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Shear Bond Strength of Four Resin Cements Used to Lute Ceramic Core Material to Human Dentin

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Abstract

Purpose: This study evaluated the effect of four resin cements on the shear bond strength of a ceramic core material to dentin.
Materials and Methods: One hundred twenty molar teeth were embedded in a self-curing acrylic resin. The occlusal third of the crowns were sectioned under water cooling. All specimens were randomly divided into four groups of 30 teeth each according to the resin cement used. One hundred twenty cylindrical-shaped, 2.7-mm wide, 3-mm high ceramic core materials were heat-pressed. The core cylinders were then luted with one of the four resin systems to dentin (Super-Bond C&B, Chemiace II, Variolink II, and Panavia F). Half of the specimens (n = 15) were tested after 24 hours; the other half (n = 15) were stored in distilled water at 37°C for 1 day and then thermocycled 1000 times between 5°C and 55°C prior to testing. Shear bond strength of each specimen was measured using a universal testing machine at a crosshead speed of 1 mm/min. The bond strength values were calculated in MPa, and the results were statistically analyzed using a two-way analysis of variance (ANOVA) and Tukey HSD tests.
Results: The shear bond strength varied significantly depending on the resin cement used ($p < 0.05$). The differences in the bond strengths after thermocycling were not remarkable as compared with the corresponding prethermal cycling groups ($p > 0.05$). Significant interactions were present between resin cement and thermocycling ($p < 0.05$). After 24 hours, the specimens luted with Variolink II (5.3 ± 2.2 MPa) showed the highest shear bond strength, whereas the specimens luted with Chemiace II (1.6 ± 0.4 MPa) showed the lowest. After thermocycling, the bond strength values of specimens luted with Chemiace II (1.1 ± 0.1 MPa) and Super-Bond C&B (1.7 ± 0.4 MPa) decreased; however, this was not statistically significant ($p > 0.05$). The increase in the shear bond strength values in the Panavia F (4.5 ± 0.7 MPa) and Variolink II (5.5 ± 2.1 MPa) groups after thermocycling was also not statistically significant ($p > 0.05$).
Conclusion: Variolink II and Panavia F systems showed higher shear bond strength values than Chemiace II and Super-Bond C&B. They can be recommended for luting ceramic cores to dentin surfaces.

In prosthodontic practice, the task of restoring endodontically treated teeth is encountered almost daily. Leempoel et al[1] evaluated a large sample of teeth with single crown restorations and found that 39% were nonvital and had received some type of dowel restoration. Many abutment teeth planned for fixed prosthodontic treatment require dowel-and-core build-ups because of extensive structural defects resulting from decay, trauma, or previous restoration. In most situations, severely compromised teeth are permanently restored with complete-coverage crowns to restore function and esthetics. The amount of the remaining tooth structure dictates the type of core build-up that can be used in pulpless teeth. When there is a minimal loss of structure, dowels and cores are not necessary.[2] When a horizontal loss of the clinical crown has occurred, and a small ferrule can be created in the remaining tooth structure,[3,4] there are few alternatives to restoration with a dowel-and-core build-up.[5]

The choice of an appropriate restoration for endodontically treated teeth is guided by strength and esthetics. The cast gold dowel and core has been regarded as the “gold standard” in dowel-and-core restorations due to its superior success rate.[6,7] Alternatives to cast dowels and cores have been developed. The use of prefabricated dowels and custom-made build-ups with amalgam or composite simplifies the restorative procedure, because all steps can be completed chairside, and fair clinical success can be expected.[8-10] New tooth-colored dowels have improved the esthetics of teeth restored with dowels and cores.[11-13] In addition, zirconia ceramic offers superior strength compared with other dowel materials.[11,14,15] The use of composite as a core material has also enhanced the ability to reproduce the shade and translucency of natural teeth. The restoration of teeth with adhesively cemented internal restorations offers improved mechanical stability over cemented restorations.[16] As an alternative to composite cores bonded to zirconia dowels,[17] a new technique allows the addition of a heat-pressed ceramic core to achieve the tooth-colored indirect dowels and cores.[18]

An ideal luting agent should provide an efficient bond between tooth and core, increase the resistance to fracture, and reinforce the remaining tooth structure. Resin cements are the first choice to achieve such conditions.[19] The composition of resin cements and their polymerization forms may influence their properties.[20,21] The different viscosities of resin cements[22] and monomer composition[23] have resulted in differences in the adhesive properties of resin cements. The polymerization method of the resin cement influences the bonding obtained to dental substrate.[24] Resin cements can be divided into three groups: chemically cured, photoactivated, and "dual-cure" materials. Chemically activated cements have a short working time, but their use is not limited by porcelain thickness. Photoactivated cements have ideal working characteristics, but to prevent incomplete polymerization, they should not be used if the restoration is thicker than 3 mm. Dual-cure cements have in their composition both a photoinitiator (camphoroquinone) and the chemical activation components (peroxide/amine) to achieve the best working and setting characteristics. Several studies have evaluated the bonding effectiveness of these resin cements to dental hard tissues[25-28] (Table 1).

The purpose of this study was to evaluate the effect of four resin cements on the shear bond strength of a ceramic core material to dentin. The first hypothesis tested was that the shear bond strength differs with the compositions of the resin cements, and the second hypothesis tested was that thermocycling affects the shear bond values of the resin cements.

Materials and methods

Four resin cements (Table 2) were tested for their bond strength to ceramic and dentin. Dentin specimens were prepared and embedded in an autopolymerizing acrylic resin. Ceramic discs were fabricated, and one of the four resin cements was applied to the dentin surface in accordance with the manufacturers' instructions. Ceramic core-resin cement-dentin specimens were tested to failure in shear after 24 hours or thermocycling after fabrication.

One hundred twenty molar teeth, free of dental caries or restoration, were cleaned and stored in saline at room temperature within 1 month after extraction. The occlusal third of the crowns were sectioned with a slow-speed diamond saw sectioning machine (Isomet; Buehler Ltd., Lake Bluff, IL) under water cooling, and the crowns were embedded in an autopolymerizing acrylic resin (Meliodent; Bayer Dental Ltd., Newbury, UK). Dentin surfaces were polished with 600- and 800-grit waterproof polishing papers for 30 seconds. All specimens were randomly divided into four groups of 15 teeth each according to the resin cement used.

One hundred twenty cylindrical-shaped, 2.7-mm wide, 3-mm high wax patterns were prepared, sprued, and invested (IPS Empress Investment; Ivoclar, Schaan, Liechtenstein) for the IPS Empress technique. After the burnout and preheating process, the core material (Empress-Cosmo; Ivoclar, Lot no: D64021) was pressed using the IPS Empress technique at 900°C and 5 bars.[29] The core cylinders were divested, and all surfaces were carefully airborne-particle abraded (Miniblaster; Belle de St. Claire, Encino, CA) at a pressure of 80 psi with 50-μm particles. The tip of the microetcher was kept 1 mm away from the surface of each specimen and was applied for 3 seconds.

Before cementation, excess water was removed with a gentle blow of compressed air, and then, the core cylinders were luted with one of the four resin cements to dentin.

For the Super-Bond C&B group, the green activator was applied to the dentin surfaces of the teeth for 5 seconds. One drop of catalyst was mixed with four drops of monomer (Super-Bond C&B; Sun Medical Co., Ltd, Moriyama City, Japan) to wet the bonding surfaces. The mixture was combined with two scoops of powder and applied to the bonding surface. The ceramic core cylinders were seated on the dentin surface with light finger pressure,[30] and excess cement was removed with an explorer.

Table 1 Summary of research on shear bond strength of resin cements to dentin surfaces

Study	Bond effectiveness of resin cements to human hard tissues
Braga et al[20]	Compared Porcelite, Dual, and C&B luting composite Dual-polymerizing resin cement bonded better than autopolymerizing resins.
Stewart et al[25]	Compared Nexus, Panavia 21, Rely X ARC, and Calibra Autopolymerizing and light-polymerizing resin cement bonded better than dual-polymerizing resins
Piwowarczyk et al[26]	Compared Perma Cem, Rely X ARC, Panavia F, Variolink II, Nexus 2, and Calibra Dual-polymerizing resin bonded better than autopolymerizing resins
Hikita et al[27]	Compared Nexus 2, Panavia F, Rely X Unicem, and Variolink II Equal bond strength if correct adhesive applied
Irie et al[28]	Compared Bistite II, Chemiace II, Compolute, Xeno Cem, Perma Cem, Fuji Cem, and Fuji Plus After 24-hour storage, shear bond strength increased

Table 2 Compositions, manufacturers, lot numbers, and mixing information of resin cements studied

Resin cement	Composition	Manufacturer	Lot number	Mixing information
Super-Bond C&B	4-META, MMA-TBB	Sun Medical, Moriyama City, Japan	LL 2	Green activator for 5 seconds, air dry. Mix liquid and powder (1:2). Apply mix.
Chemiace II	4-META, TEGDMA	Sun Medical, Moriyama City, Japan	GF 1	Green activator for 5 seconds, air dry. Mix liquid and powder (1:1). Apply mix, light-polymerize for 40 seconds.
Variolink II	Bis GMA, UDMA, TEGDMA	Vivadent, Ivoclar, Schaan, Liechtenstein	E16059 E13253	Apply Total Etch (37% phosphoric acid) only on enamel for 15 seconds, rinse, air dry, apply Syntac primer for 15 seconds, air dry, apply Syntac adhesive for 10 seconds, air dry, apply Heliobond. Apply Monobond-S for 60 seconds, air dry, apply Heliobond. Mix base and catalyst paste, light-polymerize for 40 seconds.
Panavia F	Bis GMA, MDP	Kuraray, Osaka, Japan	41150	Apply Primer ED for 5 seconds, air dry gently after 60 seconds, mix one drop each of Cleafil SE Primer, Porcelain Bond Activator for 5 seconds, apply mix universal and catalyst paste, light-polymerize for 40 seconds, apply oxyguard.

Bis GMA = bisphenol A glycerolate dimethacrylate; MMA = methyl metahacrylate; TEGDMA = triethyleneglykol-dimetacrylate; UDMA = diurethane dimethacrylate; MDP = 10-methacryloyloxydecyldihydrogen-phosphate.

For the Chemiace II group, the green activator was applied to the dentin surfaces of the teeth for 5 seconds. A surface modifier (Porcelain Liner M; Sun Medical Co., Ltd) was applied to the ceramic core surface. One drop of liquid was mixed with one scoop of powder. The crowns were cemented to the teeth, as previously described. Photo-polymerization was performed with the light-polymerizing unit (Hilux 550; Express Dental Products, Toronto, Canada) at 550 mW/cm^2 (with the light tip to specimen distance of 0 mm, 90° apart) for 40 seconds.

For the Variolink II group, the ceramic core cylinders were treated with fluoridic acid (Ceramic Etchant; Ceramco, Burlington, NJ) for 1 minute and neutralized (Ceramic Etchant Neutralizer; Ceramco) in accordance with the manufacturer's instructions. Silane (Monobond-S; Ivoclar) was first applied with a brush to the ceramic core disks for 60 seconds, and then a bonding agent (Heliobond; Ivoclar) was applied. After the dentin was etched, a primer (Syntac Primer; Ivoclar) was applied to the dentin surface for 15 seconds, an adhesive (Syntac Adhesive; Ivoclar) for 10 seconds, and then the bonding agent (Heliobond) with a brush. The cement (Variolink II, Vivadent, and Ivoclar), comprising a combination of 25% Variolink yellow base, 25% Variolink white base, and 50% catalyst, was hand-mixed following the manufacturer's directions and applied to both the dentin surface and the ceramic core cylinder. The cementation procedure and photo-polymerization were performed as previously described.

For the Panavia F group, the ceramic core cylinders were etched with phosphoric acid gel (K Etchant; Kuraray Co., Ltd., Osaka, Japan) for 5 seconds. A layer of silane–coupling agent combination (Clearfil Porcelain Bond Activator and Clearfil SE; Kuraray Co., Ltd.) was applied to the ceramic bonding surfaces for 5 seconds and then air-dried. Panavia F ED, the self-etching primer, was applied to the dentin surface for 60 seconds and gently air-dried. Panavia F was mixed for 20 seconds and applied to both the dentin surface and the bonding surface of the ceramic core disk. The cementation procedure and photo-polymerization were performed as previously described.

All specimens were stored in distilled water at 37°C for 1 day, and then, half of the specimens (n = 15) were tested; the other half (n = 15) were thermocycled 1000 times (Nova Inc., Konya, Turkey) between 5°C and 55°C (20 seconds dwell time) prior to testing. The specimens were perpendicularly engaged at their bases with a custom probe (Ultradent Products Inc., South Jordan, UT) in a universal testing machine (Testometric 500; Lancashire, UK) at a crosshead speed of 1.0 mm/min until bonding failure occurred (Fig 1).

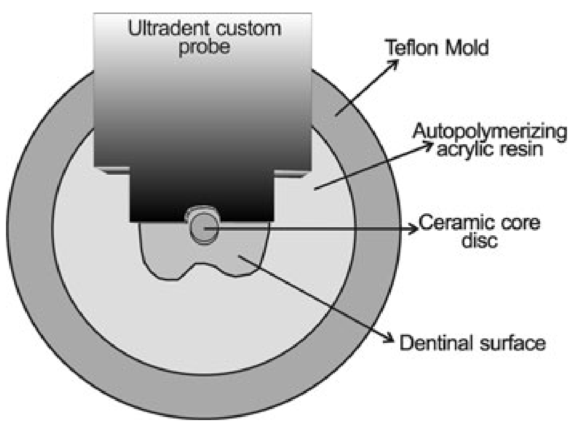

Figure 1 Shear bond test configuration.

Fracture analysis

After the specimen was tested and removed from the testing apparatus, the fracture sites were observed using a stereomicroscope (SZTP; Olympus, Tokyo, Japan) at 22× magnification to identify the mode of failure. The fractured surface was classified according to one of the four types: (1) adhesive failure between the resin cement and dentin; (2) adhesive failure between the resin cement and ceramic core; (3) cohesive failure in the resin cement; and (4) cohesive failure in the dentin.

Scanning electron microscopy (SEM) examination

A tooth from each cementation group was prepared for SEM analysis. After storing for 24 hours at 37°C, the teeth were sectioned buccolingually through the restoration. To observe the interface, the specimens were first polished with 240-, 400-, and 600-grit silicon carbide abrasive papers. The bonding interface was etched with 35% phosphoric acid for 10 seconds and then washed and gently air-dried for 3 seconds. The specimens were sputter-coated with gold, and the interfaces were observed under the SEM (435 VP; Leo SEM Products, Cambridge, UK).

Statistical analysis

The ultimate stress (MPa) of the ceramic-resin cement-dentin bonds was calculated as follows[31]:

$$\text{Stress} = \frac{\text{Failure load (N)}}{\text{Surface area } (\pi \times r^2)\ (\text{mm}^2)}$$

The shear bond strength values were analyzed with statistical software (SPSS PC, version 10.0; SPSS, Chicago, IL). Two-way analysis of variance (ANOVA) was used to analyze the data for significant differences. Tukey HSD tests were used to perform multiple comparisons among cements at a significance level set at $p < 0.05$.

Results

Two-way ANOVA indicated that the shear bond test values varied according to the resin cement used (Chemiace II, Super-Bond C&B, Variolink II, and Panavia F; $p < 0.05$), but did not vary according to the time of testing (24 hours and after thermocycling; $p > 0.05$; Table 3). The means and standard deviations of the groups are presented in Table 4.

After 24 hours, the specimens luted with Variolink II showed the highest shear bond strength in all resin cements, whereas the specimens luted with Chemiace II showed the lowest. There were no statistically significant differences between the groups bonded with Panavia F and Super-Bond C&B, or between those bonded with Variolink II and Panavia F ($p > 0.05$).

After thermocycling, the bond strength values of the specimens luted with Chemiace II and Super-Bond C&B decreased; however, this was not statistically significant ($p > 0.05$). The increase in the shear bond strength values of Panavia F and Variolink II groups after thermocycling was also not statistically significant ($p > 0.05$).

Table 3 Two-way ANOVA

	df	MS	F	*p*
Within group materials (resin cement)	3	101.1	69.1	0.000
Within testing time	1	2.6	1.8	0.184
Interaction effect (Resin cement ∗ time of testing)	3	4.5	3.1	0.03

Fracture analysis (Table 5)

In the Variolink II group, for specimens tested after 24 hours, most failures (8 of 15) were cohesive in nature within the cement (type 3). Four specimens showed cohesive failure within the dentin (type 4), and only three specimens showed adhesive failure at the bonding resin/ceramic core interface (type 2).

In the Panavia F group, 10 specimens showed cohesive failure within the cement (type 3), and two specimens showed cohesive failure within the dentin (type 4). Three specimens showed adhesive failure at the bonding resin/ceramic core interface (type 2).

In the Super-Bond C&B group, none of the specimens showed cohesive failure within the dentin. Ten specimens showed adhesive failure at the bonding resin/ceramic core interface (type 2), and five specimens showed cohesive failure within the cement (type 3).

In the Chemiace II group, eight specimens showed adhesive failure at the cement/ceramic core interface (type 2), two specimens showed adhesive failure at the cement/dentin interface (type 1), and five specimens showed cohesive failure within the cement (type 3).

Scanning electron microscopy

In the Chemiace II group, as seen in Figure 2, the hybrid layer showed discontinuity in the dentin-resin interface. In the Panavia F and Super-Bond C&B groups, long resin tags can be seen, and the Variolink II group exhibited longer resin tags and an organized hybrid layer.

Discussion

The results obtained support the first hypothesis that shear bond strength values differ with different resin cements. This result is in accordance with the results of Braga et al,[20,21] who concluded

Table 4 Shear bond strength values after 24 hours and after thermocycling (MPa) (mean ± standard deviation)

	After 24 hours	After thermocycling
Chemiace II	1.6 ± 0.4[ab]	1.1 ± 0.1[a]
Super-Bond C&B	3.0 ± 1.0[bc]	1.7 ± 0.4[ab]
Panavia F	4.0 ± 0.8[cd]	4.5 ± 0.7[de]
Variolink II	5.4 ± 2.3[de]	5.5 ± 2.1[e]

Groups with the same letters are not statistically significantly different.

Table 5 Comparison of failures at 24 hours and after thermocycling (n/n) for each failure mode

Cement type	Cohesive: in dentin	Adhesive: dentin/cement	Cohesive: in cement	Adhesive: cement/ceramic	Cohesive: in ceramic
Chemiace II	0/0	2/2	5/10	8/3	0/0
Super-Bond C&B	0/0	0/2	5/8	10/5	0/0
Panavia F	2/0	0/2	10/7	3/6	0/0
Variolink II	4/1	0/4	8/5	3/5	0/0
Total	6/1	2/10	28/30	24/19	0/0

that the composition of resin cements and their polymerization forms may influence their properties and bond strengths. The results obtained did not support the second hypothesis that thermocycling affects the shear bond values of resin cements. Piwowarczyk et al[32] concluded that after 14 days of water storage followed by thermocycling, Panavia F and Variolink II exhibited strong bond strengths to specific prosthodontic materials; however, in the current study, the increase in the bond strength values was not statistically significant. This may be due to the storage time before thermocycling.

The high value for the standard deviation might be explained by the nature of the dentin surfaces used for bonding and the testing devices. In the current study, after a flat surface was obtained, the depth prepared into the dentin was not taken into consideration. Pashley et al[33] verified that dentin depth plays an important role in bond strength. A second explanation for the high standard deviation might be the way the materials were dispensed. According to the manufacturers' recommendations, equal amounts of both base and catalyst pastes were dispensed, relying only on visual reference. Considering the constant load applied on the ceramic cylinder, and that the photoactivation (when required) was made to simulate a clinical situation, the proportioning of pastes seems to be critical for the speed of reaction. One may infer that early strength may be compromised, as is clearly demonstrated in the chemically activated material.

Pecora et al[34] used two testing devices (Ultradent testing device and unrestricted tapered knife edge) to evaluate the shear bond strength of three single-bottle adhesives with their multi-step counterparts. Higher standard deviations were observed for five of the six adhesives tested with the Ultradent testing device compared with the knife testing device. Pecora et al concluded that the reason for a higher standard deviation could be, among other things, the conceivable emergence of a torsion moment. This could mean that the area of the Ultradent testing device

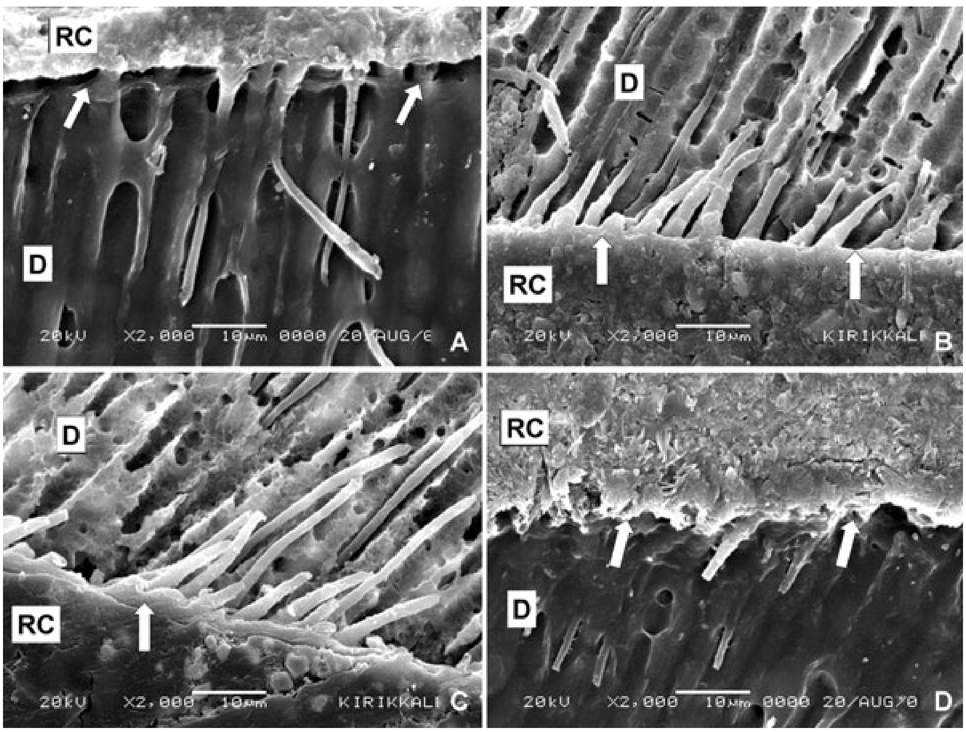

Figure 2 SEM view of the interface of the specimen luted with (A) Chemiace II, (B) Panavia F, (C) Super-Bond C&B, (D) Variolink II. RC = resin cement; D = dentin. In A, the arrows indicate the gap between dentin and resin cement. In B, C, and D, the arrows indicate the hybrid layer.

surrounding the specimen is of almost the same size as the specimen itself. Thus, the perfect adjustment of the specimens on the Ultradent testing device is very difficult.[34]

The present results can be compared with those obtained by previous authors only to a limited extent as the in vitro framework means that a large number of different variables may influence the results of the study, meaning that these are not reproducible.[35] Eick et al[36] claimed that bond strengths to dentin and enamel should be higher than 20 MPa to adequately compensate for the stress caused by polymerization shrinkage. Whether this absolute threshold is of any practical relevance is a question that cannot be answered here. It is a fact, however, that the bond strengths with dentin found in the present study were all well below 20 MPa.

The present study also addressed the question of failure modes. The failures were predominantly cohesive in the resin cement in the Variolink II and Panavia F groups. The adhesive failures occurred between the ceramic core and resin cement in the Chemiace II and Super-Bond C&B groups. No cohesive failures in dentin were observed in the Chemiace II and Super-Bond C&B groups, probably because the bond strengths obtained with different materials were generally lower than the cohesive strength of dentin.[37] Conceivably, the bond strength values may be accountable for the modes of failure at the bonded interface.[38] This study has given rise to the tentative conclusion that higher bond strength values increase cohesive failure rates.

This in vitro study allowed an immediate and after-1000 thermocycles assessment of the bond created between the resin cement and ceramic core material; however, in vitro tests cannot adequately simulate the clinical conditions in every detail. The results of the in vitro tests should be applied to the clinical situation with caution. It is admissible, however, to compare the measured in vitro results obtained under identical conditions.

The current study has shown that the composition of resin luting cements and different surface treatments on both dentin and ceramic influences the bond strength of the specific ceramic core material. It cannot be assumed that different core materials used in restorative dentistry or different resin luting cements will show the same pattern of variability as restorative materials with different compositions. This highlights the care that must be employed when using the standard test methods in comparing dental materials and suggests the need for studies similar to that reported.

Conclusion

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

1 The shear bond test values varied according to the resin cement used (Chemiace II, Super-Bond C&B, Variolink II, and Panavia F; $p < 0.05$), but did not vary according to the time of testing (24 hours and after thermocycling; $p > 0.05$).
2 Variolink II and Panavia F showed the highest and Chemiace II showed the lowest bond strength values ($p < 0.05$).

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