THE BONDING EFFECTIVENESS OF FIVE LUTING RESIN CEMENTS TO THE IPS EMPRESS 2 ALL CERAMIC SYSTEM

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Keywords: Shear bond strength, ceramic, resin cements.

SUMMARY.

Background: Variolink II is the only resin cement used for bonding IPS (Ivoclar Porcelain System) Empress 2 ceramic restorations. Alternative luting resin cements need to be investigated for their bonding effectiveness with the IPS Empress 2 ceramic. **Objectives**: To determine the shear bond strength (SBS) and the effect of thermocycling, on the bonding effectiveness, of five resin cements to IPS Empress 2 ceramic. **Materials and methods**: The projecting surfaces of one hundred ceramic discs were ground wet on silicone carbide paper. The specimens were divided into 5 groups of 20. The resin cements were bonded to the prepared ceramic surfaces, in the form of a stub. The specimens were stored under distilled water at 37°C in an oven for 24 hours. Ten specimens in each group were thermocycled for 300 cycles between 5°C and 55°C. All the specimens were stressed to failure in an Instron Materials Testing Machine. **Results**: The results were subjected to a one-way analysis of variance (ANOVA). Statistically similar mean SBS values were grouped using the Bonferroni (Dunn) multiple comparison test. The means for the non-thermocycled group were: 26.21, 19.41, 17.69, 17.43, and 15.76. The means for the thermocycled group were: 22.90, 15.72, 14.34, 13.96 and 13.45. The differences between the means were highly significant ($p < 0.0125$). **Conclusions**: The shear bond strength of Variolink II and Rely X ARC to IPS Empress 2 ceramic was effective. Thermocycling had a significant effect on the mean SBS values of Calibra. Thermocycling had no significant effect on the mean SBS values of the other resin cements.

INTRODUCTION

The use of luting resin cements to bond ceramic restorations to tooth structure is accepted by the profession[1]. The establishment of a durable and reliable bond between a dental ceramic and a luting resin cement is extremely important to ensure the success of a resin-bonded restoration[2] since the main cause of failure is at the resin-ceramic interface[3]. Luting resin cements have therefore become an indispensable link in adhesive restorative techniques[4,5]. The increase in range of applications especially with ceramic restorations, have been accompanied by an increase in the number of luting resin cements available for adhesive bonding of ceramic restorations[6].

Ceramic restorations fabricated from the IPS Empress 2 all ceramic system are cemented with the luting resin Variolink II[7], as advocated by the manufacturer. Alternative luting resin cements need to be investigated to determine the effectiveness of their bond strengths to the IPS Empress 2 ceramic, as well as to what degree temperature variations would affect these bonds. The objectives of this *in vitro* study was:

1. To determine and compare the shear bond strengths of five luting resin cements to the IPS Empress 2 all ceramic specimens.
2. To determine the influence of temperature variations (thermocycling) on these bonds.

MATERIALS AND METHODS

One hundred IPS Empress 2 ceramic discs (Ivoclar, Schaan, Liechtenstein) (10 mm in diameter and 7,5 mm high) were mounted in Bencor stainless steel rings (Bencor Multi-Testing Device, Danville Engineering, San Roman) using Excel self curing acrylic resin (Wright Health Group LTD, Manchester, United Kingdom). The protruding surfaces of the ceramic discs were ground flat (Fig. 1) and parallel

to the shear force direction on an Imptech grinder/polishing machine (IMP Innovative Products cc, Boksburg, South Africa), using wet 80 grit silicone carbide waterproof grinding paper. The latter procedure was then repeated, using wet 180 grit silicone carbide waterproof grinding paper. The ceramic discs were at random divided into 5 groups of 20 each. Stubs (4,62 mm in diameter and 1,5 mm high) of the luting resins, Variolink II (Vivadent, Schaan, Liechstenstein, Batch number: 558523), Calibra (Dentsply, Chaulk International Inc., Milford, U. S. A., Batch number: 507063), Opal (3M, ESPE, St. Paul, U. S. A., Batch number: 7555), Rely X Veneer (3M, ESPE, St. Paul, U. S. A., Batch number: 8714A3), and Rely X ARC (3M, ESPE, St. Paul, U. S. A., Batch number: 3415A3) were prepared using a rubber mould and bonded to the ceramic surfaces (Fig. 2) as follows:

Variolink II

The surfaces of the ground ceramic discs **(20)** were etched for 20 seconds with 4,9 % buffered IPS Ceramic etching gel (Ivoclar, Schaan, Liechtenstein), rinsed for 15 seconds, dried for 10 seconds with compressed air, silanated for 60 seconds with Monobond-S (Vivadent, Schaan, Liechtenstein), dried with compressed air for 15 seconds, wet with Heliobond bonding agent (Vivadent, Schaan, Liechtenstein) for 5 seconds, dried with compressed air for 10 seconds and then light cured for 30 seconds. Variolink II resin stubs were bonded to the surface-treated ceramic specimen by mixing equal quantities of the catalyst and base pastes which were then syringed into the rubber mould that was clamped firmly to the ceramic surface and then light cured for 60 seconds.

Calibra

The surfaces of the ground ceramic discs **(20)** were etched for 60 seconds with a 9,5 % buffered Universal Porcelain etching gel (Ultradent Products, Inc., South Jordan, Utah), rinsed for 15 seconds, dried for 10 seconds with compressed air, silanated for 30 seconds with Dentsply silane coupling agent (Dentsply/ Caulk International Inc., Milford, U. S. A.), dried with compressed air for 15 seconds, wet

with Prime & Bond *NT* (Dentsply /Caulk International Inc., Milford, U. S. A.) bonding agent for 5 seconds, dried with compressed air for 10 seconds and light cured for 30 seconds. Calibra resin stubs were bonded to the surface-treated ceramic specimens by mixing equal quantities of the catalyst and base pastes which were then syringed into the rubber mould that was clamped firmly to the ceramic surface and then light cured for 60 seconds.

Opal, Rely X Veneer and Rely X ARC

The surfaces of the ground ceramic discs **(60)** were etched for 60 seconds with a 9,5 % buffered Universal Porcelain etching gel (Ultradent Products, Inc., South Jordan, Utah), rinsed for 15 seconds, dried for 10 seconds with compressed air, silanated for 60 seconds with 3M ceramic primer (3M ESPE, St. Paul, U.S.A.), dried with compressed air for 15 seconds, wet with Scotchbond 1 (3M ESPE, St. Paul, U.S.A.) bonding agent for 5 seconds, dried with compressed air for 10 seconds and light cured for 30 seconds. Opal or Rely X Veneer or Rely X ARC resin stubs were bonded to the surface-treated ceramic specimens by mixing equal quantities of the catalyst and base pastes which were then syringed into the rubber mould that was clamped firmly to the ceramic surface and then light cured for 60 seconds.

All the specimens were stored under distilled water in a MEMMERT oven (LASEC Laboratory & Scientific Equipment Co (PTY) LTD, Cape Town, South Africa) at 37^{o}C for 24 hours. Ten specimens of each group, for each luting resin, were then thermocycled for 300 cycles between 5^{o}C and 55^{o}C with a dwell time of 30 seconds. The shear bond strengths of all the specimens were then evaluated in an Instron Materials Testing Machine (Instron Limited, Coronation Road, High Wycombe, Bucko, HP 12 35Y), operating in the shear mode at a crosshead speed of 0,5 mm/min. The shear force was applied at right angles to the long axis of the luting resin stub (Fig. 3). Shear bond strengths were recorded in Mega Pascals (MPa). The fractured surfaces were visually analysed with a Nikon light

microscope (Nikon Corp, Tokyo, Japan) at original magnification X 6 to determine whether the fractures were cohesive within the resin, cohesive within the ceramic or adhesive failures at the resin-ceramic interface. The type of shear bond fractures for the different resin luting cement specimens were described as follows:

Adhesive: fracture at the resin ceramic-interface.

Cohesive: fracture in resin or ceramic only.

STATISTICAL ANALYSIS

The shear bond strengths were subjected to a one-way analysis of variance test (ANOVA) to determine whether significant differences existed between the SBS values of the non-thermocycled and the thermocycled groups. Statistically similar mean SBS values were grouped by applying the Bonferroni (Dunn) multiple comparison test ($p= 0.0125$). Pair-wise comparisons of the mean SBS values for the resins in each group were also done to determine if any significant differences existed between the mean SBS values of the resins that were not thermocycled and those that were thermocycled.

RESULTS

The mean SBS values (Table I) for the non-thermocycled group were 26.21, 19.41, 17.69, 17.43 and 15.76. The mean SBS values (Table I) for the thermocycled group were 22.90, 15.72, 14.34, 13.96 and 13.45. The differences between the means for the non-thermocycled group and the thermocycled group were highly significant ($p< 0.0125$). Statistically similar mean SBS values are indicated in Table II and Table III. Mean values with the same letter are not significantly different.

Non-thermocycled group:

Shear Bond Strength:

There were no statistically significant differences between the mean SBS values of Variolink II and Calibra (p= 0, 0255). However, there were statistically significant differences between the mean SBS value of Variolink II and the mean SBS values of the other three resins (p < 0,0125). A pair-wise comparison of the mean SBS values of Calibra, Opal, Rely X Veneer, and Rely X ARC demonstrated no statistically significant differences (p > 0,0125). Variolink II demonstrated the highest mean shear bond strength value (26,21: SD± 6,9) and Rely X ARC, the lowest mean SBS value (15,76: SD± 4,64).

Fractures:

Variolink II demonstrated 10 cohesive fractures in resin. Calibra also demonstrated 10 cohesive fractures in resin. Opal demonstrated 9 cohesive fractures in resin and 1 cohesive fracture in ceramic. Rely X Veneer demonstrated 9 cohesive fractures in resin and 1 cohesive fracture in ceramic. Rely X ARC demonstrated 6 cohesive fractures in resin and 4 adhesive fractures.

Thermocycled group:

Shear Bond Strength:

There were no statistically significant differences between the mean SBS values of Variolink II and Rely X ARC (p = 0,0244). However, there were statistically significant differences between the mean SBS value of Variolink II and the mean SBS values of the other 3 resins (p < 0,0125). A pair-wise comparison of the mean SBS values of Calibra, Opal, Rely X Veneer and Rely X ARC demonstrated no statistically significant differences (p > 0,0125). Variolink II demonstrated the highest mean SBS value (22,90: SD± 8,04) and Calibra, the lowest mean SBS value (13,45: SD± 2,49).

Fractures:

Variolink II demonstrated 9 cohesive fractures in resin and 1 cohesive fracture in ceramic. Calibra demonstrated 6 adhesive fractures and 4 cohesive fractures in ceramic. Opal demonstrated 9 adhesive fractures and 1 cohesive fracture in ceramic. Rely X Veneer demonstrated 9 cohesive fractures in resin and 1 cohesive fracture in ceramic. Rely X ARC demonstrated 7 adhesive fractures and 3 cohesive fractures in ceramic.

A comparison of the mean SBS values of both groups revealed that the mean SBS value of Calibra in the non-thermocycled group was significantly higher than the mean SBS value of the Calibra in the thermocycled group ($p = 0,0085$). The mean SBS values of the other resins in the non-thermocycled group were not significantly higher than the mean SBS values of the resins in the thermocycled group ($p > 0,0125$).

DISCUSSION

The aim of a ceramic dental restoration is to produce a long lasting bond between the restoration and tooth structure[8]. This bond depends on proper preparation techniques and the use of an appropriate luting resin cement[6]. The shear bond strength of resin cements to feldspathic porcelain have been studied by Kato[9] in 2001. The latter study recorded pre-thermocycled shear bond strength values of 17,0 MPa-30,3 MPa and post-thermocycled shear bond strength values of 0 MPa-32,9 MPa. The luting resins bonded to feldspathic porcelain[9] produced greater mean shear bond strength values compared to the shear bond strength values in this study. The post-thermocycled groups of luting resin cements in this experiment however, showed no detachment prior to shear testing compared to two of the luting resin cement groups that were bonded to the feldspathic porcelain[9].

Notable shear bond strengths (post-thermocycling) that are considered acceptable, to prevent fracture between an etched ceramic surface and a luting resin cement is 15,66 MPa[10]. Variolink II and Rely X ARC demonstrated a mean shear bond strength greater than 15,66 MPa for the non-thermocycled as well as the thermocycled groups. Thermocycling had a significant effect on the mean SBS values of Calibra but no significant effect on the mean SBS values of the other resin cements. The mean SBS results therefore indicate that after thermocycling, the bond strength of Variolink II and Rely X ARC to IPS Empress 2 ceramic was effective.

Shear bond fractures that are cohesive in either resin or porcelain are indicative of a successful and strong bond between the resin and the restoration[2]. Thermocycling can have a significant effect on the quality of these bonds at the resin-ceramic interface[11,12]. The resin cements that were not thermocycled demonstrated fewer adhesive fractures compared to the resin cements that were thermocycled, indicating that even though thermocycling did not have a significant effect on the mean SBS values of these resins, it had a significant effect on the quality of the bond at the resin-ceramic interface and the type of shear bond fracture pattern.

Although Rely X ARC demonstrated an effective mean SBS value, the number of adhesive fractures demonstrated after thermocycling, suggest that Rely X ARC did not follow the shear bond fracture pattern indicative of strong bonds as mentioned previously. Further investigations are required to determine the qualitative failure of Rely X ARC at the resin-ceramic interface.

In the thermocycled group Variolink II and Rely X Veneer demonstrated the same number of cohesive fractures in resin and ceramic indicating successful and strong bonds between resin and ceramic.. Rely X Veneer however, had a lower mean SBS value when compared to Variolink II and Rely X ARC that was thermocycled.

Further investigations are required to determine the quantitative failure of Rely X Veneer at the resin-ceramic interface.

Variolink demonstrated the best mean SBS value after thermocycling as well as the best type of SBS fractures. The reasons for this may be due to the properties of the etchant or silanating agent or bonding agent or a combination of all three components. Further studies are required to confirm this hypothesis.

CONCLUSIONS

The mean shear bond strength of Variolink II and Rely X ARC to IPS Empress 2 ceramic was effective.
Thermocycling had a significant effect on the mean SBS values of Calibra.
Thermocycling had no significant effect on the mean SBS values of the other resin cements. Thermocyling had a significant effect on the quality of shear bond fractures at the resin-ceramic interface for all the resins except Variolink II.
The luting resin cement preferred for use with IPS Empress 2 ceramic is Variolink II only.

Recommendations:

1. Rely X ARC can be used to bond restorations to IPS Empress 2 ceramic.

2. Longitudinal studies are needed to determine the clinical success of this luting resin cement with the IPS Empress 2 all ceramic system.

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CPD questions: *SADJ*: 2003

1. The luting resin cement preferred for use with IPS Empress 2 ceramic is Variolink II only.

A True

B False

2. The minimum shear bond strength that is considered acceptable between an etched ceramic surface and a luting resin cement is 15, 66 MPa.

A True

B False

3. Thermocycling did not have an adverse effect on any of the specimens.

A True

B False

4. Cohesive fractures indicate a bond strength that is of poor quality.

A True

B False

5. The use of Rely X ARC luting resin is recommended for use with the IPS Empress 2 ceramic.

A True

B False

Answers: 1. B, 2. A, 3. B, 4. B, 5. A.

A. Which cement was not used in this study:

1. Rely X ARC
2. Rely X Veneer
3. Opal
4. Protec-cem

B. Which mean SBS value was significantly affected by thermocycling:

1. Calibra
2. Opal
3. Variolink
4. Rely X ARC

C. A cohesive fracture occurs in:

1. Resin only
2. Ceramic only
3. Resin or Ceramic
4. Resin-Ceramic interface

D. An adhesive fracture occurs in:

1. Resin only
2. Ceramic only
3. Resin or ceramic
4. Resin-ceramic interface

E. IPS Empress 2 ceramic is reinforced with:

1. Feldspar
2. Quartz

3. Leucite
4. Glass

Answers: A. 4, B. 1, C. 3, D. 4, E. 3.

Legends: The bonding effectiveness of five luting resin cements to the IPS Empress 2 all ceramic system.

Table I : Mean shear bond strength (SBS) values (MPa) of non-thermocycled and Thermocycled test specimens.

Table II : Statistical analysis of the mean shear bond strength (SBS) values (MPa) of non-thermocycled specimens.

Table III : Statistical analysis of the mean shear bond strength (SBS) values (MPa) of thermocycled specimens.

Fig. 1 : Surface view of disk mounted in steel ring.

Fig. 2 : Resin stub bonded to ceramic disk: surface view.

Fig. 3 : Application of shear force at right angle to the long axis of resin stub.

Figures

Figure. 1: Surface view of disc mounted in steel ring.

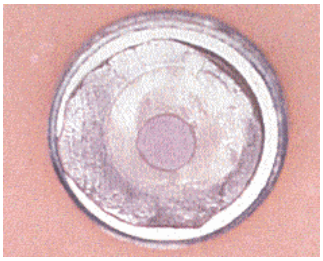

Figure. 2: Resin stub bonded to ceramic disc: surface view.

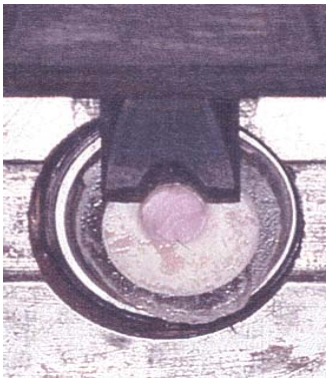

Figure. 3: Application of shear force at right angles to long axis of resin stub.

Table I: Mean shear bond strength (SBS) values (MPa) of non-thermocycled and thermocycled test specimens.

Luting resins	Mean SBS	
	Non-thermocycled (SD)	Thermocycled (SD)
Variolink II	26,21 (6,90)	22,90 (8,04)
Calibra	19,41 (5,51)	13,45 (2,49)
Opal	17,43 (6,13)	14,34 (4,44)
Rely X Veneer	17,69 (5,48)	13,96 (3,74)
Rely X ARC	15,76 (4,64)	15,72 (3,86)

(SD) = Standard Deviation

Table II: Statistical analysis of the mean shear bond strength (SBS) values (MPa) of non-thermocycled specimens.

Bonferroni (Dunn) Grouping		Mean SBS	SD	Number	Resin Cement
	A	26,21	6,90	10	Variolink II
B	A	19,41	5,51	10	Calibra
B		17,43	6,13	10	Opal
B		17,69	5,49	10	Rely X Veneer
B		15,76	4,64	10	Rely X ARC

Mean SBS values with the same letter are not significantly different.

Table III: Statistical analysis of the mean shear bond strength (SBS) values (MPa) of thermocycled specimens.

Bonferroni (Dunn) Grouping		Mean SBS	SD	Number	Resin Cement
	A	22,90	8,04	10	Variolink II
B		13,45	2,49	10	Calibra
B		14,34	4,44	10	Opal
B		13,96	3,74	10	Rely X Veneer
B	A	15,72	3,86	10	Rely X ARC

Mean SBS values with the same letter are not significantly different.