, Ivoclar Vivadent) was hand-mixed following the manufacturer's instructions and applied for both core specimens and ceramic discs. After that, ceramic discs were placed on the core speci-

TABLE 1. Materials used in this study

Material	Brand name	Manufacture	Composition	Batch No./Lot No.
Tricalcium-silicate cement	Biodentine®	Biodentine™, Septodont, Saint-Maur-des-Fossés, Creteil, France.	Powder: Tricalcium silicate, dicalcium silicate, calcium carbonate and oxide, iron oxide, and zirconium oxide. Liquid: Calcium chloride and hydrosoluble polymer.	B20459
Resin composite	Filtek™Z250 XT	Filtek™Z250 XT, 3M ESPE, St. Paul, MN, USA	Filler System: Surface-modified zirconia/silica with a median particle size of approximately 3 microns or less. Non-agglomerated/non-aggregated 20-nanometer surface-modified silica particles. Resin System: Bisphenol A-glycidyl methacrylate, urethane dimethacrylate, ethoxylated bisphenol A glycol dimethacrylate, Polyethylene glycol dimethacrylate and triethylene glycol dimethacrylate.	N773306
Resin modified glass ionomer	GC Fuji II LC	GC Corporation, Tokyo, Japan	Powder: Fluoro-alumino-silicate glass. Liquid: Polyacrylic acid, hydroxyethyl methacrylate (HEMA) dimethacrylate, camphorquinone, water.	170209A
Resin-based dental luting material	Variolink N	IvoclarVivadent Schaan, Liechtenstein, Germany	Monomer matrix: Bisphenol A-glycidyl methacrylate, urethane dimethacrylate, and triethylene glycol dimethacrylate. Inorganic fillers: Barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass, and spheroid mixed oxide. Additional contents: initiators, stabilizers, and pigments.	W11420

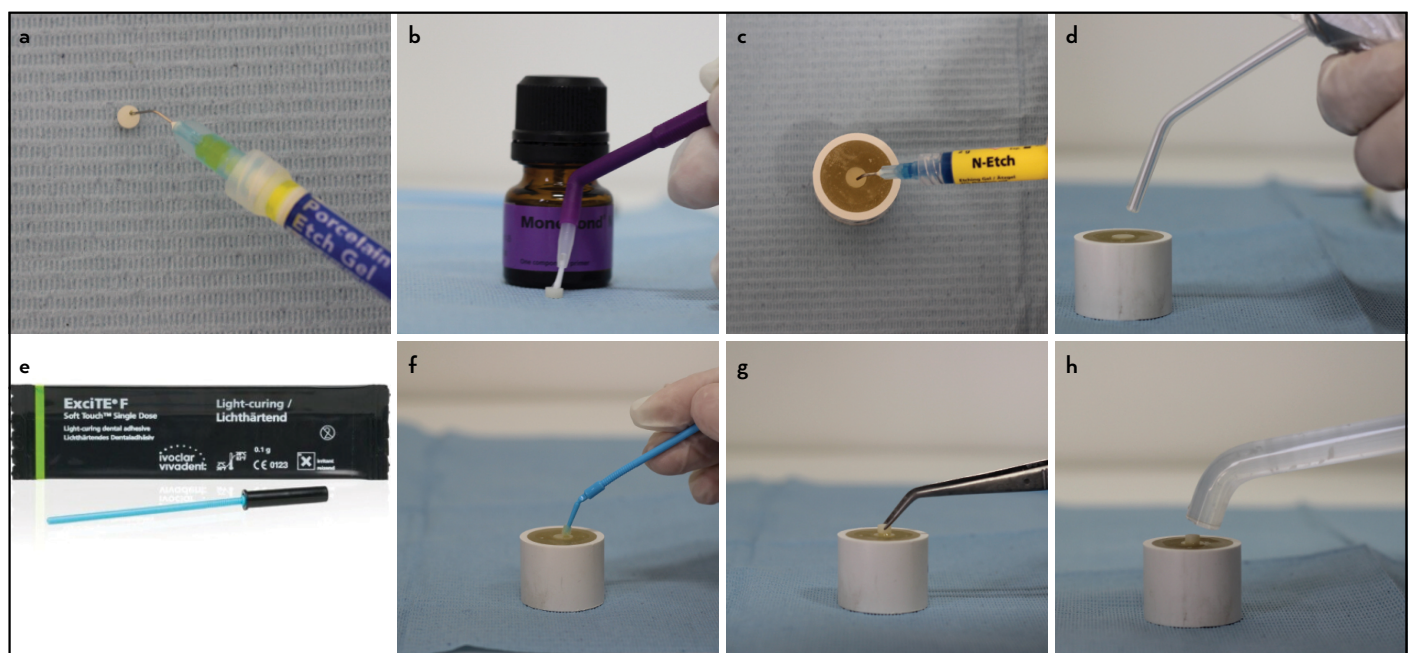


Figure 1. Bonding procedure steps. (a) Ceramic disc treatment with hydrofluoric acid. (b) Primer application. (c) Core surface etching with N-Etch. (d) Drying with air. (e) ExcITE F DSC (Adhesive agent). (f) Adhesive application. (g) Ceramic disc placement. (h) Photopolymerization

mens with light figure pressure, and the excess cement was removed with an explorer. Photopolymerization was performed with Elipar™ DeepCure-S LED Curing Light unite (3M™ ESP, Saint Paul, Minnesota, United State) at 1470 mW/cm² for 20 s.

All the bonded specimens were stored in distilled water at 37°C for 24 h before thermocycling. They were then thermocycled (Thermocycler, SD Mechatronik) between 5°C and 55°C, with a dwell time of 30 second in each bath and a transfer time of 12 second between baths, for 1.500 cycles.

Shear bond strength test

The SBS was tested using a universal testing machine (Instron 5965, Instron Corporation) with a 5 KN load cell at a crosshead speed of 0.05 mm/min (Fig. 2). Each specimen was continu-

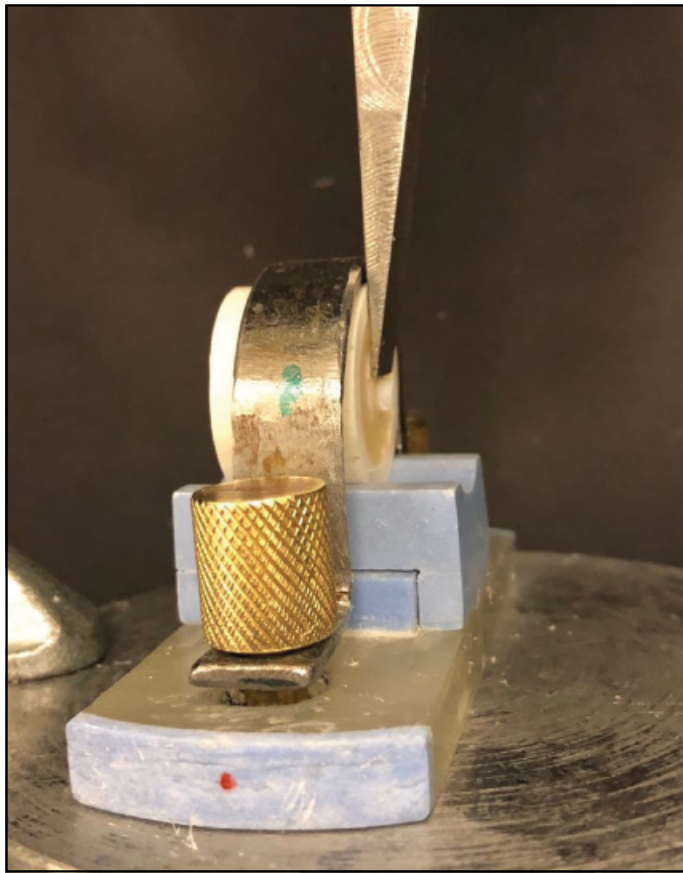


Figure 2. Shear bond strength test using a universal testing machine

ously loaded until fracture occurred. The SBS values were calculated by dividing the force at which bond failure occurred by the bonding area, and then expressed in MPa (Compressive Stress at Maximum Load).

Evaluation of fracture mode

The mode of fracture after SBS was determined by stereomicroscope (Nikon SMZ1000, Japan) at ×20 magnification by a single examiner. Failure modes were categorized as: (1) adhesive fracture, which was a failure at the interface of ceramic and resin cement/core material and resin cement, (2) cohesive fracture, which was a failure within the core materials/ceramic discs, and (3) mixed fracture, which was a combination of adhesive fracture and cohesive fracture.

Statistical analysis

The mean SBS was analyzed with statistical software (IBM SPSS Statistics 20, IBM Crop, Armonk, NY). A one-way analysis of variance (ANOVA) was used to analyze the data for significant differences. Tukey's multiple pair-wise comparison test was used to compare the mean values among the 6 pairs of 4 groups. A P-value of <0.05 was considered statistically significant.

RESULTS

The mean SBS values and standard deviations of the four study groups. Biodentine, resin composite, RMGIC, and human dentine are presented in Table 2. The highest SBS values were observed for the composite group (36.17±6.08 MPa), while the lowest values were observed for the Biodentine group (21.86±3.18MPa). One-way ANOVA showed highly statistically significant differences for the mean values of SBS among the groups (P<0.001) (Table 2). The resin composite group exhibited significantly higher SBS than all tested groups (P<0.001). The SBS of Biodentine and RMGIC groups were significantly inferior to the resin composite and human dentine groups. (P<0.001; mean±SD; 21.86±3.18MPa, 23.75±4.31MPa for Biodentine and RMGIC group respectively). However, no statically significant differences in the SBS was observed between the Biodentine and RMGIC group (P=0.176).

Table 3 shows the distribution of the failure modes among the groups. The most frequently experienced failure type was the mixed failure (62.5-80%), observed in the human dentine, RMGIC, and resin composite groups. However, Biodentine group showed predominantly cohesive failure mode (67.5%) (Fig. 3).

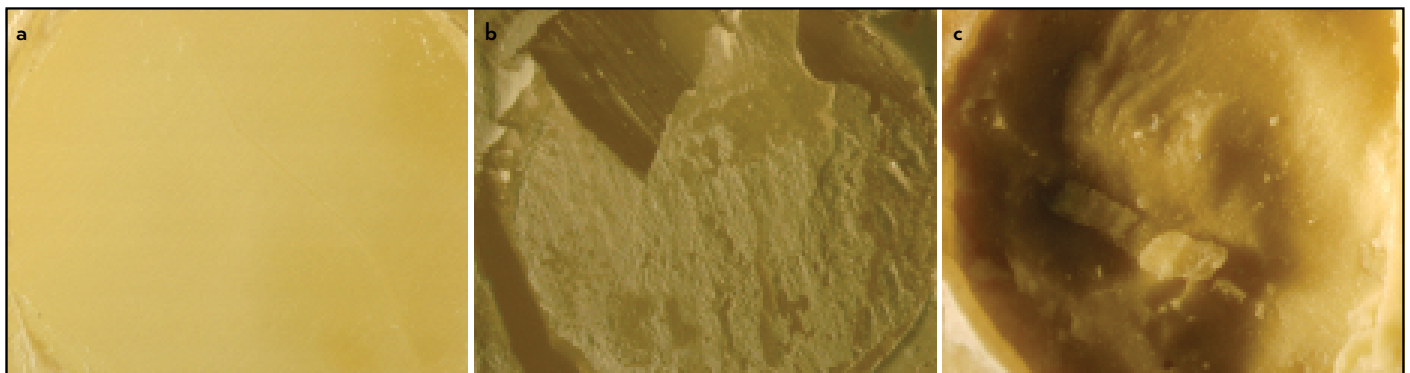


Figure 3. Stereomicroscopic images representing modes of failure. (a) Adhesive. (b) Mixed. (c) Cohesive

TABLE 2. Mean shear bond strengths (MPa), standard deviations and One-way ANOVA for tested groups

	Group	Mean MPa	SD	P-value	95% Confidence interval		Multiple comparison test			
					Lower Bound	Upper Bound	Biodentine	Composite	RMGIC	Human dentine
Mean shear bond strengths (MPa)	Biodentine	21.86	3.18	0.001	20.558	23.154	1			
	Composite	36.17	6.08		34.870	37.466	<0.001*	1		
	RMGIC	23.75	4.31		22.453	25.049	0.176	<0.001*	1	
	Human dentine	27.59	1.84		26.291	28.888	<0.001*	<0.001*	<0.001*	1

*The mean difference is significant at the 0.05 level. SD: Standard deviation

TABLE 3. Failure mode distributions for the groups

Groups	Adhesive	Mixed	Cohesive
Biodentine	0	13	27
Composite	13	25	2
RMGIC	7	31	2
Human dentine	8	32	0

DISCUSSION

The choice of a core material depends on its physical, biological, and handling properties. The core build-up materials should maintain pulp vitality in cases of teeth with vital pulp. Hydraulic calcium silicate cement (Biodentine) core build-up material, protects pulp vitality by the formation of reparative dentine (4). The core material must also withstand the mastication and parafunction forces over many years (20, 21). The current evidence indicates that the majority of indirect restoration failures occur due to shear stresses (7). A limited number of studies evaluated the SBS of Biodentine to dentine or other direct restorative materials (22-26). However, the bonding strength of Biodentine to ceramic restorations does not gain attention in the literature.

The results of the present study showed a high statistically significant differences in the SBS of Biodentine, resin composite, RMGIC, and human dentine to the e.max ceramic restoration. The resin composite material had the highest SBS to the e.max restoration, whereas Biodentine had the lowest SBS. These results are in agreement with previous reports that illustrated the effect of the differences in the types of core materials on the bonding strength of luting cement (10, 11).

The resin composite core material formed the strongest bond to the e.max ceramic discs with resin-luting cement (Variolink N). This result can be explained by the potential effect of combining two materials with a similar composition (resin composite and Variolink N). The blend of these materials will construct a robust bond through their mechanical retention and chemical adhesion features (14). The results of the resin composite group in the current study are similar to the findings of Bozogullari et al. (11) which measured the bond strength of 50 ceramic discs to different core materials using luting resin cement. The results of their study showed that resin-based core material had the highest SBS when compared to glass ionomer-based and ceramic-based core materials. In the same study, the bonding failure between ceramic discs and resin-based core material was mostly cohesive; however, in the

present study, the failure was mainly mixed. The reason for this difference might be a variation in the experimental design, as thermal cycling was not utilized in the previous study (11).

In the present study, the dentine group had the second highest SBS, at 27 ± 1.8 MPa, next to the resin composite group. Lower values have been reported in other studies (5.5 ± 2.1 MPa, 8.8 ± 1.8 MPa); (27, 28) however, a limited comparison can be made with these earlier studies, since the results of bonding strength to the dentin are influenced by a large number of variables (27). Among these variables, the nature of the dentinal surface and the testing device could affect the score values of dentinal bonding strength (29-31). The dentinal depth has also an influence on the SBS to the luting agent. Several studies have shown that the bonding strength obtained in superficial dentine was significantly higher than in deep dentine (30, 31). However, in the present study, the depth of the dentine was not considered as a variable during the dentine surface preparation. The failure mode of the dentine group in the current study was a predominantly mixed failure. On the contrary, Altintas et al. (27) showed mainly cohesive failure mode within the dentine group. This difference could be attributed to variations in the cross-head speed of the testing machine used in their study.

The RMGIC group showed a lower SBS than resin composite and dentine with a mixed mode of failure. Similar to the findings of Hewlett et al. (14), in which the SBS of RMGIC core material to resin cement was inferior to the resin composite. The failure mode for RMGIC in their study was adhesive. This finding could be explained by the lack of adequate strength in the RMGIC material as compared to the resin composite (14).

In the present study, the Biodentine group showed the lowest SBS among all the study groups. This finding agrees with the results of Subash et al. (32), who evaluated the fracture resistance of endodontically treated teeth restored with Biodentine as a core material. The Biodentine group in the former study had a significantly lower fracture resistance when compared with RMGIC and composite.

Deepa et al. (23) measured the SBS Biodentine and a resin composite to different liners, confirming a lower bonding strength for the Biodentine group. This finding could be explained by the low early strength of the Biodentine, as it is a porous material during the initial setting (23, 33). Likewise, another study showed better bonding strength between the resin composite and Biodentine when the self-etch adhesive

system was used (26). In the present study, an etch-and-rinse adhesive system was used (Variolink N) to prepare the surfaces of the core materials. The extra rinsing step in the etch-and-rinse adhesive system step could affect adversely the bond strength of Biodentine material. Under stereomicroscope analysis, the Biodentine group mainly showed cohesive mode of failure. This type of failure could be referred to as either the low tensile strength of Biodentine or the nature of shear bond test in which the stresses generated at the interface between two materials are predominantly tensile (14). Accordingly, the bond between Biodentine and resin cement may be stronger than the tensile strength of the Biodentine. Therefore, further investigation of the tensile strength of Biodentine material is needed.

The differences in the SBS values between the current study and previous reports could be interpreted as results of variations in the experimental set-up or procedures, including microstructure of the teeth, tooth storage conditions, temperature, the static load applied during cementation of ceramic discs, the use of thermocycling, and the type of universal testing machine with different cross-head speeds. The aging protocol used in the present study consisted of a thermocycling treatment of 1.500 cycles. This cycle number was higher than the recommended number according to ISO, which is 500 cycles at 5–55°C with a dwell time of 30 seconds. The applied cycles in the present study simulate the dwell time of the core materials within the oral cavity which can predict the long-term durability of the tested core material (34). In addition, different cross-head speeds could influence the SBS and the fracture pattern in the dentine substrate (35).

The design of the current study is limited to an *in vitro* model and doesn't mimic the clinical condition. The majority of SBS studies lack standardization in the method of conduction which makes the comparison between the results impractical. Therefore, the results of this study should be applied to the clinical situation with caution. The final assessment of the core materials should be based on long-term clinical studies. The finding of this study suggests that Biodentine has relatively weak bonding strength to e.max ceramic restoration as compared to RMGIC and resin composite. However, when the Biodentine is indicated to be used as core material for pulp preservation, it's advocated to be covered with a layer of resin composite material to enhance the bonding strength of the core material to e.max ceramic restoration.

CONCLUSION

Within the limitations of the present study, the following conclusions can be drawn:

1. The type of core material affects the SBS to an e.max ceramic restoration using resin-luting cement.
2. The hydraulic calcium silicate cement (Biodentine) core had the lowest SBS value for IPS e.max discs cemented with resin cement, while the resin composite core had the highest SBS.
3. The Biodentine material could have low tensile strength, as it showed predominantly cohesive failure by stereomi-

croscope after the SBS test. Further studies are needed to measure the tensile strength of Biodentine.

Disclosures

Conflict of interest: The authors declare that they have no conflict of interest.

Ethics Committee Approval: The current study was registered and approved by the Ethical Committee of Research Center (CDRC), College of Dentistry, King Saud University, Riyadh, Saudi Arabia (CDRC No. IR 0257).

Peer-review: Externally peer-reviewed.

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