MONOLITHIC ZIRCONIA PARTIAL COVERAGE RESTORATIONS: AN IN-VITRO CLINICAL SIMULATION

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ABSTRACT

Savita Gupta: Monolithic zirconia partial coverage restorations: An in- vitro clinical simulation (Under the direction of Taiseer Sulaiman)

To evaluate the survival rate and bonding efficiency and marginal integrity of monolithic zirconia partial coverage restorations bonded using air particle abrasion, a primer with 10-methacryloyloxydecyl dihydrogen phosphate and composite resin (APC) protocol.

Human maxillary premolars were prepared for monolithic 3Y zirconia partial coverage restorations. Group1 (control) full coverage crown, group 2 and 3 preserving 2mm functional or nonfunctional cusp width, group 4 overlay preparation. The restorations were bonded using the APC protocol. The samples were exposed to simultaneous thermocycling and mechanical loading, SEM analysis were performed to detect failure (cracks/micro cracks) and marginal defects.

In group 2, one specimen debonded and SEM analysis at 30X indicated marginal integrity issue of the remaining seven intact specimens. None of the specimens failed due to fracture. No other specimens demonstrated marginal integrity issued at 30X. None of the specimens demonstrated any micro crack at 100X and 150X.

Due to its fracture resistance, 3Y-zirconia may be considered as a desirable material for partial coverage restorations. Following a strict bonding protocol, bonding zirconia seems to be promising and durable. Occlusal contact on restoration margins should be avoided.

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LIST OF ABREVIATION AND SYMBOLS

°C	Degree of Celsius
°F	Degree of Fahrenheit
μ	Micron
%	Percent
~	Approximately equal
>	Greater than
wt. %	Weight percent
mol%	Molecule percent
10-MDP	10- methacryloyloxydecyl dihydrogen phosphate
4-META	4-methacryloxyethyl trimellitate anhydrate
3-TMSPA	3-trimethoxysilanyl-propyl-N-aniline
MAC-10	Methacryloyloxy-1,10-undecanedicarboxylic acid
CAD	Computer aided Design
CAM	Computer aided manufacturing
CPD	Critical point drying
CaO	Calcium oxide
MgO	Magnesia oxide

mm	Millimeter(s)
MPa	Mega Pascal
Ν	Newton
SEM	Scanning Electron Microscope
vol.	Volume
Y ₂ O	Yttria oxide
Y-TZP	Yttria Stabilized tetragonal Zirconia

CHAPTER 1: REVIEW OF THE LITERATURE

1.1 Introduction:

Restorative dentistry is continuing to improve as patients preserve their natural teeth until old age. Direct restorative care remains the most conservative and non-invasive treatment as it minimizes the amount of unsound tooth structure removed, maximizes the remaining amount of intact sound tooth structure, limits damage to the pulpal and surrounding tissues¹ and is associated with lower financial cost as compared with indirect restorative procedures.

Creation of proper anatomy, resulting in proper function, is more difficult to directly accomplish when a large amount of tooth structure is missing. In these cases, indirect restorations fabrication enables better control over re-establishment of natural tooth contours.

Restorative material that have been used to fabricate partial coverage restoration include cast metals, ceramics and composite resin.² Gold is considered to be "gold standard" as compared with all other restorative material in terms of clinical longevity. A classic study by Donovan et al, evaluated the long-term success rate of 1314 cast gold restoration in service for 20 years or more, placed by single dentist and concluded survival rate of 97% at 9 years, 90.3% at 20 years.³

Increasing cost of high-noble and noble metal alloys and the increased demand for esthetics has resulted in declining use of cast metals over the past decade along with a corresponding increased use of tooth-colored dental materials.² Ceramic materials are considered an alternative for the posterior teeth with extensive loss of tooth structure due to their mechanical strength.⁴ Compared to resin composite these restorations have superior aesthetics, chemical durability, biocompatibility and resistance to compression and wear.⁵ Esthetic demands, principles of

minimally invasive dentistry and advancement in adhesive technologies have expanded indications for tooth colored partial coverage restorations.⁶

Preservation of natural tooth structure increases the likelihood of ongoing pulpal vitality as well as serves as guide for the creation of a restoration that will mimic natural tooth anatomy in the arch. Multiple restorative efforts, each an insult to the pulp, increases the risk of irreversible damage.⁷ Increased amounts of remaining enamel and dentine improves thermal insulation and, along with heat dissipation properties of pulpal vascularity, help to limit the impact of temperature rise during tooth preparation.⁸

Longer preparation times and higher temperature used in more aggressive preparations increases the risk of thermal damage. Zach and Cohen found that 10°F rise in temperature results in tissue necrosis of 15% of teeth tested, a 20°F rise resulted in pulpal necrosis of 60% of teeth tested and a 16.6°F rise caused necrosis of all the teeth tested.⁹ Intrapulpal increase of temperature up to 16°F during extensive tooth preparation depend on the rotary cooling method, pressure, bur design and degree of vibration.¹⁰ Felton et al. found 13.3% of teeth restored with full coverage crowns lost vitality over the long term as compared to 0.5% of unrestored teeth.¹¹ Irreversible pulpitis has been reported with full coverage to occur in 5.7% of cases in which crowns were placed on vital teeth.¹² According to Edelholf and Sorensen,onlay preparation removes 39% of the total tooth structure, whereas preparation for a complete crown requires removal of between 72.3% and 75.6%.¹³

Partial coverage restorations are minimal invasive dentistry which not only preserve healthy tooth structure but are also kind to the periodontium because most of the margins are supragingival.^{5,14} Partial indirect restorations classified as inlays (without covering the cusps),

Onlays (covering at least 1 cusp) and overlays (covering all cusps)^{5,15,16} allow more dental structure to be conserved.^{13,17}

Three factors must be considered when using partial coverage restorations to conserve sound tooth structure: cavity preparation design, type of ceramic material/manufacturing process, and type of luting cement.¹⁷⁻²⁰

1.1.1 Cavity preparation design: It should be conservative, meet the requirement of the restorative material and replace destroyed tooth tissue. Several studies have examined the influence of preparation design on the fracture resistance of restorations. Study by Stappert et al demonstrated that different preparation design of the partial coverage restorations has no significant influence on the restoration's fracture resistance and the preparation should be defect oriented.¹⁷

1.1.2 Type of restorative material: Composite resin may be chosen for small to moderate sized restorations, however larger restorations should be restored with ceramic. Ceramics have shown to have better marginal adaptation, color match, wear resistance, anatomic form and survival probability compared to composite resin.¹⁸

1.1.3 Type of luting cement: Dual-cured resin luting cement is the material of choice for cementing this type of restoration as it bonds to enamel, dentin, and the restorative material and successfully polymerizes in areas that light may not be able to reach.¹⁹ Resin cements limit micro leakage, and increases the strength of the restorations.²⁰

1.2 Material used for partial coverage restorations:

A variety of ceramic materials are available today. Products include silica-based ceramic (feldspathic porcelain, leucite-reinforced ceramics, and lithium disilcate ceramics) and non-silicabased ceramics (alumina based, and zirconia based). For the purposes of clarity, these non-silicabased ceramics will be simply referred to as alumina and zirconia.

Available long-term clinical data for ceramic partial coverage restorations have revealed that most common complication is the ceramic bulk fracture, despite ceramic thicknesses of at least 1.5 mm.^{15,21-23}The value of these finding is limited because most of the studies were of low strength feldspathic porcelain or medium- strength leucite- reinforced glass ceramics.^{15,24-26}

Properly created etchable feldspathic porcelain and glass ceramic veneers have demonstrated long term clinical success in the treatment of anterior teeth.²⁷ Fracture of etchable ceramic materials is the most common complication when considering the veneering of posterior teeth, especially when covering the one or more cusps or whole occlusal surfaces, however, when properly designed, enables durable clinical performance.^{15,17,21,22,28} Limiting material flexure, and resultant fracture, requires etchable ceramic restorations to be 1.5-2 mm thick.^{29,30}

A systemic review and meta-analysis on the survival rate of resin and ceramic Inlays, onlays, and overlays concluded that the survival rate at 5years is 92-95% and 10years is 91% and ceramic fractures were the most frequent cause of failure.²³

With the recent development of Computer-Aided Design/Computer-Assisted Manufacture (CAD-CAM) technologies and new ceramic materials, it is possible to mill full contour zirconia restorations, and thus, eliminate the need for veneering with porcelain. This full contour restoration can avoid ceramic chipping and reduce wear of antagonists.^{31, 32}

Manufacturers are advertising monolithic restorative materials with improved mechanical and optical properties for use with CAD/CAM technologies.³¹ Limited studies of these materials have compared the various mechanical properties when configured as CAD-CAM fabricated partial coverage restorations.^{33,34} Sen et al compared the translucency and biaxial flexural strength of 5 monolithic CAD-CAM restorative materials and concluded that zirconia-reinforced glass-ceramic revealed higher biaxial flexural strength than resin nanoceramic, feldspathic ceramic, lithium disilicate ceramic, and dual-network ceramic.³⁴ Lithium disilicate glass ceramics have been developed for the fabrication of partial coverage restorations and have demonstrated increased fracture resistance as compared with leucite reinforced glass ceramic restorations.³⁵

Alumina and zirconia with no glassy matrix are available as CAD/CAM blocks. Zirconia has superior mechanical properties.^{36, 37} It is the ceramic material of choice for full coverage indirect restorations of posterior teeth in areas of high stress. Extensive research has been accomplished on the use of zirconia for full coverage dental restorations.

1.3 Zirconia as a restorative material for full coverage restorations:

Zirconia holds a unique place amongst oxide ceramics due to its excellent mechanical properties, biological properties, as well as, when properly polished, low wear of the antagonist dentition. In addition, the inherent phase transformation phenomenon, occurs during the phase transition, results in superior resistance to fractures seems to restrict micro-crack propagation during function.^{38, 39} In the past high strength zirconia was generally layered with veneering porcelain, which is prone to fracture because of weak interface.⁴⁰ Therefore, to eliminate the risk of veneer chipping or fracture, full anatomical monolithic zirconia restorations are available.⁴¹

Unalloyed Zirconia is a polymorphic material that may exist in three crystallographic forms, depending on temperature and pressure: monoclinic(stable at room temperature up to 1170° C), tetragonal(stable at 1170-2370°), and cubic (stable from over 2370° to its melting points 2716°C).^{42,43} At room temperature, pure zirconia is present in the most stable phase, monoclinic. As the temperature rises to about 1170°C, the monoclinic phase transform into the tetragonal phase, accompanied by shrinkage in volume of approximately 4-5 percent. The tetragonal phase converts into cubic phase at about 2370°C, with only minimal changes in volume.^{42, 43} This transformation can be prevented by stabilizing tetragonal zirconia at room temperature by alloying with various oxides such as CaO, MgO and Y2O and leading to high toughness.^{44, 45, 46} Zirconia ceramics in dentistry are commonly stabilized with 3mole% Yttria.

Materials containing only tetragonal phase are strongest, while cubic containing zirconia is significantly weaker but more translucent (cubic zirconia).^{47,48} The yttrium oxide, or yttria, content in zirconia based dental material largely defines the mechanical and physical properties. Different generations of zirconia used in dentistry have been classified by the yttria content (mol %)

1 st Generation	2 nd Generation	3 rd Generation	4 th Generation
3 mol % yttria	3 mol % yttria	5 mol % yttria	4 mol % yttria
0.25 wt. % alumina	0.05 wt. % alumina	0.05 wt. % alumina	0.05 wt. % alumina
>15% cubic phase	>15% cubic phase	50% cubic phase	25%cubic phase
Flexural	Flexural	Flexural	Flexural
strength:1000+MPa	strength:900+MPa	strength:~500MPa	strength:~700MPa
Least translucent	More translucent than	Most translucency	Less translucent than
	1 st generation		3 rd generation

Table1.1

One major advantage of monolithic zirconia restorations is that crowns can be fabricated with more conservative preparations as compared to lithium disilicate crowns while at the same time demonstrate greatly superior strength. All etchable glass-ceramic materials require 1.5-2.0 mm of material thickness to limit flexure under load and resultant fracture. ^{29, 30} In addition, all etchable glass-ceramic materials must be adhesively attached to supporting tooth structure for adequate resistance to catastrophic fracture secondary to occlusal loading. First and second-generation zirconia materials do not require adhesive cementation for clinical durability. Furthermore, the inherent resistance to fracture, even in restoration dimensions similar to those required for metal alloys, represents another major advantage of first- and second-generation zirconia materials.^{38, 39} However, there are clinical circumstances (e.g. preparations with inadequate resistance and retention form) that indicate the use of adhesive bonding technologies with zirconia restorations so as to increase the likelihood of successful clinical outcomes.

1.4 Bonding to zirconia:

Zirconia, with no glassy amorphous phase, cannot be etched and adhesively attached to underlying tooth structure in the same way as other glass-ceramics. Therefore, it is considered to be a non-adhesive restorative material. A definite approach for optimal bonding to dental zirconia, more specifically yttria-stabilized tetragonal zirconia polycrystals (Y-TZP), has yet to be found. It has been widely reported that following the strict protocol and application of products containing the phosphate ester monomer 10-methyacryloyloxydecyl dihydrogen phosphate (10-MDP) results in better zirconia crown cementation. ⁴⁹⁻⁵³ The 10-MDP molecule contains a terminal phosphate functional group, which reacts with zirconia and forms a P-O-Zr bond. The other end of the molecule is containing a vinyl group that is available for copolymerization with resin cement. These two functional groups are separated by a carbon chain which, when combined with other resin types and filler amounts, is responsible for handling characteristics such as viscosity, rigidity, hydrophobicity, and solubility. Resin mixtures containing 10-MDP promote better adhesion than those containing other monomers with affinity for zirconia such as 4-META, MAC-10, or 3-TMSPMA.⁵⁴⁻⁵⁸ However, the use of resin cements containing 10- MDP alone does not seem to be able to maintain adequate adhesion levels after thermocycling.^{59,60} Priming of the zirconia surface with 10-MDP in their chemistry as well as traditional composite resin cements that do not contain 10-MDP.^{56,60-62}

Tzanakakis et al. extensively reviewed studies and listed the methods and materials used to enhance adhesion to the surface of zirconia in order to better understand and apply the best methods.⁶³ However, a standardized adhesive cementation protocol has not been identified at that point.

Many studies have evaluated the effect of different surface treatments on the bond strength of zirconium dioxide to a resin luting agent.^{50,51,64} Bottino et al. evaluated the effect of silica coating on zirconium-dioxide ceramic bond strength to resin. It was found that tribo-chemical silica coating systems increases the tensile bond strength between zirconia and phosphate monomer contain resin composite.⁵¹

Wegner and Kern found that the thermal cycling induced bonding failure between zirconia ceramics and conventional Bis-GMA resin luting agents.⁶⁵ These results were also supported by various laboratory studies that showed that the bond strength to silica coated zirconia ceramic

statistically significantly decreased after a few months of artificial ageing.^{50,52} Improved bond stability was obtained with airborne particle abrasion of the zirconia surface followed by application of the adhesive phosphate monomer 10-methacryloxydecyl dihydrogen phosphate(10-MDP)⁵⁰ Air abrasion with alumina particles accomplished surface cleaning/roughening. The 10-MDP monomer contains phosphate ester and methacrylate groups which promotes chemical bonds to oxide ceramics and therefore, surface priming with 10-MDP enabled more durable resin adhesion.⁶⁶

Fairly recent meta-analysis by Inokoshi and Thammajark et al. concluded that the combination of mechanical and chemical zirconia surface pretreatment contributed to adhesive resin cement bond durability irrespective of cement used.^{67,68} Therefore, airborne particle abrasion has become a standard pretreatment practice. The resin bond to high-strength ceramics has been investigated for than 2 decades now. The classic articles by Kern et al. demonstrated that strong and durable long-term resin bonds were achieved only after surface pretreatment with air particle abrasion and use of an adhesive composite resin luting agent that contains special adhesive phosphate monomers, 10-methacryloyloxydecyl-dihydrogen phosphate (10-MDP) ^{50,65}

To simplify this protocol, Blatz has now introduced a simplified zirconia bonding concept (The APC Zirconia Bonding Concept) that summarizes the 3 critical procedural steps: ⁶⁹

APC – Step A: Air particle zirconia abrasion with aluminum oxide.

APC-Step P: Priming of abraded zirconia with 10-MDP and

APC- Step C: Cementation with dual-cure or self-cure composite resin luting agent.

Air particle abrasion (traditionally referred to as "sandblasting") uses material erosion, by means of energy released from the impact of high-energy alumina particles (Al₂O₃), to create a rough surface with improved wettability.⁵⁹ Air particle abrasion is able to modify the zirconia surface.⁷⁰ However, the inherent strength of the zirconia material may be compromised when excessive particle impact energy results in deep surface defects and fractures.⁴⁹ Levels of material erosion require control of surface impact energy and may be accomplished by careful attention to abrasion parameters of 1) air pressure, 2) particle size, 3) particle source distance and 4) total time of particle impact.

In 2013, Ozcan proposed a protocol for blasting zirconia, alumina particle size with a diameter between 30 and 50 μ m, at a pressure of the abrasion between 0.5 and 2.5 bar for a duration of at least 20 s. Abrasion technique requires that the particle delivery tip be kept in constant motion to create uniform surface erosion without localized deep defects.⁷⁰ It is essential to follow surface material erosion with the use 10-MDP-based primers and cements designed for this purpose.⁷¹

Long term randomized clinical trials are the best means to assess actual performance of dental restorations. Clinical trials are costly, time consuming, involve ethical approvals and, in order to publish a properly conducted 5-year clinical trial, it may take up to 10 years.^{72,73} Therefore, a consideration should be given to the design of in vitro testing parameters that are most likely to provide prediction of actual clinical performance.

1.5 Laboratory fatigue testing

In 1969, Bjorn Hedegard outlined the following goal," With sound clinical research on a larger and more penetrating scale, data and information may be obtained, that will make it possible

to set up more meaningful test procedure in the laboratory. And that is the goal: to be able to characterize the dental material in the laboratory and correctly predict its clinical performance".⁷⁴

How much closer are we to this goal?

Chewing simulation is a relatively new technique of laboratory testing in which restorative dental materials are fatigued by emulating the intra oral environment. These simulators are purposed for in-vitro predicator tests prior to in-vivo studies. Chewing simulators are complex mechanical machines that perform a wide range of movements according to preset parameters, accompanied by controlled artificial aging with different solutions. This machine offers control over test parameters, is computer controlled, can be optionally combined with thermo-cycling and may be used for fatigue testing and wear studies.⁷⁵

In general, restorative materials and teeth fail as a result of occlusal stresses that are too small to induce immediate fracture. These stresses result in propagation of defects, that are inherent to the material, over time eventually result in tooth/restoration failure by fatigue. The most clinically relevant in vitro studies involving ceramic materials involve fatigue testing under water, as ceramics fail at much lower loads underwater than in air.⁷⁶ In human mouth, physiological occlusal forces show a high variability between individuals during food mastication and swallowing, and range between 10 and 120N.^{77, 78} This protocol thermomechanical fatigue application of 1.2 million cycles were equivalent to 5 years of clinical performance.⁷⁹

The fatigue testing machine allow adjustment of the following testing parameters⁸⁰⁻⁸⁵: 1) Loading Force, 2) Loading frequency, 3) Vertical/Lateral movement and 4) Antagonist material and shape Testing parameters in fatigue testing machines:

- Loading force: loading of samples uses at least three different approaches: 1) use greater forces than those found in humans to speed up the simulation process, 2) use forces comparable to those in humans but with much higher number of cycles, 3) use various load levels to determine sample fatigue resistance by submitting clinically relevant specimens with a sufficient number of cycles in a wet environment to a range of different descending loads.⁸⁰
- Loading frequency: review of the dental literature reveals frequency of loading (also known as "chewing rate") to be ≤2 Hz (cycles/s).^{81,82,83}
- 3) Vertical and lateral movement: in most chewing devices, the distance and speed of the vertical and lateral jaw movement can be determined and set at different ranges. Researchers have various opinions as to the relative importance of vertical and/or lateral force delivery. Kim et al. recommended the consideration of the lateral movement in any laboratory simulation.⁸⁴ Rosentritt et al. found no statistically significant difference between applying no lateral movement and 1mm lateral movement while vertically loading their specimens.⁸⁵
- 4) Antagonist: natural tooth antagonists are more accurately approximate clinical conditions, but standardization is difficult due to the large variability in size, form and shape. Therefore, ceramic and stainless- steel indenters are the most commonly used antogonists.⁸⁰

The variation of simulation parameters, such as chewing frequency, thermal cycling, cyclic loading, level of humidity, movement while loading, type of abutment (e.g. natural tooth, metal),

simulated periodontal ligament or antagonist type may result in different testing outcomes. Despite the large amount of laboratory simulation data available (e.g. a search of PubMed results in approximately 200 references to chewing simulator or simulation), the validation of laboratory tests that simulate chewing is limited and data correlating in-vitro outcomes with in-vivo experience are rare.^{86,87} Moreover, there is no definitive guidance on the criteria and standardization of chewing simulation parameters.²⁴ Even so, the use of chewing simulation parameters that create cyclic loading and thermal cycling in a wet environment may help to predict how a new dental material or new configuration of a previously tested material may perform clinically.

1.6 Zirconia as a restorative material for partial coverage restorations:

Research focusing on partial coverage with zirconia as a restorative material is sparse. The few published research studies have evaluated the load bearing capacity and fracture resistance of bonded zirconia onlays but did not evaluate the fatigue resistance of the partial coverage restorations.^{88,89,90}Therefore, the overall objective of this study was to examine the effect of clinically simulated aging, through thermo-mechanical cyclic loading, of bonded monolithic second-generation zirconia partial coverage restorations on tooth/restoration fatigue resistance and resistance to debonding and marginal integrity.

CHAPTER 2: RESEARCH STUDY

2.1 Specific aims of this research study:

To evaluate the effect of thermo-mechanical cyclic loading (chewing simulation) of monolithic zirconia partial coverage restorations on:

- 1. Tooth/restoration fatigue resistance
- 2. Bonding efficiency of restoration to tooth structure (bond longevity with aging)
- 3. Marginal integrity

2.2 The null hypotheses:

Thermo-mechanical cyclic loading (chewing simulation) does not have an effect:

- 1. On tooth/restoration fatigue resistance
- 2. On bonding efficiency of restoration to tooth structure (bond longevity with aging)
- 3. On marginal integrity

2.3 Materials and methods:

2.3.1 Test sample preparation

Thirty two freshly extracted intact human maxillary premolars free of caries, cracks and restorations, as determined by examination under 4.5 magnification loupes, were cleaned of calculus and soft tissues by hand scaling and were stored in 0.05% thymol solution at 25°C prior to use. This research study was assessed by the UNC Institutional Review Board to be human studies exempt (18-2933). Teeth were randomly divided into 4 groups of eight specimens each. A single operator prepared teeth for one group at a time. Preparations were created using a high-

speed electric hand piece (~200,000rpm) with air and water cooling using with 330MWV, 846 KR .31.016M and 8846KR.31.016F diamond modified flat-end taper burs (Brassler, Savannah, GA) Standardized tooth preparations were completed as follows:

Group 1 (control, full crown preparation): Premolar preparation began with 1.5mm depth cuts on the occlusal table through the occlusal groove, preparation was tapered ~4-8 degrees and a wide 1.0mm wide rounded shoulder margin was placed 1.0mm above the CEJ. All internal line angles were rounded, and all surfaces were smoothed. (Figure 2.1 A)

Group 2 MODL onlay preparation: Premolar preparation began with creation of 2mm depth cut mesiodistally through the central groove and extended laterally to completely include the nonfunctional cusp and preserve 2mm of the buccal-lingual width of the functional cusp. Occlusal reduction was then completed. Mesial, lingual and distal axial wall preparation resulted in a 2mm pulpal depth from the occlusal cavo-surface margin of functional cusp and axial wall height of approximate 3mm. A wide 1mm wide rounded shoulder margin was placed above the CEJ. All the internal preparation angles were rounded, and all surfaces were smoothed. (Figure 2.1 B)

Group 3 MODF onlay preparation: Premolar preparation began with creation of 2mm depth cut mesiodistally through the central groove and extended laterally to completely include the flattened functional cusp and preserve 2mm of buccal-lingual width of the non- functional cusp. Occlusal reduction was then completed. Mesial, facial and distal axial wall preparation resulted in approximate 2mm pulpal depth from the occlusal cavo-surface margin of nonfunctional cusp and axial wall height of approximate 3mm. A wide 1mm wide rounded shoulder margin was placed 1mm above the CEJ. All the internal preparation angles were rounded, and all surfaces were smoothed. (Figure 2.1 C)

Group 4 MODFL onlay veneer: Premolar preparation began with creation of 1mm depth cuts so as to allow uniform removal of tooth structure following the occlusal anatomy. A wide 1mm wide rounded shoulder margin was placed with axial height of 1mm inclined towards the occlusal surface. All the internal preparation angles were rounded, and all surfaces were smoothed. (Figure 2.1 D)

All the measurement for preserved buccal lingual width were verified with a digital caliper (63731, Pittsburgh, PA, USA) to within 0.25mm. Other dimensions were verified with periodontal probe.



A:Group 1: Full crown(control)



B:Group 2: Preserving 2mm nonfunctional cusp



C:Group 3: Preserving 2mm functional cusp



D;Group 4: Overlay/Tabletop

Figure 2.1. Group preparation designs

2.3.2 Fabrication of the restorations

CAD/CAM blocks of 3Y-Monolithic zirconia (ZirCad, A1 LT Ivoclar Vivadent) were used. (Table 2.1). An intraoral scanner, Planmeca Emerald (Planmeca, Finland)) were used to scan the preparations and restorations were designed using software (Romexis E4D, Texas, USA) (figure 2.2). Minimal occlusal thickness of the onlays was 1mm, minimal axial thickness was 1mm were milled in PlanMill 40S (Planmeca, Finland). Preset tolerances within the software programing ensured minimal occlusal thickness of 1.5mm and minimal axial thickness of 1mm for all restorations. The milled restorations were 20-25% enlarged to compensate for shrinkage after the sintering process. Restorations were crystallized in speed sintering oven Programat CS4 (Ivoclar)





2.3.3 Cementation of restorations:

Restorations were bonded according to APC concept:

A: Air particle abrasion with aluminum oxide particles 50µ particle size, low pressure (2 bar) for 15 seconds at the distance of 10mm and internal surfaces were thoroughly rinsed the with air/water spray and dried.

P: Primer Monobond Plus (Ivoclar Vivadent: Schaan, Liechtenstein) was applied to abraded surfaces for 60 seconds and dispersed it with a strong stream of air per manufacturer's instructions. (Table 2.1)

Multilink Primer (Ivoclar Vivadent: Schaan, Liechtenstein) was applied to the tooth preparations per manufacturer's instructions. In brief, primer A and B were mixed in a 1:1 ratio (1 drop of Primer A and 1 drop of Primer B). The self-etching and self-curing mixture were then applied with a scrubbing action for 30 seconds to the entire preparation using a micro brush, starting with the enamel surface.

C: Composite resin: Cementation of restorations was accomplished using the Multilink automix (Ivoclar Vivadent, Schaan, Liechtenstein) per manufacturer's instructions. (Table 2.1)

The restorations were seated with finger pressure and stabilized while excess material was immediately removed with a microbrush and glycerin gel was applied to cover the restoration margins during resin cement gelation. Cemented restorations were light cured after 2 minutes for 20 seconds per surface (Blue phase G2 Ivoclar Vivadent, Schaan, Liechtentein). A radiometer (Bluephase Meter II, Ivoclar Vivadent, Schaan, Liechtenstein) was used to measure light irradiance of approximately 1200mW/cm².

All Specimens were stored for 24±1 hour at 37°C and relative humidity 100% in deionized water after bonding restorations to prepared teeth and before submitting to artificial ageing.

Material	Manufacturer	Type of material	Composition	Application procedure
IPS	Ivoclar	3Y-TZP	ZrO ₂ ,Y ₂ O ₃ ,HfO ₂ ,	
e.max	Vivadent;		Al ₂ O ₃	
ZIrCAD	Schaan,Liechte			
Alumina (Al ₂ O ₃)	Danville; San Ramon, CA, USA	Alumina	50µm Al2O3	Sandblast the internal surface from a distance of 10mm for 10-15
				seconds at 2bar pressure
Multilink Automix	Ivoclar Vivadent; Schaan, Liechtenstein	Multilink Primer A/B	PrimerA: Aqueuos solution of initiator PrimerB: HEMA,phosphoric acid, acrylic acid monomers	Mix equal amounts of primer A and B and apply self-curing primer A/B on enamel surface and for 15s on dentin surfaces with micro brush, rinse with water and dried with oil-free air
		Monobond Plus	10- Methacryloyloxyde cyl dihydrogen phosphate, silane methacrylate, ethanol, sulphide methacrylate	Apply to the pre- treated surfaces. Allow the material to react for 60 seconds. Disperse with air
		Self-cure composite resin cement	22-26% Dimethacrylates,6- 7% HEMA,<1% Benzoyl peroxide,40% Barium Glass,YF ₃ , spheroid mixed oxide	Apply luting material directly to restoration after dispensing from the auto mix syringe. Seat with pressure Remove excess material, self-curing with option of light cure.

Table 2.1. Materials Used

2.3.4 Fatigue testing

Specimens in each group were exposed to dynamic loading of 70 N at a frequency of 1.4 Hz for 1.2 million cycles with simultaneous thermocycling (10,000 cycles, 5-55°C, 30s dwell time/cycle) in a computer controlled multifunctional mastication simulator (SD Mechatronik, Fedkirchen- Westerham, Germany) to accomplish fatiguing. This protocol of thermomechanical fatigue application of 1.2 million cycles were equivalent to approximately 5 years of clinical performance.⁷⁹

Test specimens were oriented parallel to the long axis of the tooth and embedded in autopolymerizing acrylic resin (VariDur 200, Buehler, Lake Bluff, IL, USA) up to 1 mm below the CEJ in the cylinder specimen holders of the chewing simulator. A 4mm diameter stainless steel antagonist was used to represent an opposing cusp. Initial contact of the antagonist was associated with the internal incline of the palatal cusp followed by 1 mm of lateral movement to the central groove (representing a functional slide). A 1mm vertical indentation threshold facilitated constant contact during this motion. (Figure 2.3)



Figure 2.3. Schematic diagram of mechanical loading pattern

2.3.5 Evaluating failure

Wear detectors mounted in each chamber monitored the specimens and recorded the cycle number during which specimens' failure may have occurred. All surviving specimens were examined under 4.5X magnification for visible damage in the restoration or tooth following fatigue testing. Failure was defined as restoration or tooth fracture development and restoration debonding.

2.3.6 Scanning electron imaging:

Scanning electron imaging (SEM) was performed following fatigue testing to evaluate the microcrack in tooth/restoration and restoration-antagonist contact areas were evaluated for marginal integrity (Hitachi S-4700 Field Emission Microscope). Samples were attached to an aluminum SEM sample holder with double-stick carbon tape. Samples underwent alloy conductive

coating with a layer of 10nm Gold-Palladium (AuPd). SEM operating conditions were as follows: Normal mode, 2kV accelerating voltage, 10microA beam current, and a working distance of close to 12mm was maintained throughout the analysis.

Also, CPD (Critical point drying) was performed on Tousimis Aotosamdri-931 CPD system.

Statistical analysis: The failure rate for each group was determined by visual inspection of each test sample after testing. Restoration-antagonist contact areas will be evaluated by Scanning Electron Microscopy for evidence of material fatigue. A test of Proportions statistical analysis will be conducted to identify survival rates.

2.4 Results:

2.4.1 Fatigue testing:

All specimens in Group 1, 3 and 4 survived the simulated aging process. There were no macroscopically visible cracks or fractures within the tooth structure and ceramic restorations. One Group 2 restoration debonded. All samples in Group 2 showed minimal ceramic cohesive fracture (chippings) at the occlusal margin in the area of the antagonist initial contact.

2.4.2 Scanning electron imaging:

The findings from the visual inspection were verified by SEM and failure modes as observed with SEM were similar in group 2. The crack origin was located at the margin during the cyclic loading.

Evaluation of other Group 2 samples at 30X magnification revealed minor chipping of the restoration margins where the antagonist came into contact with the occlusal surface. None of the

restorations in Group 1, 3, 4 had chipping of the restoration margins. Further assessment of the occlusal surfaces of all restorations at 100X and 150X magnification revealed no evidence of ceramic fracture development.



Figure 2.4. SEM images. A: Group 1. B: Group 2. C: Group 3. D: Group 4

Table 2.2. Findings	of SEM Observation:
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	Margin	Crack or Fracture	
	30X	100X	150X
Groups	Yes Problem	Yes Problem	Yes Problem
1	0	0	0
2	7	0	0
3	0	0	0
4	0	0	0

Statistical analysis results:

2.5 Methods: Test of Proportions (P=.30)

- Although there was 1 failure in group 2, this failure rate was not statistically significantly different from 0 observed in the other group(P=.30)
- For SEM analysis at 30X all margin of 7 samples in group 2 showed a marginal integrity issue. This rate was statistically significantly different from the rate of 0 in the other groups(P=0.0001)

2.6 Discussion:

This in vitro research study exposed adhesively bonded partial coverage zirconia restorations to simultaneous thermo-cycling and mechanical testing in an attempt to more closely simulate an environment in which dental ceramics are normally fatigued.²⁴

Extracted human premolar teeth for restoration abutments were used in this study because of their bonding characteristics, thermal conductivity, modulus of elasticity and strength more closely represent actual clinical conditions as compared with other types of abutments^{.17,91} Other research studies used a cyclic loading of 49N.^{92,93,94}Research has shown that occlusal forces in the posterior dentition may exceed functional loading forces of 49N during mastication or swallowing so, therefore a cycle loading force of 70N was chosen.⁹⁵

The first null hypothesis was accepted as none of the tested samples fractured. The high survival rate in this study is comparable to two in-vitro studies.^{96, 97} Differences in test parameters limit direct comparison of specific results with these studies. To the best of the author's knowledge, no study has investigated the effect of thermomechanical cyclic loading of monolithic zirconia partial coverage restorations on the bonding efficiency of restoration to tooth structure.

The second null hypothesis was also accepted since this study showed high survival rate of monolithic zirconia partial coverage restorations following a strict bonding protocol and manufacturer's instructions regardless of the preparation design specifics. This is in agreement of Sasse and Kern studies on single-retainer zirconia-based resin bonding fixed denture prosthesis.^{98, 99} High 5-year survival rates were demonstrated in these studies when zirconia restorations were adhesively bonded with either 10 MDP- containing composite resin or an adhesive bonding system with 10 MDP. Another clinical study by Kern et al, demonstrated excellent clinical outcomes of cantilever single-retainer zirconia-based restorations (with no retentive features) that had been adhesively bonded with MDP containing composite resin.¹⁰⁰

The single bond failure in Group 2 (1 out of 8, a 12.5% failure rate) was not found to be statistically significant (P=.30). Visual examination revealed that the antagonist made contact on the natural tooth and slide over the margin of Group 2 specimens. It may be that antagonist contact with the restoration margin resulted in tensile forces that stressed the adhesive bond of the ceramic onlay to the tooth. It may be that the failure rate in this group may have increased if the number of cycles had been increased.

The third null hypothesis was rejected since all remaining samples in Group 2 showed degradation of marginal integrity after thermo-mechanical cyclic loading. It has been well described in the literature that failure in ceramic restorations can initiate from a number of different sites. Near contact occlusal surface modes including outer and inner Hertzian cone cracks or partial cone cracks when sliding contact occurs.¹⁰¹⁻¹⁰⁴ This is in agreement with data from previous investigations, which reported that crack initiation started at the cement interface.²⁴When a ceramic layer is uniformly supported by, and bonded to, a less stiff material, high tensile stresses develop in the ceramic at its interface with the cement, directly below the loaded area. These so-called radial cracks are known to be a severe failure mode in monolithic ceramics.^{103, 104}

The steel ball contacting the tooth surface and sliding over the margins results into chipping of ceramic from Hertzian contact stresses. Marginal design of partial coverage zirconia restorations should be carefully and precisely determined so as to avoid direct margin loading, as this has critical effect on the local marginal integrity longevity of the zirconia restoration.

The results from this study add to the existing body of evidence that 3Y zirconia is durable and able to withstand the normal forces of mastication. This is likely because of its excellent flexural strength plus the ability of 3Y zirconia to undergo phase transformation so as to limit defect propagation. Groups 1, 3 and 4 survived extensive thermo-mechanical fatigue testing with no debonding or chipping. Samples in these groups received primarily compressive forces, while samples in Group 2 received a combination of compressive and tensile force. Bulk fracture was not detected in any of the restoration configurations when using SEM assessment at 30,100 and 150X magnification.

It appears that the combination of this specific formulation of zirconia, coupled with 10-MDP primer and resin cement can successfully resist significant occlusal forces in a wet environment as long as the occlusal forces are primarily compressive. It is tempting to speculate that there may be a difference in success when occlusal forces are primarily compressive as compared with compressive and tensile in nature. While it may be that the preparation configuration of Group 2 samples exposed the adhesive interface to tensile forces, the survival of 7 out of 8 of the onlays suggests that the presence of tensile forces may not be relevant. Posterior restorations are exposed to compressive, tensile and shear forces during mastication. When possible, all tooth designs should include retention and resistance features designed to help protect the adhesive interface against tensile forces. For example, it might be prudent to consider shoeing the functional cusp when planning a partial coverage restoration with zirconia. This would have the effect of minimizing tensile forces on the adhesive interface as well as protect restoration/tooth interface from direct occlusal contact.

Control groups to assess the role of each variable of the APC technique were not included in this study. A control group to compare the APC technique with conventional cementation, that may typically be used with zirconia preparation designs such as the one utilized in Group 1, was not included in this research study. Therefore, no definitive statements can be made relative to the roles of the APC technique or retention/resistance form in the clinical longevity of partial coverage 2^{nd} generation zirconia restorations. Further research studies should be designed to assess these issues.

The results indicate that it is possible to bond successfully to zirconia using a form of the APC technique. Whether the achieved bond is sufficiently strong and durable to retain non-retentive restorations like veneers and table tops has yet to be determined. The tooth preparations for groups 2, 3 and 4 had less resistance and retention form than a complete crown preparation, but all the tooth preparations used in this study had both retention and resistance form. Future studies should include tooth preparations with no resistance and retention form.

2.7 Limitations of the study:

One of the significant restrictions of this study is that only one formulation of a second generation of zirconia material was evaluated. There is evidence that 5Y zirconia may not have the ability to undergo phase transformation ⁴⁷ and little is known about 4Y zirconia It is notable that not all zirconia materials are the equivalent and PSZ from numerous producers have various details and substance structure, rendering distinctive physical and optical properties between these

materials.¹⁰⁵ Further research of various zirconia formulations is warranted. The findings of this study may not be predictive of the performance of other zirconia formulations.

This research was also limited by the use of water during testing rather than artificial saliva. It may be that lubricating effects of salivary components are greater than that of water. Further studies should seek to assess the potential difference. This may improve the clinical simulation of this laboratory testing process of zirconia.

2.8 Conclusions:

Within the limitations of this in vitro research study:

- Due to its fracture resistance, 3Y zirconia may be considered as desirable material for partial coverage restorations
- 2. Following a strict bonding protocol, bonding zirconia seems to be promising.
- Marginal design should be carefully and precisely determined to avoid direct occlusal contact.

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