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# Original Article

# **Effect of Bioactive Cement Versus MDP-Containing Resin Cement on Shear Bond Strength of Super High Translucent Zirconia to Dentin (An In-vitro Study)**

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# **Abstract**

**Aim:** Bonding to zirconia still represents a challenge due to being chemically inert. In an attempt to overcome this problem, we aimed to demonstrate the effect of newly introduced bioactive calcium aluminate-based cement versus MDP-containing resin cement on the shear bond strength of zirconia to human dentin; and detect their mode of failure. **Subjects and methods:** Twenty caries-free human maxillary first molars were flattened and mounted in acrylic resin. Twenty super high translucent zirconia discs (Zolid fx Zirconia ML) were obtained and air-abraded. Teeth and zirconia discs were randomly equally distributed into two groups (n=10) according to the cement type; Groups CB; bioactive cement (Calibra bio), Group TH; MDP-containing resin cement (TheraCem). Both groups were cemented according to their manufacturer instructions under a uniform vertical load of 5-kg and subjected to thermocycling. Shear bond strength was tested using a universal testing machine. After testing for normality, data sowed parametric distribution and were expressed as mean and SD. Data were statistically analyzed using independent t-test at a level of significance (P≤0.05). Failure modes were assessed visually, using scanning electron microscope and Energy dispersive x-ray spectroscopy analysis. **Results:** Groups CB showed statistically insignificant higher bond strength (19.06MPa  $\pm$ 2.76) than Group TH (17.85MPa ±3.57). All specimens in both groups showed mixed failure mode, with change in surface elemental compositions in both zirconia and dentin. **Conclusion:** Bioactive Calibra Bio and MDPcontaining TheraCem cements showed comparable shear bond strength to super high translucent zirconia after six-month aging simulation, which was higher than clinical acceptable values.

**Keywords:** Ceramic, Thermocycling, Scanning electron microscope (SEM), Energy dispersive x-ray spectroscopy (EDX).

#### **Introduction**

Zirconia restorations have been widely used in the dental field due to their superior fracture strength, high durability, biocompatibility and toughness.<sup>1</sup> Being highly opaque, conventional zirconia was avoided in the anterior esthetic zone unless veneered with esthetic ceramics. However, such technique increased the risk of veneer chipping.<sup>2</sup> Monolithic translucent, high translucent and ultra-high translucent zirconia were introduced to overcome such drawbacks. More advancement led to the introduction of multi-chromatic multilayered zirconia offering excellent esthetics with natural tooth shade gradient and maximum conservation aided by its durability.<sup>3</sup>

Although advancements were made to optimize the use of zirconia in esthetic areas, the main challenge persists in bonding to zirconia. The unique composition of zirconia having no intrinsic glass content with non-polar homogenous dense structure caused them to be chemically inert with compromised bond strength.<sup>4</sup> Thus, advancements in dental cements were made to improve the bond strength of zirconia.<sup>5</sup>

Ideal luting agent should have high dimensional stability, high esthetics, biocompatibility, durability, low viscosity, low film thickness, low chemical solubility, radiopacity, bioactivity, bacterial resistance and ease of use.<sup>6</sup> It should enhance the restoration retention and seal the gap between the restoration and the tooth structure.<sup>7</sup> Many types of cement were used for luting zirconia; however, it was found that zinc phosphate and glass ionomer cements could not form a lasting bond with zirconia.<sup>8</sup> On the other hand, conventional and MDP- containing resin cement showed higher bondability even after aging.<sup>8</sup>

An MDP-containing, dual-cured, selfadhesive resin cement; TheraCem, was believed to enhance bonding to zirconia and tooth structure through an adhesion promoting monomer (MDP; 10-methacryloyloxyidecyldihyidrogenphosphate). Also, it allowed calcium and fluoride ions release; helping tooth remineralization.<sup>9</sup>

A Bioactive, non-resin, self-curing, calcium aluminate glass ionomer cement; Calibra Bio, was also introduced as a type of bioactive cements, which combined the advantages of glass ionomer cements; providing adhesion to the tooth structure and low initial pH, and calcium aluminate cement; contributing to the apatite formation, stable pH and reduced solubility or degradation.<sup>10</sup> Its bioactivity was thought to

occlude the marginal gap with the deposition of mineralized deposits within the gaps when immersed in simulated body fluid.<sup>11</sup> They also allowed fluoride release, which was believed to help prevent secondary caries formation between the restoration and the tooth margin.<sup>12</sup>

By searching the literature, no sufficient information was revealed regarding the effect of bioactive cement (Calibra Bio) versus MDP-containing resin cement (TheraCem) on shear bond strength of Multi-layered super-high translucency (ML-S) zirconia to dentin. Thus, the current study aimed to evaluate the effect of these cements on the shear bond strength of (ML-S) zirconia to human dentin. The null hypothesis stated that either type of cements tested would not affect shear bond strength of (ML-S) zirconia to human dentin.

# **Subjects and Methods**

The materials used in the present study were presented (**Table 1**)

# **Sample size calculation**

Sample size calculation was performed to ensure results reliability.<sup>13</sup> Sample size was performed using statistical software (G\*Power software, Version 3.1.9.7, USA), with  $0.05$ - $\alpha$  and power of 80% rendering 10 samples per group.

# **Teeth preparation**

Twenty caries-free, crack-free freshly extracted human maxillary first molars, extracted due to periodontal reasons, were collected and cleaned using ultrasonic scaler (Woodpecker UDS-P LED, China) to remove all tissue tags then polished using pumice (Preppies, Whip Mix, USA) to ensure surface cleanliness. Cleaned teeth were stored in distilled water for a maximum of six months until all teeth were collected,<sup>9</sup> to preserve them from dehydration and to avoid microorganisms' growth, in a simple low-cost storage medium.<sup>14</sup>

<b>Brand</b> name	<b>Description</b>	Composition	<b>Manufacturer</b>	<b>Batch</b> number
<b>Ceramill</b> Zolid fx ML	Multi-layered super- high translucency Zirconia CAD/CAM blank (Shade $A2/A3$ ).	$\geq$ 99.0% ZrO <sub>2</sub> + HfO <sub>2</sub> + Y <sub>2</sub> O <sub>3</sub> . $8.5\% - 9.5\%$ Y <sub>2</sub> O <sub>3</sub> $\leq$ 5% HfO <sub>2</sub> $\leq$ $0.5\%$ Al <sub>2</sub> O <sub>3</sub> $\leq$ 1% Other oxides	Amann Girrbach, Koblach, Austria	2001001
Calibra <b>Bio</b> cement	Bioactive, permanent bioceramic, self- curing, water-based dental luting cement capsules (white shade).	Powder: Calcium aluminate, Glass powder, $<$ 5% Inert strontium fluoride Liquid: Water, < 10% Polyacrylic acid, < 5% Tartaric acid, Lithium chloride, < 1% Nitrilotriacetic acid/trisodium salt, BPA-free and <b>HEMA-free</b>	Dentsply LLC, USA.	00060257
<b>TheraCem</b> cement	Dual-cured, Automix, MDP calcium fluoride releasing, self- etching, self- adhesive resin cement (opaque shade).	Base: 20–50% Portland Cement, 30–50% Ytterbium w/ Barium Glass, 1–5% Ytterbium Fluoride, 1-5% BisGMA, 1-10% Proprietary Catalyst: 10-30% 10- Methacryloyloxydecyl Dihydrogen Phosphate (hydrophilicmonomer), 1-5% 2- Hydroxyethyl Methacrylate 1-5% Tert-butyl Perbenzoate	BISCO, Schaumburg, Illinois, USA.	2100005166

**Table 1: The materials used in the present study**

BPA: Bisphenol A., HEMA: 2-hydroxyethyl methacrylate, BisGMA: Bisphenol A-glycidyl methacrylate, MDP: 10-Methacryloyloxydecyl Dihydrogen Phosphate

The buccal surface of the gathered teeth was flattened using linear precision sawing machine (Isomet 4000, Linear Precision Saw, Buehler, USA) at low speed under copious water to expose the dentin surface, which was then finished with 600-grit abrasive paper (Dura-Gold, Dura-Gold store, USA). The roots of the prepared teeth were cut off manually; 1-2 mm apical to the cemento-enamel junction using a diamond disc (Strauss Brasseler, USA) at lowspeed**.**

Each prepared tooth was mounted in an acrylic resin (Acrostone Cold Cure Acrylic Material, England) block to facilitate their handling. The mounted teeth were then cleaned using a digital ultrasonic cleaner (CODYSON, CD-4820, China) for 3 minutes followed by airdrying to remove all debris on the bonded surface.<sup>15</sup> Each mounted tooth was then stored in distilled water in small numbered plastic jars till the time of cementation.

#### **Zirconia disc preparation**

To ensure standardization, uniform cylinders were cut from presintered multilayered super-high translucent zirconia blank (Ceramill Zolid fx ML, Amann Girrbach, Koblach, Austria) and sliced to the desired thickness (3.6 mm), which was 25% larger than the desired final size of the specimen to compensate for sintering shrinkage that would occur later. Slicing was performed using the same linear precision sawing machine at lowspeed of 2050 rpm using a diamond disc of 0.5 mm thickness under copious water to reduce heat generation. All discs were checked using a precise digital caliper (Micro-Etcher ERC**,**  Danville engineering Inc, USA) to verify their

thickness and diameter. Afterward, they were cleaned using ultrasonic cleaner for  $3 \text{ minutes}^{15}$ and air-dried to eliminate any residuals from the slicing procedures. All discs were sintered in a Programmat S1 sintering furnace (Ivoclar Vivadent AG, Switzerland) according to the manufacturer's recommendations to reach maximum strength without affecting their final physical properties,<sup>7</sup> then rechecked using digital caliper to verify the final discs dimension (5-mm diameter and 3-mm thickness).

The bonded surface of all zirconia discs was air-abraded using 50-μm aluminium oxide particles  $(Al_2O_3)$  (Danville materials zest anchors, LLC company, Germany) at 2.5-bars pressure for 20 seconds according to the manufacturer instructions at an adjusted distance of 10 mm perpendicular to the surface. To standardize the distance between the sandblaster device (Micro-Etcher ERC, Danville engineering Inc, USA) and the disc, a special custom-made holding device was fabricated (**Figure 1**). The holding device comprised a vertical metallic arm fixed to a flat horizontal metallic base to which a plastic cylinder was fixed to help carry the specimen during air-abrasion. A movable metallic housing was fitted to the vertical arm, which carried a horizontal arm that held another metallic housing carrying the sandblaster pen. A metallic endodontic ruler was used to aid in precisely adjusting the distance from the specimens. After completing air-abrasion, zirconia discs were cleaned again by the digital ultrasonic cleaner to eliminate any powder particles to guarantee good bonding. Each was then placed in a small numbered sealed plastic bag, which helped in randomization and blinding.



**Figure (1)**: Custom-made apparatus used for air abrasion. (Normal view); A: Horizontal metallic base, B: Sandblaster pen, C: Fixed vertical arm, D: Container filled with  $(A1<sub>2</sub>O<sub>3</sub>)$ , E: Plastic cylinder, F: Zirconia disc, G: Tack material, H: Endodontic metallic ruler, I: Sandblaster nozzle, J: Moving vertical arm with metallic screw.

#### **Randomization**

Two randomized sequences, by which zirconia discs and the prepared teeth were allocated to the tested groups, were generated using computer software (random.org), to ensure the unpredictability of the sequence generated. Sample allocation ratio was 1:1. Randomization ensured the elimination of any bias, that might affect the results' reliability.<sup>16</sup>

#### **Cementation procedures**

#### *Cementation of specimen in Group (CB)*

Calibra Bio cement capsule was activated and mixed following the manufacturer's instructions. Cement was extruded to the dentin

surface, then the zirconia disc was seated and stabilized in place by slight finger pressure. All excess cement was removed with micro-brush. The specimen was then transferred to a custommade loading device, through which a vertical uniform load of 5 kg was applied.<sup>17</sup> The specimen was left in the loading device for 4 minutes till complete cement setting.

#### *Cementation of specimens in Group (TH)*

TheraCem cement was directly applied to the exposed dentin surface then the air-abraded disc was seated and stabilized by slight finger pressure until the initial cement setting. All evident excess cement was removed as was previously mentioned. The specimen was then transferred to the custom-made loading device, tack cured using 1200 mw/cm<sup>2</sup> light curing device (3M ESPE, Elipar, S10 LED, USA) for 3 seconds to facilitate removing excess cement using a probe.<sup>11</sup> Final curing was then proceeded for 20 seconds at the disc peripheries to ensure full curing. After cementation completion, all specimens of both groups were stored at 100% humidity for 24 hours till thermocycling procedures.<sup>9</sup>

# **Thermocycling and Shear bond strength (SBS) testing procedure**

All cemented specimens were thermocycled (THE-1100, SD Mechatronik, Feldkirchen-Westerham, Deutschland) in distilled water for 5,000 cycles between 5°C and 55°C temperature with 60 seconds dwell time to simulate 6 months of intraoral service.<sup>10</sup> After thermocycling completion, each specimen was visually evaluated, where none of the specimens showed any signs of failure.

Specimens were then tested for shear bond strength using a universal testing machine (Model 3345; Instron Industrial Products, Norwood, USA) with a load cell of 5000 N, by a 0.5 mm mono-bevel chisel at a crosshead speed of 0.5 mm/min. until failure occurred.<sup>17</sup> Loads at failure were recorded in Newton, then converted into megapascal (MPa) using the following equation:

$$
\tau = P/\pi r^2
$$
  
Where: τ = shear bond strength (MPa)  
P = load at failure (N)  

$$
\pi = 3.14
$$
  
r = radius of zirconia disc (mm)

#### **Failure mode determination**  *Visual examination*

All failed specimens were visually inspected to determine the failure mode either; Adhesive (between disc and cement or between tooth structure and cement), cohesive (within cement) or mixed (combination of both).<sup>18</sup>

#### *SEM examination*

A scanning electron microscope (SEM Quanta 250, FEI Company, Eindhoven, Netherlands) was used to verify the failure mode. Both the dentin surface and the zirconia disc surfaces of failed specimens were scanned at magnification (800x, 1000x and 2000x), after being carbon sputter-coated.<sup>11,19</sup> An additional scanning of an extra air-abraded zirconia disc (manipulated similarly to the discs used in the present study) was scanned to serve as a reference when comparing the results.

#### *EDX analysis*

EDX analysis was performed (JCM-6000Plus versatile Benchtop SEM, Akishima, Tokyo, Japan) at  $250x$  after carbon sputtering<sup>19</sup> to analyze debonded surfaces' elemental composition. The extra air-abraded zirconia disc and custom-made discs of both cements were also scanned to serve as a reference when comparing the results.

#### **Statistical analysis**

The shear bond strength data (quantitative in nature) were analyzed by an expert statistician, who was blinded to the tested groups. Statistical analysis was performed using IBM® SPSS® Statistics Ver. 26 (IBM Corporation, NY, USA). Data were first explored

for normality, by checking the data distribution using Shapiro-Wilk test, and were found to be normally distributed, hence, were presented as mean and standard deviation (SD). Independent t-test was used to compare the tested groups, with the level of significance set to  $P \le 0.05$ .

#### **Results**

#### **Shear bond strength testing results**

The results showed insignificant differences between both groups  $(P-value > 0.05)$ , as presented in (**Table 2**).

#### **Failure mode:**

#### *Visual examination*

Visual examination revealed mixed failure mode for all specimens in both groups (**Figure 2**).

#### **Scanning electron microscope (SEM)**

**SEM examination of dentin surface of failed specimens:** In Group (CB), SEM images obtained at 800x and 1000x magnification, showed small islands of cement on dentin surface. Upon increasing the magnification to 2000x, it revealed blockage of some dentinal tubules with cement remnants. In Group (TH), SEM images obtained at 800x and 1000x magnification also showed small islands of cement on the dentin surface, however, they were smaller in size with more uniform distribution compared to Group (CB). Upon increasing the magnification to 2000x, cement particles appeared to cover more dentin surface compared to Group (CB) (**Figure 3**).

**SEM examination of zirconia surface of failed specimens:** In Group (CB), SEM images obtained at 800x and 1000x magnification, showed shallow surface cracks with sporadic rounded patches of cement on zirconia surface presented at the meeting point of the surface defects. Upon increasing the magnification to 2000x, it revealed small pinpoints cement remnants spread over the zirconia surface. In Group (TH), SEM images obtained at 800x and 1000x magnification, showed solitary islands of cement on zirconia surface with no evident surface defects compared to unbonded airabraded zirconia specimen. Both groups showed changes in microstructure with obliteration of the induced surface roughness. Upon increasing the SEM magnification to 2000x, it revealed more pinpoint remnants of cement spread over the zirconia surface compared to Group (CB) (**Figure 4**). The unbonded air-abraded zirconia surface showed shallow micro-irregularities interrupted by numerous irregular pores (**Figure 5**).

# **Energy dispersive x-ray spectroscopy (EDX) analysis**

EDX analysis of debonded specimens of both groups (CB, TH) showed changes in surface elemental compositions compared to the unbonded air-abraded zirconia disc where both groups showed an increase in carbon, calcium, silicon, sulfur and barium & decrease in oxygen, aluminum, zirconium and phosphorous constituents (**Table 3**).



# **Table (2): Results of shear bond strength testing**

*SD: Standard deviation, P-value: level of significance.*



Figure (2): Mixed failure mode seen in Group CB (a) and Group TH (b).



**Figure (3):** SEM images of dentin surface of failed samples; a: Group CB-800x, b: Group TH-800x, c: Group CB-1000x, d: Group TH-1000x, e: Group CB-2000x, f: Group TH-2000x.



**Figure (4):** SEM images of zirconia surface of failed samples; a: Group CB-800x, b: Group TH - 800x, c: Group CB-1000x, d: Group TH-1000x, e: Group CB-2000x, f: Group TH-2000x.



**Figure (5):** SEM images at 800x (a), 1000x (b) and 2000x (c) magnification of air-abraded zirconia.

<b>Average Atomic%</b>													
<b>Elements</b>	$\mathbf C$	$\Omega$	Al	Si	P	S	Ca	Zr	Ba	Be	Bi	Co	N
Group (TH)	65.04	26.71	1.42	5.14	0.65	0.36	0.32	0.25	0.11	$\Omega$	$\Omega$	$\boldsymbol{0}$	$\boldsymbol{0}$
Group (CB)	56.62	29.99	1.57	5.86	2.71	$\overline{0}$	2.73	0.41	0.08	$\theta$	$\theta$	$\mathbf{0}$	$\mathbf{0}$
Air-abraded zirconia disc	3.87	37.46	3.63	0.016	23.66	$\theta$	$\theta$	21.77	$\Omega$	9.71	0.08	0.11	0.52
Calibra Bio	14.67	48.35	15.64	4.39	$\Omega$	$\overline{0}$	9.81	$\theta$	$\Omega$	$\overline{0}$	$\theta$	$\overline{0}$	$\overline{0}$
<b>TheraCem</b>	32.8	45	1.92	14.37	0.63	$\theta$	1.36	$\boldsymbol{0}$	0.73	$\overline{0}$	$\mathbf{0}$	$\boldsymbol{0}$	$\boldsymbol{0}$
<b>Average Weight%</b>													
<b>Elements</b>	$\mathbf C$	$\mathbf 0$	Al	<b>Si</b>	P	S	Ca	Zr	Ba	Be	Bi	Co	N
Group (TH)	53.59	29.3	1.99	9.91	1.39	0.33	1.42	$1.1\,$	0.98	$\overline{0}$	$\overline{0}$	$\boldsymbol{0}$	$\boldsymbol{0}$
Group (CB)	42.27	29.81	2.63	10.23	5.26	$\theta$	6.92	2.32	0.63	$\Omega$	$\theta$	$\overline{0}$	$\overline{0}$
Air-abraded zirconia disc	1.87	43.74	4.6	0.01	17.92	$\Omega$	$\Omega$	53.14	$\Omega$	4.53	0.9	0.33	0.16
<b>Calibra Bio</b>	8.38	35.87	19.53	5.74	$\theta$	$\overline{0}$	18.21	$\overline{0}$	$\theta$	$\overline{0}$	$\overline{0}$	$\overline{0}$	$\boldsymbol{0}$
<b>TheraCem</b>	19.7	36	2.62	20.16	0.98	$\overline{0}$	2.77	$\Omega$	5	$\overline{0}$	$\overline{0}$	$\boldsymbol{0}$	$\overline{0}$

**Table (3): EDX analysis of debonded zirconia discs in Group (TH) and Group (CB), air-abraded zirconia disc and cements discs**

#### **Discussion**

The results of shear bond strength test failed to reject the null hypothesis, where the results showed statistically non-significant difference between both tested groups.

In the present study, super high translucent zirconia was used as it showed excellent mechanical and optical properties as well as wear and fatigue resistance. $20$  Its enhanced translucency allowed its use in highly esthetic areas.<sup>21</sup> The present study was in-vitro to provide simple, reliable, clinically relevant information about the properties and features of new materials or techniques without the need for animal involvement.<sup>22</sup> It also enable performing single-variable experiments with a tight control on experimental circumstances.<sup>22</sup>

Natural teeth were used to simulate the clinical substrates.<sup>23</sup> Maxillary first molars were chosen because they provided large surface area for bonding in comparison to other teeth.<sup>24</sup> Only teeth with no caries or cracks were used to avoid any confounding factor that might affect the final results.<sup>24</sup>

Teeth were flattened to expose dentin as the substrate, since most preparations for full coverage restorations involve bonding to dentin, which is considered a complex substrate due to its morphological and physical properties and variations. Its heterogeneous microstructure being composed of less mineral, more water and organic matrix makes it difficult to achieve a promising adhesive bond.<sup>25</sup>

Teeth flattening was done using precision saw to provide a flat surface devoid of any undercuts with high quality and minimum errors.<sup>19</sup> Teeth were finished with 600 grit abrasive paper to produce even surface without any irregularities which might affect the accuracy of the bond strength test, and to increase surface area for adhesion.<sup>26</sup>

All prepared teeth were individually mounted in an acrylic resin base to allow easy handling during cementation and testing procedures, in addition to allowing proper force application during testing to be directed at dentin-cement interface.

Zirconia discs were cut using precision saw to make use of its advantages previously mentioned and to standardize the dimensions of all discs in both groups. They were checked before and after sintering using the digital caliper to ensure their standard dimensions to avoid affecting the measured outcome (shear bond strength) which depended on the specimen diameter.<sup>27</sup>

Air-abrasion was employed as it is considered the simplest, most reliable, widely used method that was found to improve zirconia bond strength<sup>28</sup> by reducing the contact angles of the surfaces, promoting surface roughness and increasing surface energy.<sup>29</sup> Air-abrasion was performed according to the manufacturers' instructions aided by the custom-made holding apparatus to allow optimum surface roughness required for bonding and retention.<sup>29</sup>

Cementation was done following manufacturer's instructions to obtain optimum physical and mechanical properties of the tested cements. Custom-made cementation device was used to standardize the cementation procedures and the applied load as it is believed that the loading pressure can affect the cement adaptation, film thickness and the final strength; and consequently the shear bond strength. $30$  A 5kg load was used during cementation to simulate intraoral loading.<sup>17</sup> Excess cement was meticulously removed to avoid any error during the testing force application allowing the stylus of testing machine to be placed at the interface exactly.<sup>31</sup>

Thermocycling was performed to mimic the extreme conditions that restorations experience intraorally. It can clarify the effect of differences in thermal expansion coefficient between the tooth structure and the restorative materials, which could cause cement/ceramic interface degradation.<sup>32</sup>

Shear bond strength test was employed to test zirconia restoration bonding in the present study, because it is a widely used highly reliable method, offering ease of sample preparation, lower incidence of pre-test failure and simple testing protocol with large bonding area close to the clinical situation.<sup>33</sup>

After bond strength testing, debonded surfaces were evaluated for failure mode determination; visually and scanning electron

microscope (SEM), which is a powerful magnification tool that provides details of any solid surface with high resolution and magnification level reaching up to 10 nanometers, allowing obtaining the data in detailed digital three-dimensional and topographical imaging.34

Energy dispersive x-ray spectroscopy (EDX) was also used to help in failure mode assessment, where it provides a reproducible, reliable, and precise technique to identify and quantify major components of a surface.35

Regarding shear bond strength results: It showed insignificant difference, which might be attributed to the standardization of procedures among the tested groups. Although insignificant, Calibra Bio (bioactive) cement recorded higher mean bond strength values than TheraCem (MDP-containing) cement. This might be due to the increased amount of aluminum found in cured Calibra Bio cement compared to TheraCem, as was shown in Edx analysis, which is believed to enhance shear bond and compressive strength by forming three dimensional crosslinks.<sup>36</sup> It might also be due to the different nature of the cements tested, where TheraCem; being self-adhesive resin cements, the smear layer cannot be totally removed, what can adversely affect their bond.<sup>26</sup> Shear bond strength values of both groups were higher than the minimum acceptable range of bond strength (10-12 MPa) required for restoration survival in the oral environment<sup>37</sup> indicating the reliability of both cements.

Our results came in agreement with **Elattar et al. (2021)** who also found statistically insignificant difference between shear bond strength of bioactive cement (ACTIVA) and selfadhesive resin cement (Rely X Unicem) either with wet or dry dentin. $11$  However, our results disagreed with **Dandoulaki et al. (2019)**, who found that bond strength values of bioactive cement were below the acceptable range.<sup>38</sup> The difference might be due to testing different brand of cement (Ceramir) and different zirconia

material (BruxZir) with different composition than those used in our study.

With regards to the failure mode, all specimens in both groups showed visible mixed failure on both zirconia and dentin substrate surfaces. These findings were emphasized upon SEM examination. The Calibra Bio cement islands seen on the dentin substrate might be due to the ability of the bioactive cement to interact superficially with the tooth structure with minimal penetration in dentinal tubules.<sup>38</sup> This might be attributed to the essential reaction that occurs in the presence of water involving the dissolution mono-calcium aluminate and precipitation of other components [katoite  $(C_3AH_6)$  and gibbsite  $(AH_3)$ ] on contact points or areas and within the material itself leading to filling any gaps or voids, reducing the final porosities and enhancing the bond strength.6,39 It might also be due to the presence of nano particles within the cement that improved the cement flow and hybrid layer formation.<sup>40</sup>

TheraCem also showed small islands of cement on dentin surface; with smaller size and more uniform distribution covering more areas compared to Calibra Bio cement. This might be due presence of 10-MDP functional monomer in the cement, which have an affinity to hydroxyapatite, where it chemically bond to calcium ions of hydroxyapatite forming stable calcium phosphate and calcium-carboxylate salts, along with limited surface-decalcification and partial dissolution of the smear layer.39,41

Lack of bonding in some areas in both groups might be due to dentin high-water content that increased water fraction presence and interfered with complete cement polymerization.<sup>11</sup> The mixed mode of failure seen on zirconia surface was also emphasized by SEM, where patches of cements were seen in both groups. This might be attributed to airabrasion, which formed shallow microirregularities, increasing the surface area for micromechanical interlocking and cements flow. $42$ 

The difference between both groups might be due to the different composition of cements, with high affinity of MDP present in TheraCem cement to bond with aluminum oxide particles  $(Al_2O_3)$ .<sup>11,25</sup>

EDX showed changes in surface element compositions compared to unbonded air-abraded zirconia disc. This indicates the occurrence of chemical reaction between air-abraded zirconia disc and the tested cements especially bioactive cement (Calibra Bio) as it had higher concentration of these elements than that of MDP-containing resin cement (TheraCem).<sup>35</sup> It also justified the mixed failure mode seen in our results.

Limitations of the present study involved using teeth of unspecified age and lack of CAD/CAM machine milling of zirconia. It is recommended to conduct further studies testing the effect of bioactive cement on marginal adaptation, fracture resistance and color stability of different restorative materials. Also test the effect of extended aging parameters on shear bond strength of bioactive and MDP-containing cements are recommended.

# **Conclusions**

Within the limitations of the present study, the following can be concluded:

- 1. Bioactive Calibra Bio and MDPcontaining TheraCem cements showed comparable shear bond strength to (ML-S) zirconia after six-month clinical aging simulation.
- 2. Both bioactive and MDP-containing cements showed chemical and micromechanical interaction with zirconia and human dentin at varying proportions.
- 3. Bond strength of both tested cements was higher than the clinical acceptable values.

#### **Clinical implications**

Upon cementing super high translucent zirconia restorations to dentin, both bioactive Calibra Bio cement and MDP-containing TheraCem cement are considered a reliable in terms of bond strength.

#### **Conflicts of Interest**

The authors declare no competing interests.

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# **Ethics**

This study protocol was approved by the ethical committee of the faculty of dentistry- Cairo university on: 27/10/2020, approval number: (No.9-10-20).

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