Bond Strength of Different Self-Adhesive Resin Cements to Zirconia

Thesis

Master of Science in Oral Biology (MSOB) degree program

By

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Dedication

Jai Shree Krishna:: Jai Swaminarayan:: Om Namah Shivay:: Shree Ganeshay Namah

I want to dedicate this manuscript to my father, late Dr. Raju J Patel, my mother Dr. Neeta R Patel, my late grandparents, my in laws Mukesh and Hemangini Patel, Anand and Nilam Patel, my brother Ronak and my husband Sneh Patel. My family is my source of support, strength and motivation and I would not be here without them.

I also want to dedicate this to my little ones Anaaya, Anaaika and Nandini and show them that you can achieve anything if you put your mind to it.

Acknowledgement

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Abstract

Objectives: The purpose of the study was to compare the shear bond strength of five self-adhesive resin cements used to bond zirconia.

Methods: Seventy-two KATANA Zirconia STML (n=12) specimens were sectioned and sintered in an induction furnace (CEREC SpeedFire, Dentsply Sirona, Germany). Specimen surfaces were ground finished with 800 grit silicon carbide abrasive with cooling water and cleaned with ultrasonication in alcohol. Specimens were air-particle abraded with 50 µm aluminum oxide at 2.8 bar pressure. Cylindrical composite resin specimens (2.1 mm in diameter, 3 mm in height) were bonded to the zirconia samples with self-adhesive resin cements Panavia SA Universal (PSA), TheraCem (TCM), SpeedCem 2.0 (SCM), RelyX Unicem 2 (RCM), PermaCem 2.0 (PCM) and dual-cure multi-step composite resin cement Panavia V5 (control, PV5, after application of ceramic primer) following manufacturers' instructions. A load of 1000 g was applied to the composite cylinders during bonding in an alignment apparatus, then light cured for 80 s. Samples were stored in distilled water at 37° C for 24 h, then subjected to 10,000 thermal cycles. Shear bond strength was determined using a universal testing machine at a crosshead speed of 0.5 mm/min expressed in MPa. The fractured surfaces of specimens were inspected with a stereo microscope and classified as

adhesive, cohesive, or mixed failures. One-way ANOVA test and Tukey test were applied for statistical analysis.

Results: Shear bond strength values [MPa] were for PSA 8.64 (1.78 SD), TCM 9.05 (2.64 SD), SCM 8.54 (1.92 SD), RCM 7.60 (1.26 SD), PMC 7.59 (1.10 SD) and PV5 9.59 (1.55 SD). One-way ANOVA test revealed no statistically significant differences in shear bond strength between the resin cements tested ($p>0.05$). Pair wise comparison using Tukey test revealed that the shear bond strength using RCM was statistically lower than that using PV5 ($p<0.05$).

Conclusions:

According to the results of this in vitro study, self-adhesive resin cements can achieve shear bond strengths to zirconia that are comparable multi-step resin cements. In routine clinical application, self-adhesive resin cements can be a user-friendly, less technique sensitive alternative for bonding Zirconia restorations following the APC concept.

Literature review and Introduction:

Zirconia

An increasing number of all ceramic restorations are being currently used in clinical practice. High strength ceramics like yttrium stabilized zirconia offer a wide variety of clinical applications including anterior and posterior single crowns, FDPs, implant supported restorations, etc. This is due to their excellent physical properties as well as improved optical properties.[1,2]

"High-strength dental ceramics" include the following non-glass-based ceramic systems: crystalline-based systems with glass fillers (e.g., glass- infiltrated alumina) and polycrystalline solids (e.g., alumina and zirconia).[4]

Zirconia has a monoclinic crystal structure at room temperature and a tetragonal and cubic structure at increasing temperatures. Formulations used in dentistry contain mainly tetragonal crystals that are partially stabilized with yttrium oxide (Y_2O_3) and have a flexural strength of 900 to 1,400 MPa, a modulus of elasticity of 210 GPa, and a fracture toughness of 10 MPa/m0.5. Pure zirconia exhibits a polymorphic phase transformation from a cubic to a tetragonal to a monoclinic phase accompanied by a high-volume change when cooling down from high temperatures after sintering which makes the sintered body unstable. However, the

addition of 3–6 wt% Y_2O_3 stabilizes the zirconia in the tetragonal phase which is otherwise not stable at room temperature. Zirconia with Y_2O_3 added in concentrations less than those required to stabilize it completely is called yttrium-oxide–partially-stabilized zirconia (YPSZ) when the sintered ceramic contains a mixture of cubic and tetragonal phases. YPSZ has optimized physical properties and exhibits much more fracture toughness and fracture strength than alumina ceramic.[1,4]

Properties termed active crack resistance or transformation toughening[23] are unique to this material: external stresses and cracks cause transformation of the tetragonal particle into a monoclinic one with greater volume (approximately 3% to 5%), subjecting a crack under compressive stresses and impeding its growth. However, the actual effects of this phase transformation on ultimate strength and its role in an accelerated aging process (low- temperature surface degradation) are discussed controversially.

Since the late eighties, YPSZ has been used in orthopedics for total hip replacement. In dentistry, YPSZ has been successfully tested in animals as a material for oral implants. Recently, YPSZ posts for restoring endodontically treated teeth have been introduced and YPSZ abutments for dental implants are currently being developed.[1]

The use of zirconia in restorative dentistry has grown exponentially over the past decade. Early zirconia formulations were used for frameworks because of their high flexural strength and unesthetic opacity in porcelain-fused-to-zirconia restorations. Through processing refinements, materials with increased translucency were introduced so that veneering with feldspathic porcelain was not required and the material could be used in monolithic form.[1,2,4]

Restorations are typically milled from green-stage or pre-sintered (white-stage) zirconia blocks before full sintering. Only very few CAD/CAM systems mill from fully sintered blocks, which have a significantly higher hardness and flexural strength, making the milling process time-consuming and taxing on the milling equipment. First-generation conventional zirconia copings and frameworks are veneered with feldspathic ceramic (porcelain-fused-to-zirconia, PFZ) for esthetic reasons since they are rather opaque and monochromatic white. Early studies indicate a high incidence of veneer fractures and chippings.[45,46] The development of veneering ceramics that better matched the thermal (coefficient of thermal expansion [CTE]) and physical properties of zirconia as well as firing and cooling protocols to control internal thermal stresses significantly increased reliability of PFZ restorations.[18] More recent investigations show long-term success rates of PFZ crowns that are not different from metal- ceramics.[18,47] Nevertheless, concerns about possible veneering ceramic fractures made monolithic full-contour

restorations the predominant all-ceramic choice. A fully digital CAD/CAM process has made full-contour zirconia (FCZ) restoration fabrication highly predictable and cost-effective. Second-generation zirconia materials have a higher translucency and slightly lower flexural strength than conventional zirconia. A customized, tooth-like appearance is created through infiltration of liquid dyes in a green or pre-sintered stage and firing of stains and glazes after sintering. Some manufacturers offer pre-shaded and even multilayer zirconia blanks that mimic natural tooth appearance and can be further customized.[4]

The latest generation of zirconia features significantly greater light transmission with optical properties suitable even for anterior teeth. The higher translucency is achieved by slight changes of the Y_2O_3 content (5 mol-% or more instead of 3 mol-%), resulting in a higher amount of cubic-phase particles.[48,49] However, the flexural strength (between 550 and 800 MPa) is significantly lower than that of conventional zirconia but still considerably higher than any silica-based ceramic. Some clinicians have begun using FCZ for resin-bonded partial-coverage inlays/onlays and laminate veneers.[50]

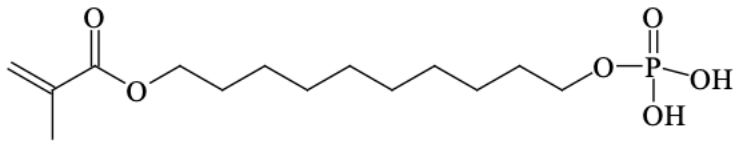
In the literature, historically, there was limited data regarding bonding methods to zirconia ceramic. Hydrofluoric etching and silanization, which enhances the resin bond to conventional silica-based ceramics, does not improve the resin bond strength to alumina or zirconia ceramics. However, it has been shown that silica coating followed by silanization can be successfully used for bonding glass-infiltrated alumina ceramic. In the same study, phosphate monomer-containing resin composites were successfully bonded to sandblasted alumina ceramic, while using a conventional Bis-GMA resin composite a durable resin bond to alumina ceramic was not achieved.

With emerging long-term studies, evidence now suggests that air particle abrasion followed by application of 10-methacryloyloxydecyl dihydrogen phosphate (MDP) containing primer and composite resin cement help achieve a robust bond between zirconia and tooth structure.[9,5]

Composite resin cements and compatible bonding systems increase retention, improve marginal seal and fracture strength of all-ceramic restorations. While considered "cementable," some zirconia restorations benefit from insertion with composite resin-cement systems. These include zirconia restorations that are less strong, are thin, lack retention, or rely on resin bonding, such as resin-bonded fixed prostheses, high translucency or ultra-high translucency zirconia or bonded laminate

veneers.[3,9] The success of resin bonding relies on the proper materials selection and adequate treatment of tooth and restoration bonding surfaces. [13,14]

The gold standard for resin bonding to Zirconia includes the traditional multi-step process including priming and bonding agents for tooth as well as restorative material surfaces. This process is time-consuming and can be technique sensitive.[7,13,14,44]



Methacrylate monomer with phosphoric acid group (MDP)

Resin cements and resin ceramic bonding

Adhesive bonding techniques and modern all-ceramic systems offer a wide range of highly esthetic treatment options.[25,26,29,27,28] Bonding to traditional silica- based ceramics is a predictable procedure yielding durable results when certain guidelines are followed.[10,18] However, the composition and physical properties of high-strength ceramic materials, such as aluminum oxide-based (Al_2O_3)[30-34] and zirconium oxide-based (ZrO_2) ceramics,[35] differ substantially from silica-based

ceramics [31,36,37] and require alternative bonding techniques to achieve a strong, long-term, durable resin bond.

Resin-based composites are the material of choice for the adhesive luting of ceramic restorations[24]. Composite cements have compositions and characteristics similar to conventional restorative composites and consist of inorganic fillers embedded in an organic matrix (for example: Bis-GMA, TEGDMA, UDMA). Composite cements can be classified according to their initiation mode as auto-polymerizing (chemically activated), photoactivated, or dual-activated materials.

Photoactivated composites offer wide varieties of shades, consistencies, and compositions[24]. Clinical application is simplified through long handling times before and rapid hardening after exposure to light. Shade, thickness, and transmission coefficient of the bonded ceramic restoration and the composite itself influence the conversion rate of the photo-activated material and limit its application to thin silica-based ceramics.

Dual-activated composites offer extended working times and controlled polymerization,[24] although chemical activators ensure a high degree of polymerization. Most dual-activated resin cements still require photopolymerization and demonstrated inferior hard- ness when light polymerization was omitted.[38,39] Various dual-activated resin cements showed no

differences in resin-bond strengths between glass ceramics and enamel.[40] Auto-polymerizing resin cements have fixed setting times and are generally indicated for resin bonding metal-based or opaque, high-strength ceramic restorations[24].

Self-adhesive resin cements were developed based on the chemistry of resin cements and self-etch adhesives. They etch, prime, and bond to dentin without the need of separate agents for each of these steps. Therefore, the application is very simple, and the concept of the smear layer as a bonding substrate has been reintroduced with the expectation of a low incidence of postoperative sensitivity and pulp response. [41,42,43,44]

These cements provide good bond strengths with less steps making them more user friendly.[44] Their application can be accomplished in a single clinical step, similar to cementation procedures with conventional luting agents. Besides simplified application techniques, they seem less susceptible to moisture contamination. A low incidence of postoperative sensitivity is expected with these luting agents.[16]

Due to these properties and widespread applications, these cements have become popular in clinical use.

<u>Measuring bond strengths:</u>

The shear strength is the maximum stress that a material can withstand before failure in a shear mode of loading.

Bond strength tests are abundant in the dental literature in part because they are relatively easy to perform and are not equipment intensive. They, however, present several limitations that reduce their usefulness as a selection criterion in clinical practice. These drawbacks are briefly explained below.

Macro shear Bond Strength Tests [51]

In a macro shear bond strength test, a composite cylinder is built on the bonding substrate. After a pre- determined storage time, the specimen is positioned in a universal testing machine where a single-edged chisel, a flat-end rod, or a wire loop is attached to the actuator used to dislodge the composite cylinder from the substrate.

It is important to note that in shear tests, it is a tensile stress that actually causes debonding. In other words, the term shear test refers to the loading mode, and not to the stress causing interfacial failure. When the loading distance from the interface increases, tensile stress also increases due to creation of a bending moment in the composite cylinder.

The location and configuration of the loading device influences the stress distribution at the bonded interface and therefore affects the bond strength. Computer simulation using finite element analysis shows that, for a nominal stress of 15 MPa, the maximum tensile stress at the interface is 178 MPa when a chisel is used for loading. With the wire loop, the maximum tensile stress is 69 MPa. The higher the stress concentration at the load application area, the lower the bond strength. Therefore, the use of a knife-edge chisel results in lower bond strength values than the wire loop, where the load is distributed over a larger area. Typical dentin bond strength values with macro-shear tests are 10 to 50 MPa. Cohesive and mixed failures are very frequent and may affect up to 55% of the specimens.

Another aspect of interest is the elastic modulus of the composite used for the specimen cylinder. The larger the mismatch between the elastic modulus of the composite and the elastic modulus of the substrate, the higher the stress concentration at the interface. This lowers the measured bond strength.

When measuring shear bond strength values, stresses at the interface are not uniformly distributed— Bond strength is reported as the nominal stress value (in MPa), that is, the failure load (in Newtons) divided by the entire bonded area (in mm^2). This is often not accurate because the stress distribution at the interface is very heterogeneous. Debonding occurs due to stress concentration around a critical

size flaw, or void, at the interface that causes a crack to propagate. The actual stress level that initiates crack propagation can be several times higher than the nominal (or average) value. Therefore, nominal bond strength does not represent the failure stress.

Results of different studies are not comparable—Bond strength values for a specific material can vary a lot among studies due to differences in the bonding substrate, specimen preparation, storage conditions, and loading method. Unfortunately, there is very little standardization among research laboratories. Comparisons among different studies must be done very carefully.

Bond strength tests lack clinical significance—Based on what was described above, a threshold bond strength value that can be associated with a good clinical performance cannot be determined. Nevertheless, similar trends can be found in the literature for some adhesive systems. Systems that show poor performance in vitro generally have poor clinical performance.

Interfacial bond strength can be tested by a variety of methods. Using the dimensions of the bonded area, bond strength methods can be categorized as macro (4-28 mm^2) or micro (approximately 1 mm^2). The interface can be loaded either in tension or shear.

The ceramic-composite bond is susceptible to chemical,[53,54] thermal,[55] and mechanical[56] influences under intraoral conditions. The simulation of such influences in the laboratory is compulsory to draw conclusions on the long-term durability of a specific bonding procedure and to identify superior materials and techniques. Long-term water storage[57] and thermo- cycling of bonded specimens are accepted methods to simulate aging and to stress the bonding interface. Most studies that apply these methods reveal significant differences between early and late bond strength values.[58-62] Application of mechanical cyclic loading (fatigue load) causes significant reduction of bond strengths.[63,64]

Material selection and clinical recommendations on resin bonding to ceramics are based on mechanical lab- oratory tests that show great variability in materials and methods.[65,66] Preferred bond strength tests are the 3-point bending test, the tensile and micro-tensile test, and the shear and micro-shear test. Øilo[67] discussed the accuracy and clinical relevance of the different testing methods. The most common testing method is the shear bond test; however, some researchers prefer modified tensile tests to eliminate the occurrence of nonuniform interfacial stresses typical to conventional tensile and shear bond tests. Their specific fracture pattern may cause cohesive failure in the ceramic,[68] which may lead to erroneous interpretation of the actual data and taint an absolute ranking of the tested methods and materials.[65,69]

Research objective:

The purpose of this in vitro study was to compare the shear bond strength of five new self-adhesive resin cements used to bond zirconia.

Hypothesis:

H0: There is no difference in shear bond strength to zirconia between multi-step dual cure resin cement and self-adhesive resin cements.

HA: There is a difference in shear bond strength to zirconia between multi-step dual cure resin cement and self-adhesive resin cements.

Materials and Methods:

Square-shaped samples of unprocessed zirconium-oxide ceramic (KATANA Zirconia STML, Kuraray Noritake, Japan) with the dimensions 9 mm × 8 mm × 3 mm were fabricated and then sintered in an induction furnace (CEREC SpeedFire, Dentsply Sirona, Germany). N=12, Total N=72. Specimens were embedded in copper molds using PMMA (Polymethyl methacrylate) with one surface exposed for bonding. Specimens were ground finished with up to 800-grit silicon carbide abrasive under cooling water. Cylindrical composite resin specimens (2.1 mm diameter, 3 mm height) were fabricated using a standardized mold and packable composite resin material. Composite resin specimen surfaces were standardized using 800- grit silicon carbide abrasive. The zirconia specimens were cleaned with ultrasonication in alcohol for 3 minutes and air-particle abraded with 50 um aluminum oxide particles, 2.8 Bar pressure from a distance of 1 cm at 90-degree angle for 15 seconds.[9] Cylindrical composite resin specimens (2.1 mm diameter, 3 mm height) were bonded to the zirconia surfaces with five different self-adhesive composite resin cements (Panavia SA Universal; Kuraray Noritake, Speedcem Plus; Ivoclar and TheraCem; Bisco, PermaCem 2.0; DMG and RelyX Unicem 2; 3M. 12 specimens were used for each cement system and for the control group Dual cure resin cement (Panavia V5; Kuraray Noritake).

Figure 1

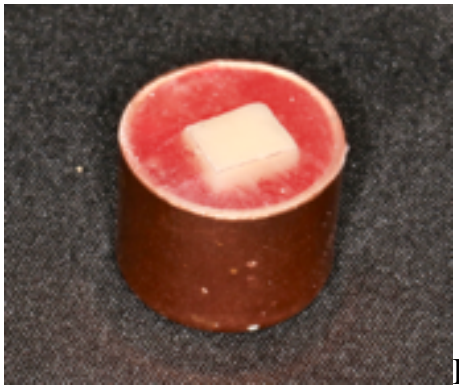


Figure 2

Figure 3

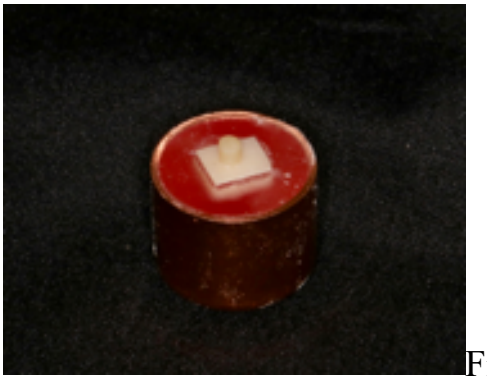


Figure 4

Control group:

<u>Panavia V5; Kuraray Noritake (PV5):</u>

Figure 5

Properties:

- Filler loading: 61 gew% (38 vol%)
- Flexural strength[1•2]: 127 MPa
- Flexural modulus[1•2]: 6,3 GPa
- Compressive strength[1]: 310 MPa
- Water sorption[1•2]: 21 µg/mm³
- Film thickness[1•2]: 12 µm
- Radiopacity[2]: 180% Al
- Fluoride releasing (28 days)[1]: 58 µg/g
- Working time: (23˚C) 2 min.
- Curing time: (light) 10 sec. ——————————

[1]Dual curing of the paste (combination of self- and light-curing)
[2]According to ISO 4049:2009. Source: Kuraray Noritake Dental Inc.

Contents:

Paste A/Paste B
• Bisphenol A diglycidylmethacrylate (Bis-GMA)
• Triethyleneglycol dimethacrylate (TEGDMA)
• Hydrophobic aromatic dimethacrylate
• Hydrophilic aliphatic dimethacrylate
• Initiators
• Accelerators
• Silanated barium glass filler
• Silanated fluoroalminosilicate glass filler
• Colloidal silica Bisphenol A
• diglycidylmethacrylate (Bis-GMA)
• Hydrophobic aromatic dimethacrylate
• Hydrophilic aliphatic dimethacrylate
• Silanated barium glass filler
• Silanated alminium oxide filler
• Accelerators
• dl-Camphorquinone
• Pigments
CLEARFIL™ Ceramic Primer Plus
• 3-Methacryloxypropyl trimethoxysilane
• 10-Methacryloxypropyl dihydrogen phosphate (MDP)

• Ethanol
PANAVIA™ V5 Tooth Primer
• 10-Methacryloyloxydecyl dihydrogen phosphate (MDP)
• 2-Hydroxyethyl methacrylate (HEMA)
• Hydrophilic aliphatic dimethacrylate
• Accelerators
• Water

The test groups are:

Panavia SA Universal; Kuraray Noritake (PSA)

Figure 6

Properties:

- F-ion release: Contains surface treated sodium fluoride (less than 1%)
- Storage temperature: 2 – 25°C / 36 – 77°F
- Filler loading: 62 wt % (40 vol%)
- Compressive strength[1]: 300 MPa
- Flexural strength[1•2]: 91 MPa
- Flexural modulus[1•2]: 4.6 GPa
- Water sorption[1•2]: 27 µg/mm3
- Film thickness: Approx. 14 µm
- Radiopacity[2] : 170 % Al
- Working time (23°C): 60 sec.

[1]Dual curing (combination of self- and light-cure)
[2]According to ISO 4049: 2009 Source: Kuraray Noritake Dental Inc.

Contents:

Bisphenol A diglycidylmethacrylate
Triethylene glycol dimethacrylate
2-hydroxyethyl methacrylate

sodium fluoride
Silanated barium glass filler
Silanated colloidal silica
Aluminum oxide filler
10-Methacryloyloxydecyl dihydrogen phosphate
Hydrophobic aromatic dimethacrylate
Silane coupling agent
dl-Camphorquinone
Peroxide
Accelerators
Catalysts
Pigments

TheraCem; Bisco (TCM)

Figure 7

Contents

Base
Ytterbium w/ Barium Glass
Portland Cement
Ytterbium Fluoride
Brombenzenesulfinic Acid, Sodium Dihydrate
BisGMA
Proprietary materials

Catalyst
10-Methacryloyloxydecyl Dihydrogen Phosphate
2-Hydroxyethyl Methacrylate

Tert-butyl Perbenzoate

Speedcem Plus; Ivoclar (SCM)

Figure 8

Properties

Mechanical and physical properties	Note	Specification	Unit
Flexural strength	1	≥ 60	MPa
Shear bond strength	2	≥ 6	MPa
Working time	at 23°C : at 37°C :	$120 \leq t_w \leq 180$ $80 \leq t_w \leq 140$	s
Setting time	at 23°C : at 37°C :	$180 \leq t_s \leq 300$ $120 \leq t_s \leq 200$	s
Solubility (7 days)	1	≤ 7.5	$\mu g/mm^3$
Water absorption (7 days)	1	≤ 40	$\mu g/mm^3$

Contents:

Base paste
Urethane dimethacrylate
Triethylene glycol dimethacrylate
Polyethylene glycol dimethacrylate

Catalyst paste
Ytterbium trifluoride
Urethane dimethacrylate
Triethylene glycol dimethacrylate
Methacrylated phosphoric acid ester
Polyethylene glycol dimethacrylate
1,10-decandiol dimethacrylate
Dibenzoyl peroxide

PermaCem 2.0; DMG (PCM)

Figure 9

Content

Barium glass in a Bis-GMA-based matrix of dental resins (Bis-GMA, TEDMA, EBPADMA, UDMA, HEMA)
MDP
Starters: CQ and BPO
Pigments
Additives
Catalysts

RelyX Unicem 2; 3M (RCM)

Figure 10

Contents:

Base Paste
Methacrylate monomers containing phosphoric acid groups
Silanated fillers
Methacrylate monomers
Initiator components
Stabilizers
Rheological additives
Catalyst Paste
Methacrylate monomers
Alkaline (Basic) Fillers
Silanated fillers

Initiator components
Stabilizers
Pigments
Rheological additives

A load of 1000 g was applied for 10 min during the cementation process and light irradiated for 20 s from buccal, lingual, mesial and distal sides for a total of 80 seconds.

Specimens were stored in distilled water at 37° C for 24 h. Short term thermocycling of 10,000 cycles was applied over 9 days 30 second dwell time and 5 seconds in between baths. Specimens were placed in a fixture on a universal testing machine (Instron), aligned with the shearing blade just touching the bonding interface. A shear load was applied until failure at a crosshead speed of 0.5 mm/min. Loads were converted to MPa by dividing the failure load by the bonding surface area.

The fractured surfaces were inspected with a stereo microscope to evaluate the failure mode and were classified as adhesive, cohesive or mixed failures approximated by the amount of remaining resin cement on the ceramic surface in respect to the bonding surface area.

The data was analyzed by one-way ANOVA and pairwise multiple comparisons (Tukey Test) with $\alpha=0.05$.

Figure 11.

Figure 12.

Results:

Group Name	N	Missing	Mean	Std Dev	SEM
PSA MPa	12	0	8.642	1.786	0.516
TCM MPa	12	0	9.056	2.646	0.764
PV5 MPa	12	0	9.591	1.551	0.448
SCM MPa	12	0	8.544	1.926	0.556
PCM MPa	12	0	7.679	1.157	0.334
RCM MPa	12	0	7.701	1.320	0.381

Table 1 Use for means and Standard One-Way Analysis of Variance

Source of Variation	DF	SS	MS	F	P
Between Groups	5	33.915	6.783	2.099	0.076
Residual	66	213.229	3.231		
Total	71	247.144			

The differences in the mean values among the treatment groups are not great enough to exclude the possibility that the difference is due to random sampling variability; there is not a statistically significant difference (P = 0.076).

Group	N	Missing	Median	25%	75%
PSA MPa	12	0	8.100	7.540	9.615
TCM MPa	12	0	8.221	7.397	10.868
PV5 MPa	12	0	9.074	8.574	10.559
SCM MPa	12	0	7.926	7.044	9.926
PCM MPa	12	0	7.971	6.897	8.500
RCM MPa	12	0	7.265	6.632	8.779

Table 2 Kruskal-Wallis One Way Analysis of Variance on Ranks

H = 11.133 with 5 degrees of freedom. (P = 0.049)

The differences in the median values among the treatment groups are greater than would be expected by chance; there is a statistically significant difference (P = 0.049)

To isolate the group or groups that differ from the others use a multiple comparison procedure.

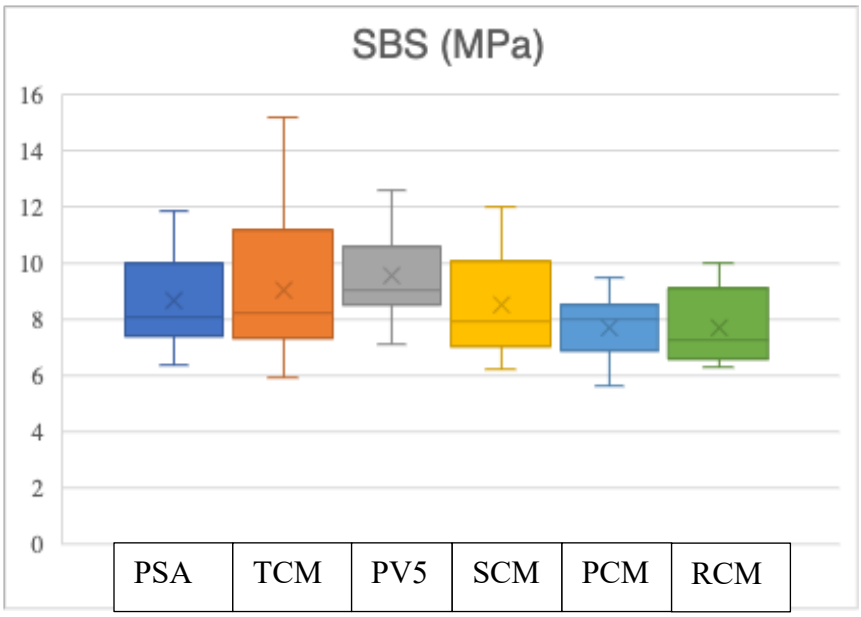
All Pairwise Multiple Comparison Procedures (Tukey Test):

Comparison	Diff of Ranks	q	P<0.05
PV5 MPa vs RCM MPa	303.000	4.179	Yes
PV5 MPa vs PCM MPa	279.000	3.848	No
PV5 MPa vs SCM MPa	182.000	2.510	Do Not Test
PV5 MPa vs PSA MPa	162.500	2.241	Do Not Test
PV5 MPa vs TCM MPa	150.500	2.076	Do Not Test
TCM MPa vs RCM MPa	152.500	2.103	No
TCM MPa vs PCM MPa	128.500	1.772	Do Not Test
TCM MPa vs SCM MPa	31.500	0.434	Do Not Test
TCM MPa vs PSA MPa	12.000	0.166	Do Not Test
PSA MPa vs RCM MPa	140.500	1.938	Do Not Test
PSA MPa vs PCM MPa	116.500	1.607	Do Not Test

PSA MPa vs SCM MPa	19.500	0.269	Do Not Test
SCM MPa vs RCM MPa	121.000	1.669	Do Not Test
SCM MPa vs PCM MPa	97.000	1.338	Do Not Test
PCM MPa vs RCM MPa	24.000	0.331	Do Not Test

Based on One Way ANOVA test, there was no statistically significant difference between the shear bond strength to zirconia for the test groups compared to the control group (P= 0.076).

Pair wise comparison using Tukey test revealed that shear bond strength with RCM group was significantly lower than the control PV5 group (P=0.049). No significant difference was noted between the other test groups with the control group or in inter-group comparison.



Graph 1

The fractured surfaces were inspected with a stereo microscope to evaluate the failure mode and were classified as adhesive, cohesive or mixed failures approximated by the amount of remaining resin cement on the ceramic surface in respect to the bonding surface area.

A scanning electron microscope (FEI Quanta 600 ESEM; FEI Co) in the Singh Center for Nanotechnology at University of Pennsylvania was used to image and evaluate the failure modes.

Images of the specimens were captured at 100x, 500x and 1000x magnification for detailed evaluation.

Majority of the specimens had a mixed failure mode and photos from SEM evaluation can be noted here.

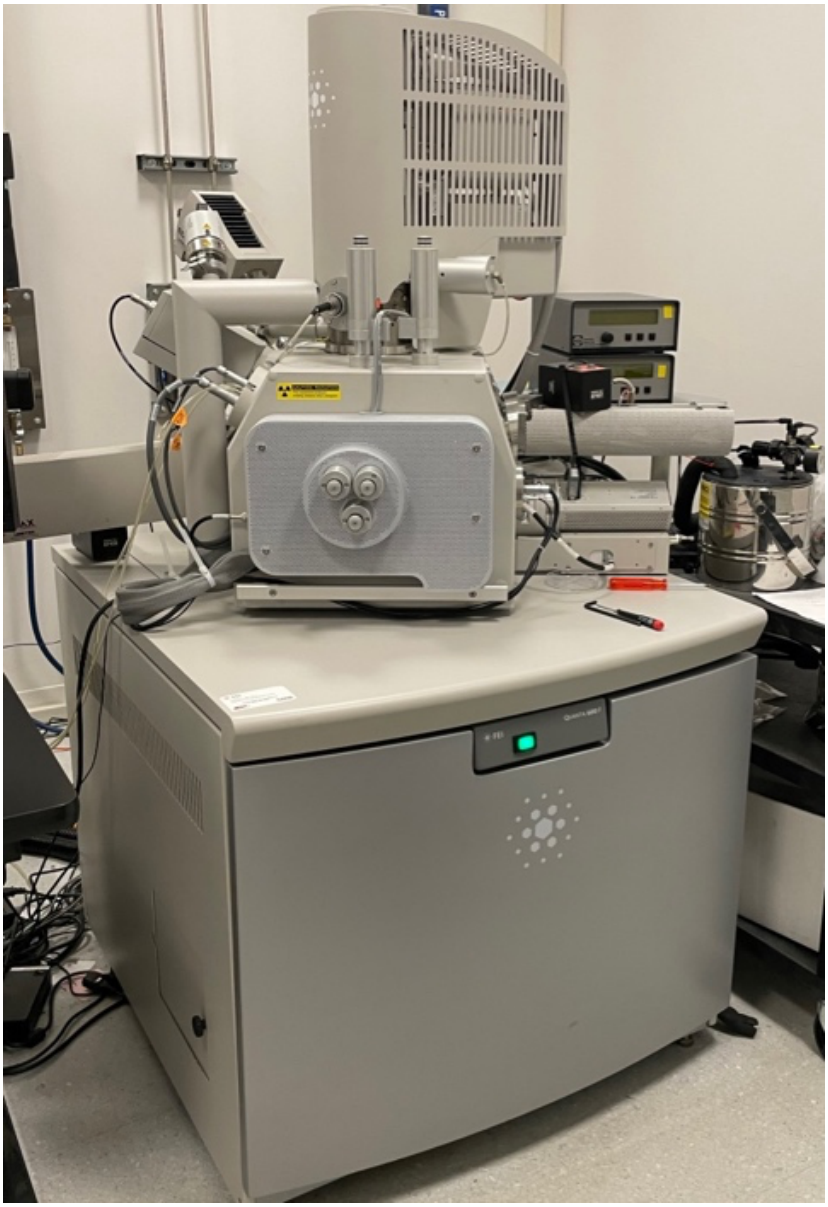


Figure 13 Scanning electron microscope

Panavia V5 (Control group)

Sample shown below in Figures 14,15 and 16 has mixed failure with 70% of the specimen showing cohesive failure within the resin and 30% showing adhesive failure exposing the zirconia surface.

All tested samples in this group showed mixed failures with >50% areas of cohesive failure within the resin.

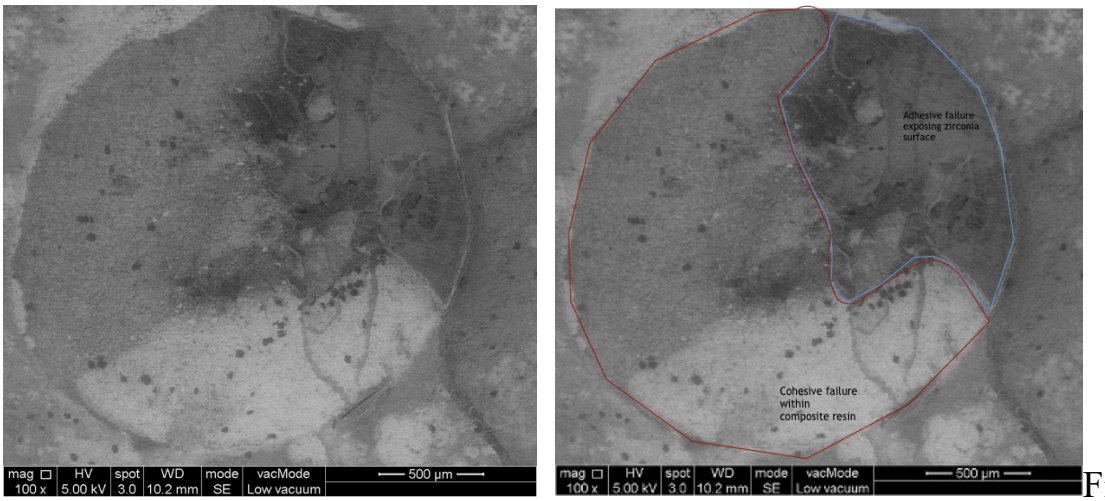


Figure 14

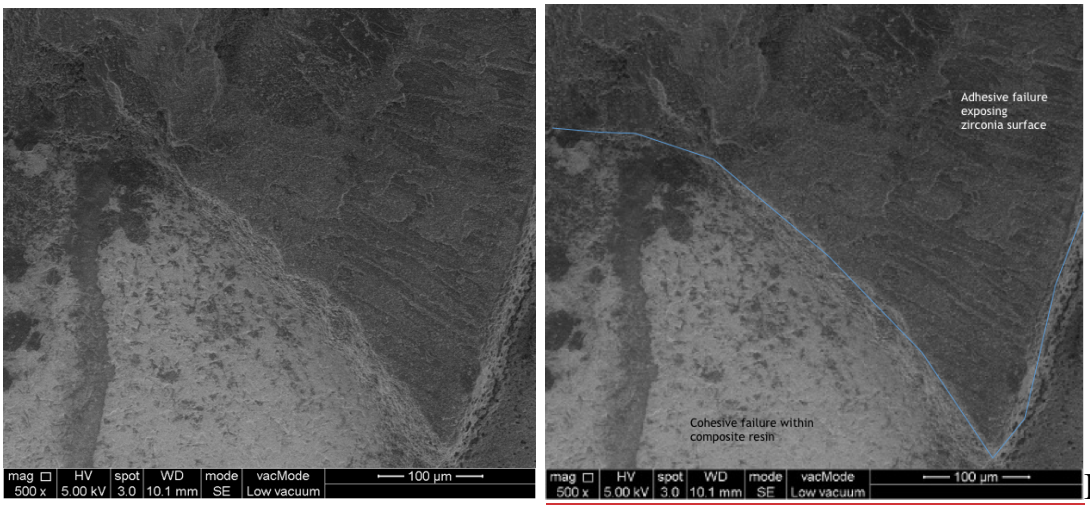


Figure 15

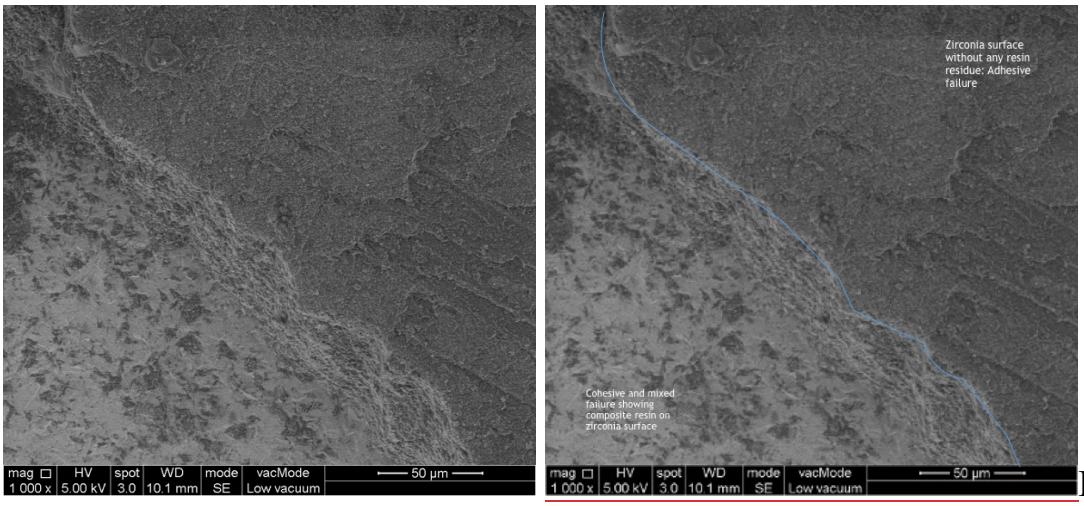


Figure 16

Panavia SA Universal:

Sample shown below in figures 17, 18 and 19 has mixed failure with 50% of the specimen showing cohesive failure within the resin and the remaining surface showing resin infiltrates on zirconia surface (mixed failure).

All tested samples showed mixed failures with variable amounts of cohesive and adhesive failures.

Figure 17

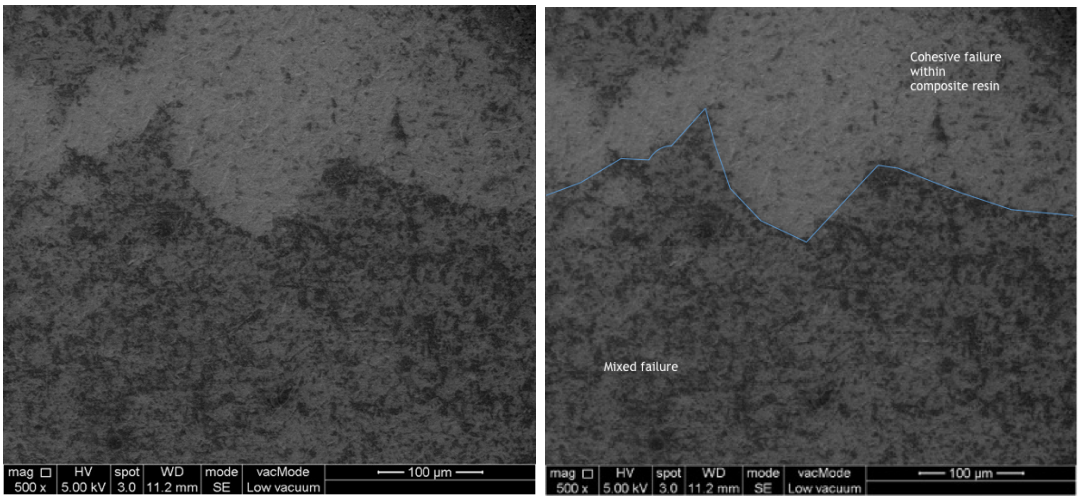


Figure 18

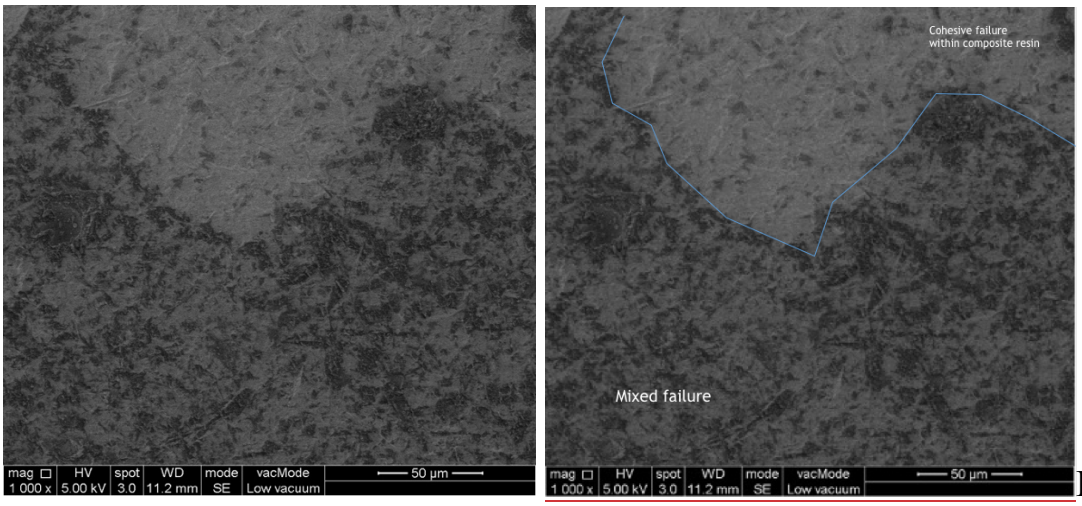


Figure 19

PermaCem 2.0:

Sample shown in figures 20, 21 and 22 has mixed failure with 80% of the specimen showing adhesive failure exposing the zirconia surface and 20% cohesive failure within the resin showing.

All tested samples showed mixed failures with variable areas of cohesive and adhesive failures.

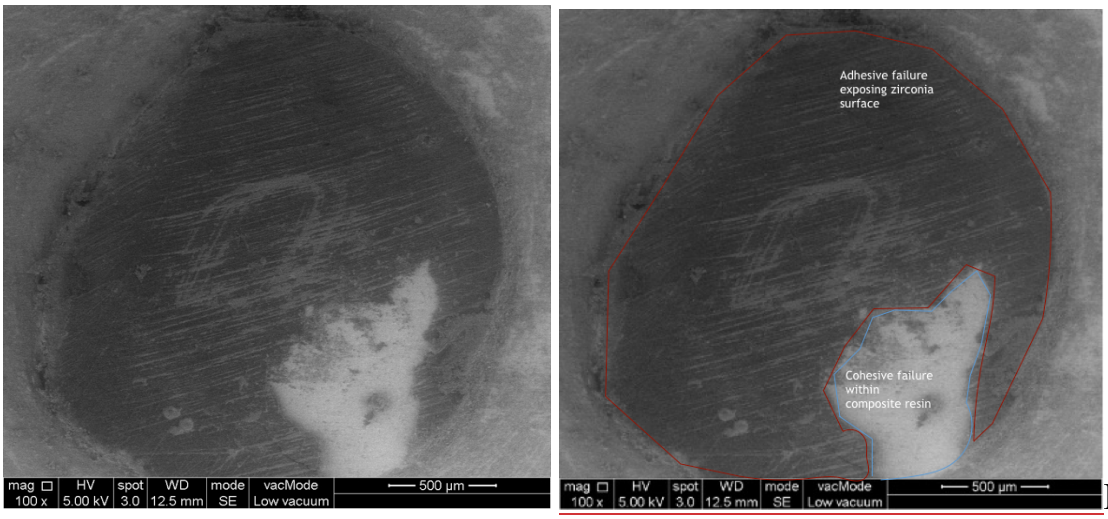


Figure 20

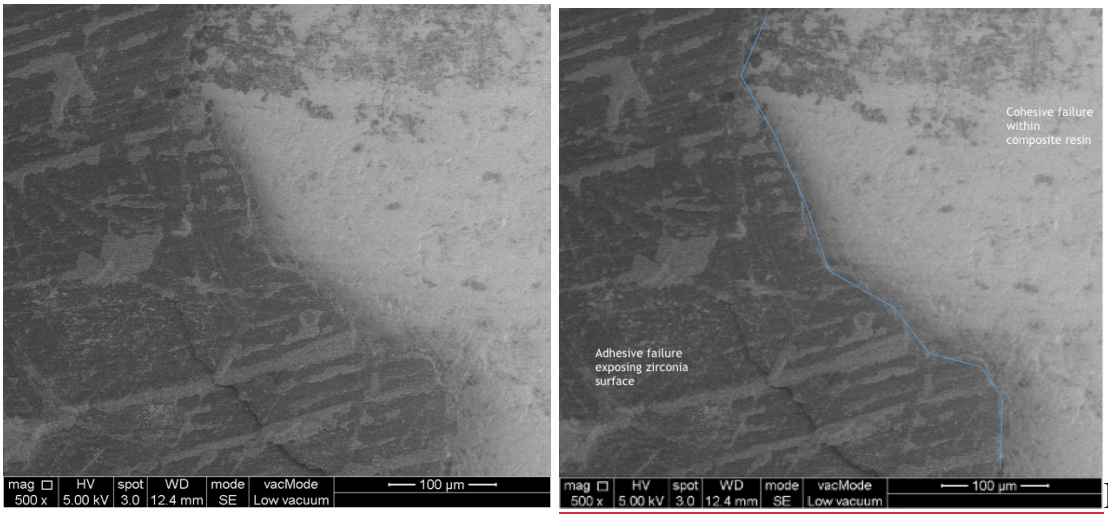


Figure 21

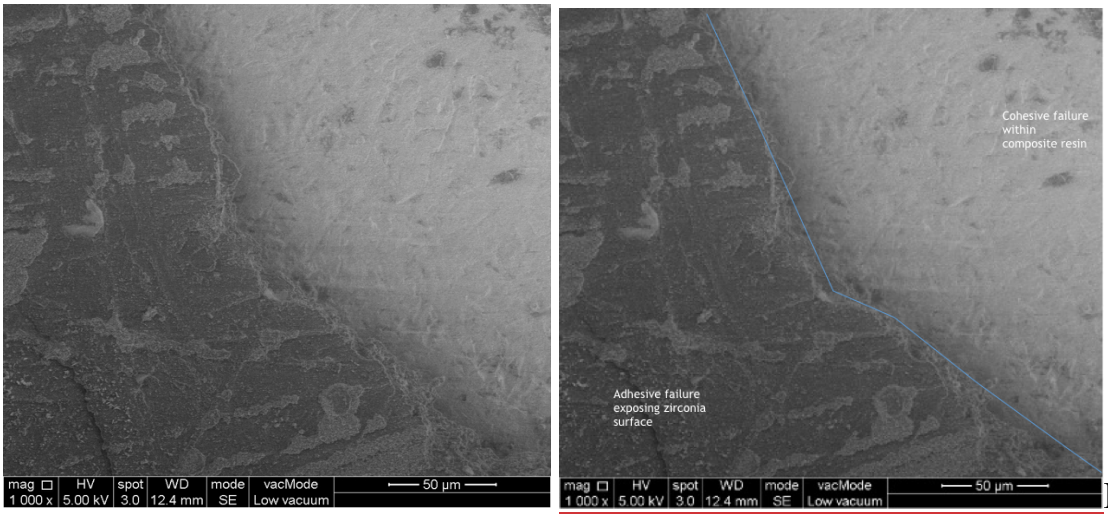


Figure 22

RelyX UniCem 2:

Sample shown in figures 23, 24 and 25 has mixed failure with 80% of the specimen showing cohesive failure within the resin and 20% showing adhesive failure exposing the zirconia surface.

All tested samples showed mixed failures with variable amounts of adhesive and cohesive failure.

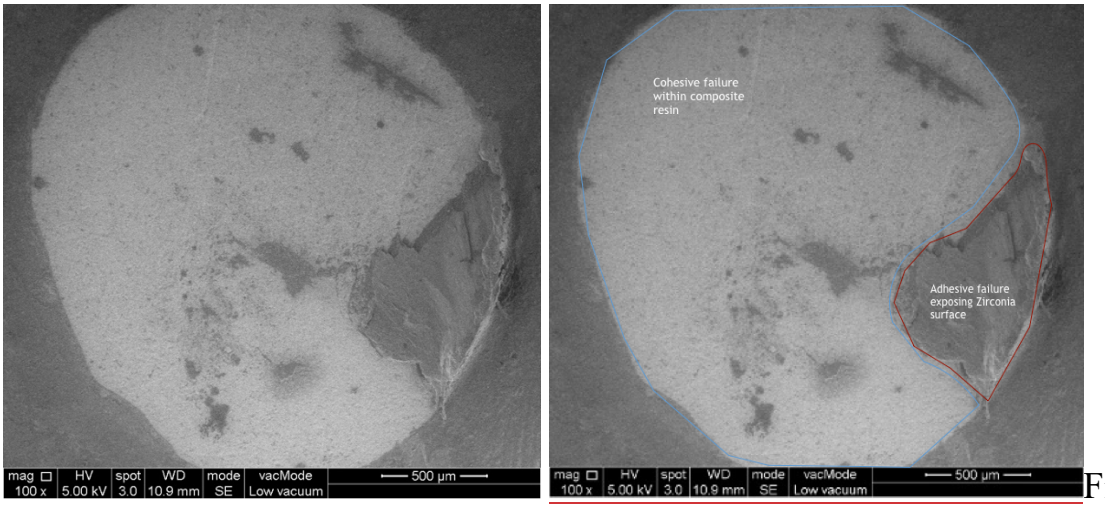


Figure 23

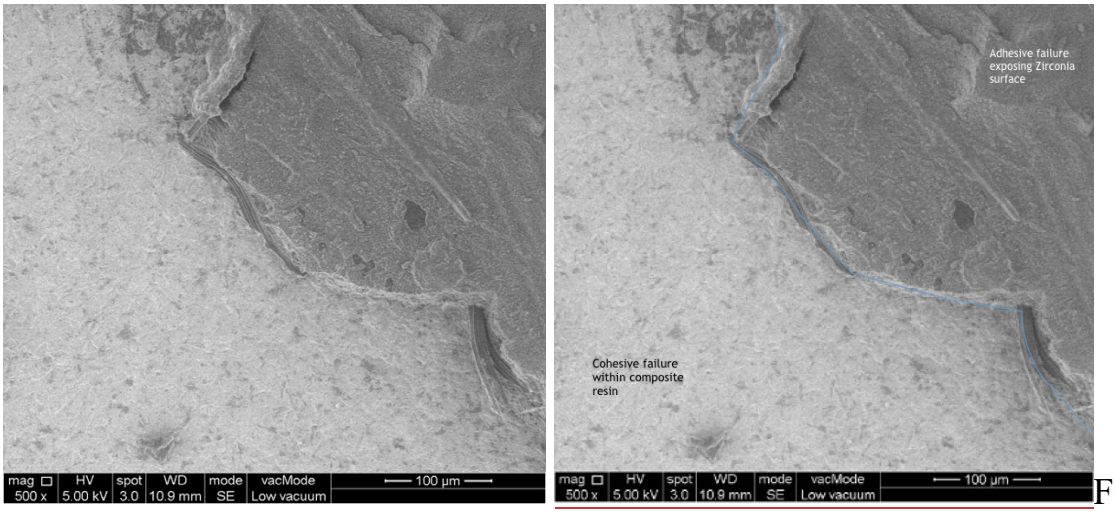


Figure 24

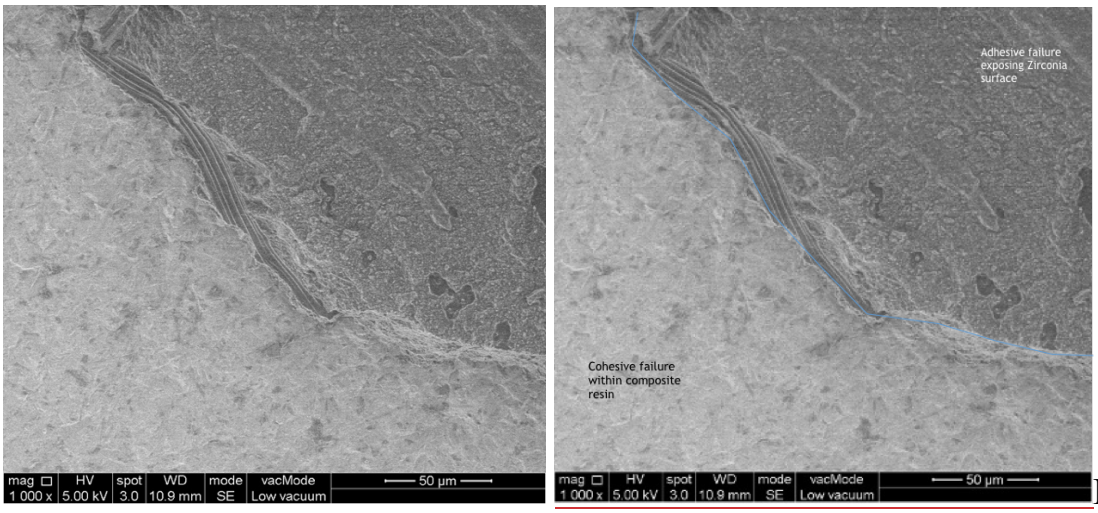


Figure 25

TheraCem:

Sample shown in figures 26, 27 and 28 has mixed failure with 60% of the specimen showing cohesive failure within the resin or mixed failure and 40% showing adhesive failure exposing the zirconia surface.

Majority of the tested samples showed mixed failures and one sample has adhesive failure and no resin was noted on the specimen.

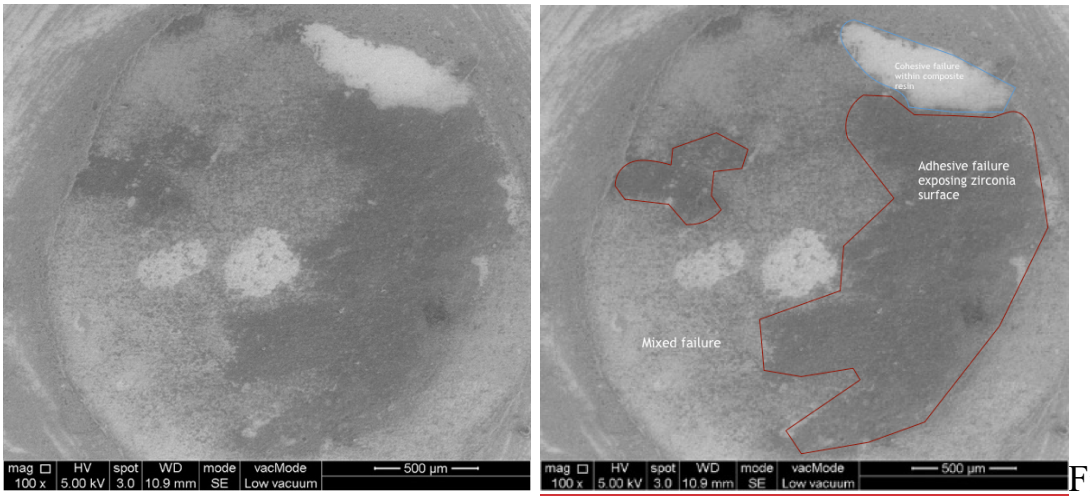


Figure 26

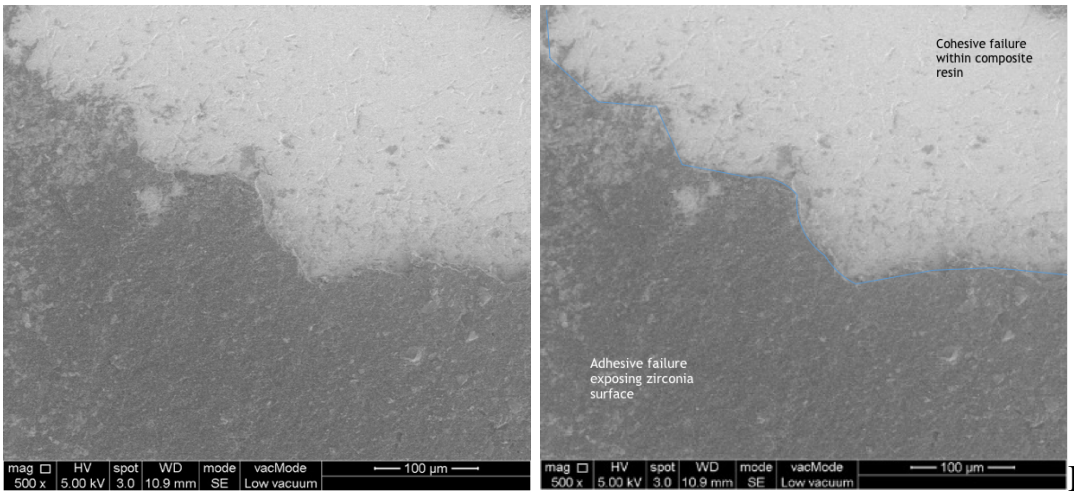


Figure 27

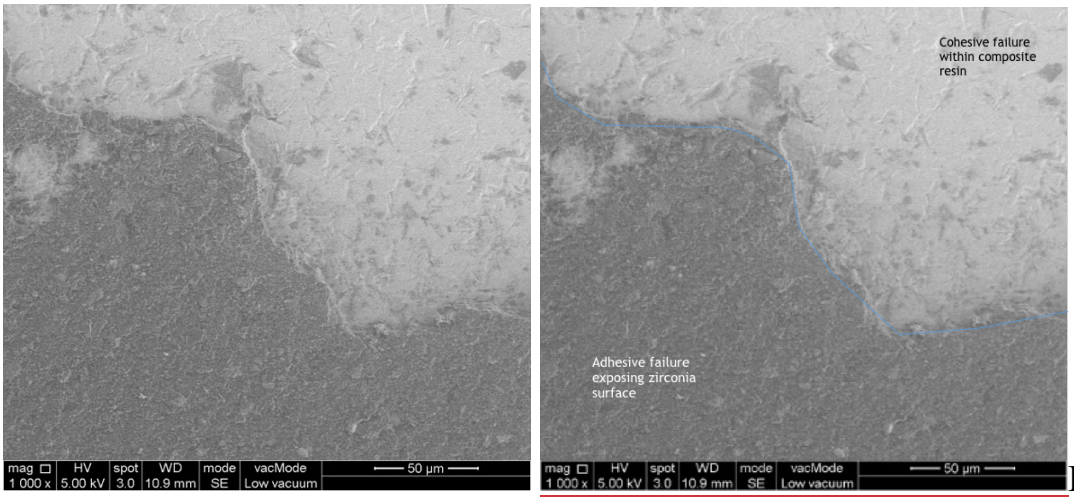


Figure 28

SpeedCem Plus:

Sample shown in figures 29, 30 and 31 has mixed failure with 70% of the specimen showing adhesive failure exposing the zirconia surface and 30% showing cohesive failure within composite resin.

Majority of the tested samples showed mixed failures and one sample had adhesive failure and no resin was noted on the specimen.

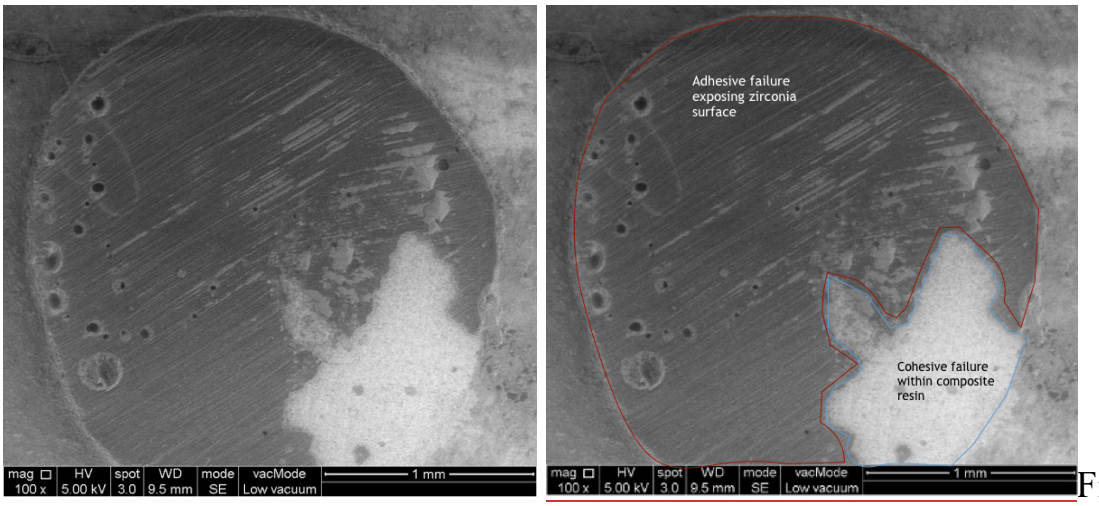


Figure 29

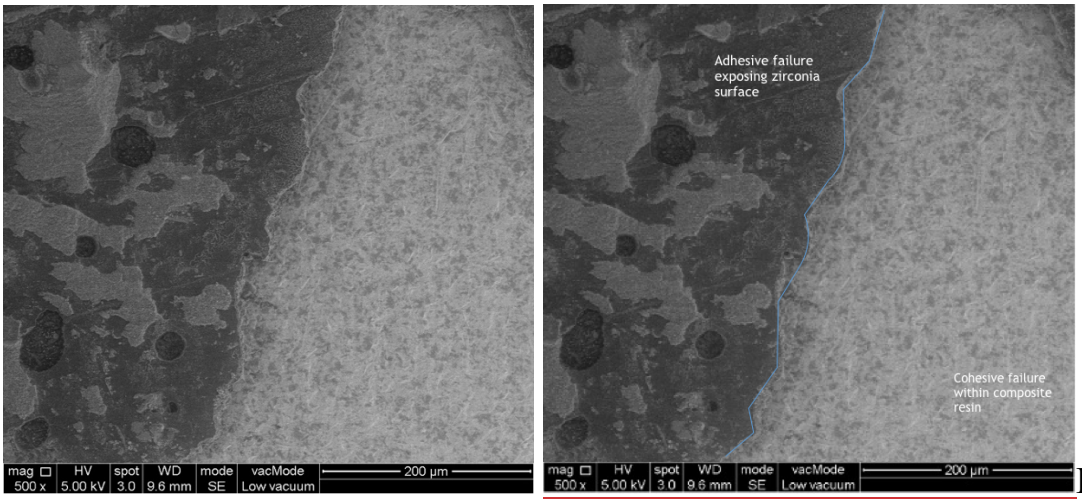


Figure 30

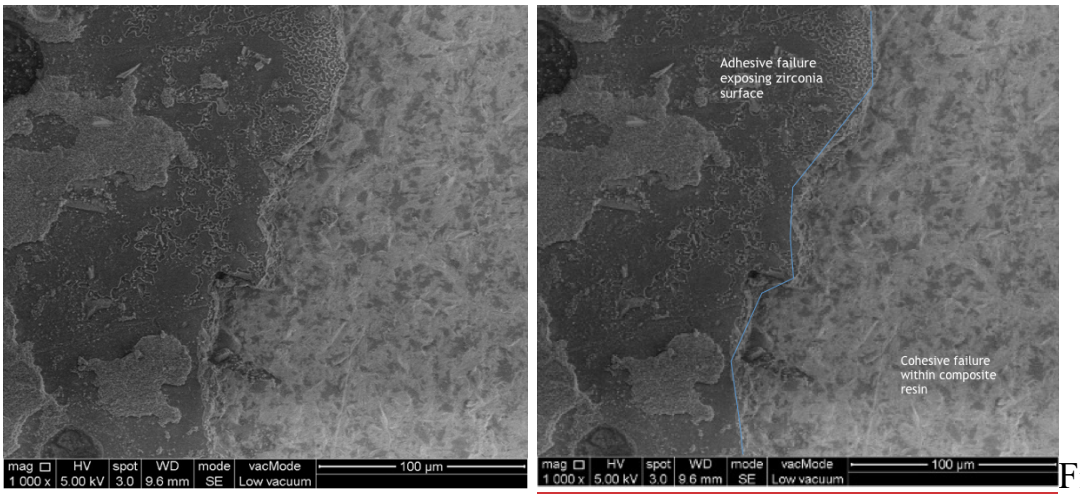


Figure 31

Discussion:

The results of this study partially reject our null hypotheses that there is no difference in shear bond strength to zirconia between multi step dual cure resin cement and self-adhesive resin cements. One of the self- adhesive resin cements (RCM) tested had significantly lower bond strength values compared to control dual cure resin cement. However, all other groups tested did not show statistically significant difference. Based on the results, it can be implied that self- adhesive resin cements can be a less technique sensitive, more user-friendly alternative to multi-step dual cure resin cements for bonding to zirconia. Success of the cement groups tested can be attributed to the fact that all groups tested contained 10-methacryloyloxydecyl dihydrogen phosphate (MDP) in their cement composition or ceramic primer (control). MDP has been a key element in achieving a robust bond to zirconia along with air particle abrasion.[2,5,9]

In terms of clinical implications, self-adhesive resin cements can be used for bonding full coverage zirconia restorations.

The most common test method to assess bonding effectiveness to zirconia is the shear bond-strength test [10], most likely because it requires nearly no further specimen processing of the fully sintered zirconia, once the bonding procedure is completed. However, the shear-bond strength test has been repeatedly documented

to result in inhomogeneous stress distribution along the interface, for instance often leading rather to 'cohesive' failures in the substrate than to 'adhesive' failures at the actual interface.[10,66]

Conventional shear bond and tensile tests are limited by specific fracture patterns, which may cause cohesive failure in the ceramic or composite. The fractures seen in the present study are predominantly mixed or cohesive within the composite resin. This failure indicates that the bond strength exceeds the fracture resistance of the composite resin material, which limits the ability to interpret the acquired data since fracture strength rather than the bond strength is tested in those instances. In the present study, there were no cohesive failures in zirconia ceramic since the fracture strength of zirconia ceramic far exceeds that of the composite resin and the bond strength.

In the present study, the bonding of the specimens was achieved under 1000g load to achieve homogenous film thickness and uniform interface. Majority of the specimens had mixed failures with part of the specimen showing adhesive failure and part of the specimen showing cohesive failure within composite.

While in vitro studies indicate a significant increase in flexural strength of high-strength ceramic restorations after resin bonding [52], the evidence on the exact influence of the cementation medium on clinical performance is limited. For full-

coverage high-strength ceramic crowns and FDPs and based on the specific clinical situation, the clinician can choose between resin bonding with composite resins, insertion with a self-adhesive resin, or conventional cementation with zinc-phosphate, glass ionomer, or resin-modified glass ionomer cement. [13]

In the mouth, water and repeated thermal changes constantly degrade and hydrolyze the ceramic/composite-resin interface. Long-term water storage at a constant temperature or thermal cycling are the most often used conditions to simulate aging of resin bonds. However, it has been shown that different bonding systems are influenced differently by these two parameters.[10]

Multiple studies have shown the effect of simulated aging on the resin-ceramic bond.[3,4,5] Even though direct clinical correlations and conclusions cannot be drawn from such ageing methods, their significant effects on the bonding interface have been demonstrated by multiple studies. Long term storage and thermocycling dramatically decreased resin bonds to high strength ceramic materials, underlining the need for such methods in order to enhance clinical relevance. Studies have employed short term water storage for 3 days [3,21], short term thermocycling of 10,000 cycles[70] or long term thermocycling of 12,000 cycles over 180 days [3,21]. In the present study, bonded specimens were stored at 37° C for 24 hours before short term thermocycling of 10,000 cycles over 10 days between 5° C and 55° C with a dwell time of 30 seconds. Although it is difficult to calculate the amount of clinical

ageing associated with amount of simulated ageing, it is important to incorporate some form of simulated ageing before testing bond strength to enhance clinical relevance.

Limitations of this study include the in-vitro set up, short term thermocycling application as well as being single-operator study. Other factors besides bond strength also play a role in success of full coverage and partial coverage zirconia restorations. Clinical studies with multiple operators and long-term performance in the oral environment can provide more clinically relevant data.

Conclusion:

According to the results of this in vitro study, self-adhesive resin cements can achieve shear bond strengths to zirconia that are comparable multi-step resin cements. In routine clinical application, self-adhesive resin cements can be a user-friendly, less technique sensitive alternative for bonding Zirconia restorations following the APC concept.

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