A focus on zirconia: an in-vitro lifetime prediction of zirconia dental restorations

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CHAPTER 6

Fracture force and marginal adaptation of computer aided manufactured zirconia FPDs.

Keywords: all-ceramic, zirconia, CAD/CAM, fracture force, marginal adaptation
6.1 Abstract

Objective. The aim of this in-vitro study was to compare the fracture resistance and marginal adaptation of zirconia three-unit fixed partial dentures (FPDs).

Materials and Methods. Sixteen FPDs were fabricated from three different zirconia systems and corresponding ceramic veneering. Eight FPDs of each system were adhesively bonded or cemented. After artificial aging, the fracture resistance of the FPDs was determined. The marginal adaptation at the interfaces between cement-tooth and cement-crown was evaluated with scanning electron microscopy using replica specimens before and after aging.

Results. The three tested zirconia systems Cercon (1525 N), Digizon (1332 N) and Lava (1062 N) showed no significantly different fracture force with conventional cementation. The fracture force with adhesive bonding was about 60-90% lower when compared to conventional cementation. Prior to aging, all systems showed about 85%-95% perfect margins at the interfaces of cement-tooth or cement-crown. After aging, the interfaces of the conventionally cemented restorations and the adhesively bonded Lava FPDs deteriorated by about 5%.

Conclusion. Conventional cementation or adhesive bonding showed only limited influence on fracture resistance and the marginal adaptation of zirconia FPDs.
6.2 Introduction

High aesthetics and good biological compatibility, which are confirmed by long-term experience, recommend pressable all-ceramics for clinical application. However, a low fracture resistance restricts the indication of these all-ceramics to anterior indications and posterior crowns. The use of high-strength hot isostatic pressed (hip) or partly stabilized zirconia ceramics in combination with computer-aided manufacturing (CAM) allows for enlarging the indication of all-ceramics to replace posterior teeth and to fabricate longer span, tooth–coloured restorations. Zirconia has high fracture strength with a small range of strength variation and a high structural reliability compared to conventional dental ceramics [1, 2]. The fabrication concepts reach from manufacturing pre-sintered ceramics (Cercon, DeguDent, Germany; Lava, 3M Espe, Germany) to the milling of high-strength hipped zirconia (Digizon, Girrbach, Germany). All systems use CAM, but modelling of the restoration may follow a different philosophy, such as scanning a wax-up (Cercon) or computer-aided design (CAD-Lava, Digizon). For aesthetics, function and protection, the zirconia cores are veneered with modified conventional glass-ceramics in a layering or press technique.

Compared to press ceramics, where adhesive bonding is required for increasing the strength of the entire restoration [3], alternative (conventional) cementation is permitted for zirconia ceramics [4]. Strong differences in modulus of elasticity, film thickness, fracture toughness, compressive strength and others [5-7] were found between the available types of cements. All of them might influence the strength of a restoration, but e.g. finite element analysis [8] showed only small influence of film thickness or modulus on the stress of the restoration. Good bonding was found for zirconia with adhesive bonding [9, 10], but also some deterioration was described after in-vitro aging [11]. Conventional cementation of zirconia crowns showed comparable retentive strength compared to adhesive bonding [12]. Admittedly, a conventional cementation may be restrained due to the obtuse preparation angle applied in the data digitizing process. Loading under clinical conditions, as well as in-vitro mechanical and thermal loading or moisture deteriorate the strength of the zirconia and may decrease the marginal adaptation of the restoration. Scarce clinical reports of zirconia dental materials [13-15] are provided, and the long experience of zirconia in surgery allows no conclusions to the in-vivo behaviour in a dental application.

The aim of this investigation was to compare different competitive zirconia core materials with corresponding veneering after a simulated wearing period of five years. The influence of the cementation on the marginal adaptation and the fracture resistance were determined.
6.3 Materials and Methods

The roots of the human molars (n=96) were coated with a 1 mm thick layer of polyether material (Impregum, 3M Espe, Seefeld, Germany) to simulate human periodontium and then they were inserted into PMMA resin (Palapress Vario, Kulzer, Wehrheim, Germany) forming an oral gap of 10 mm. Loaded with 50 N, the layer allows a maximum mobility of the single tooth in axial and vertical directions of 0.1 mm. Human molars were used to ensure a clinically relevant modulus of elasticity of the abutments and to simulate a relevant bonding between fixed partial dentures (FPDs) and tooth. Varying dimension of the teeth was, therefore, accepted. All teeth were prepared according to the directives for ceramic restoration techniques using a 1 mm deep circular shoulder crown preparation. Sixteen FPDs of each zirconia material group listed in Table 6.1 were fabricated according to the manufacturer’s instructions.

The yttria-stabilized Cercon and Lava cores were milled in a pre-sintered ceramic condition and sintered to the final dimensions. Digizon FPDs were milled of a sintered, high-strength hipped zirconia ceramic. All zirconia frameworks were veneered with the corresponding veneering ceramics (Table 6.1).

Table 6.1: Materials, manufacturer, cementation and pre-treatment.

<table>
<thead>
<tr>
<th>Material/ Veneering</th>
<th>Manufacturer</th>
<th>Conventional Cementation</th>
<th>Adhesive Bonding</th>
<th>Zirconia Pre-treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lava/Lava Ceram</td>
<td>3M Espe, USA</td>
<td>Glass-ionomer Ketac Cem, 3M Espe, USA</td>
<td>Syntac classic/ Variolink2, Ivoclar-Vivadent, FL</td>
<td>Rocatec Soft, 3M Espe, USA</td>
</tr>
<tr>
<td>Digizon/GC Initial</td>
<td>Amman-Girrbach, Austria/GC, USA</td>
<td>Resin-modified Glass-ionomer: Fuji Plus, GC, USA</td>
<td></td>
<td>Al₂O₃ (110 µm, 2 bar)</td>
</tr>
<tr>
<td>Cercon/Cercon Ceram S</td>
<td>DeguDent, Germany</td>
<td>Zinc Phosphate: Harvard, Richter &amp; Hoffmann, Germany</td>
<td></td>
<td>Al₂O₃ (50µm, 2bar)</td>
</tr>
</tbody>
</table>

For comparing the type of cementation, eight FPDs of each group were adhesively luted with dual-curing composite, and eight FPDs were cemented with a conventional cement that was recommended by the manufacturer.

An artificial aging was performed to simulate a 5-year period of oral service. The loading parameters were [16] 1,200,000 mechanical loads with 50 N and a simultaneous thermal...
cycling with distilled water between 5°C and 55°C (3,000 times with 2 min each cycle). A human molar was adjusted as antagonist on the pontic of the FPD in a dental articulator (Amann-Girrbach, Koblach, Austria) and both tooth and FPD were transferred to the artificial simulator. Antagonist-tooth relation was controlled with an occlusal foil.

Fracture testing: After aging, all FPDs were loaded until failure using a testing machine (Zwick, Ulm, Germany, v = 1 mm/min). The force was applied using a steel ball (d = 12mm) while a tin foil (1 mm) between pontic and antagonist was used to prevent force peaks. The FPDs were optically examined before and after the fracture testing. Failure mode was divided into the fracture of the veneering or the core. Medians and 25%/75% of the fracture resistance [N] were calculated.

Marginal adaptation: For the semi-quantitative analysis of the marginal adaptation [17] both the cement-tooth- and the cement-restoration-interfaces were examined using the scanning electron microscope (SEM) Stereoscan 240 (Cambridge Instruments, Nußloch, Germany). Therefore, replicas (Epoxy VP 1031; Ivoclar-Vivadent, Schaan, Liechtenstein) of the approximal marginal areas distal of the replaced teeth of the FPDs were made before and after TCML by taking impressions (Permadyne, 3M Espe, Seefeld, Germany). The replica were prepared for the analysis in the SEM and the marginal areas were classified as „perfect margin“ (= smooth transition, no interruption of continuity) or „marginal gap“ (= separation of the components due to adhesive and/or cohesive failure) [18]. Statistical analysis was performed using Mann Whitney-U test (α=0.05).

### 6.4 Results

Fracture Force: The three tested zirconia systems Cercon (1525 N), Digizon (1332 N) and Lava (1062 N) showed no significantly different fracture force after conventional cementation. The median fracture forces with adhesive bonding were 1227 N (Cercon), 843 N (Digizon) and 992 N (Lava). Only Digizon had significantly higher fracture force (P=0.003) with conventional cementation compared to adhesive bonding. No failures occurred during TCML. The failure mode of all restorations after the facture test was a chipping of the veneering ceramic or a fracture of the zirconia core. No flamboyant differences were found between the failures of adhesive bonded or cemented FPDs: Lava FPDs showed two chippings and six core fractures when either adhesively bonded or conventionally cemented. The hipped zirconia Digizon provided two core and six chippings with adhesive bonding and three core fractures and five chippings with conventional cementation. Cercon had five core fractures and three chippings with resin bonding and five chippings and three core failures with zinc-phosphate cementation. Details are given in Table 6.2.
Table 6.2: Fracture force [N] of FPDs with different cementation (median, 25%/75%); number and type of failures; (ZnPh: Zincphosphate cement; RGIC: Resin modified glass-ionomer cement (GIC)).

<table>
<thead>
<tr>
<th>Material</th>
<th>Cementation</th>
<th>Fracture force [N] median</th>
<th>25%</th>
<th>75%</th>
<th>Failure mode: core/veneering [number]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Digizon</td>
<td>Adhesive</td>
<td>843</td>
<td>738</td>
<td>945</td>
<td>2/6</td>
</tr>
<tr>
<td></td>
<td>RGIC</td>
<td>1332</td>
<td>1131</td>
<td>1474</td>
<td>3/5</td>
</tr>
<tr>
<td>Lava</td>
<td>Adhesive</td>
<td>992</td>
<td>815</td>
<td>1596</td>
<td>6/2</td>
</tr>
<tr>
<td></td>
<td>GIC</td>
<td>1062</td>
<td>941</td>
<td>1146</td>
<td>6/2</td>
</tr>
<tr>
<td>Cercon</td>
<td>Adhesive</td>
<td>1227</td>
<td>1115</td>
<td>1467</td>
<td>5/3</td>
</tr>
<tr>
<td></td>
<td>ZnPh</td>
<td>1525</td>
<td>1323</td>
<td>1802</td>
<td>3/5</td>
</tr>
</tbody>
</table>

Marginal Adaptation: results and statistical information of the marginal adaptation are shown for the interfaces between cement-crown (Fig. 6.1) and cement-tooth (Fig. 6.2).
**Fig. 6.1:** Marginal adaptation (Perfect Margin (%)) at the interface: **Cement-Crown**

Above: conventional cementation; below: adhesive bonding (statistics: Mann-Whitney U-test, p=0.05; median, 25%/75%).

<table>
<thead>
<tr>
<th></th>
<th>Digizon</th>
<th>Lava</th>
</tr>
</thead>
<tbody>
<tr>
<td>Before/after TCML</td>
<td>adhesive</td>
<td>conventional</td>
</tr>
<tr>
<td>Lava</td>
<td>0.000/0.000</td>
<td>0.897/0.451</td>
</tr>
<tr>
<td>Cercon</td>
<td>0.151/0.287</td>
<td>0.752/0.202</td>
</tr>
</tbody>
</table>

The adhesively fixed Digizon and Cercon FPDs had a perfect margin of 100% at the interfaces, cement-tooth and cement-crown, before and after TCML. Only Lava showed significantly lower values (90%) before aging, which deteriorated further to about 85% perfect margin after TCML.
**Fig. 6.2:** Marginal adaptation (Perfect Margin (%)) at the interface: **Cement-Tooth**
Above: conventional cementation; below: adhesive bonding (statistics: Mann-Whitney U-test, p=0.05; median, 25%/75%).

<table>
<thead>
<tr>
<th>Before/after TCML</th>
<th>Digizon</th>
<th>Lava</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>adhesive</td>
<td>conventional</td>
</tr>
<tr>
<td>Lava</td>
<td>0.000/0.000</td>
<td>0.041/0.091</td>
</tr>
<tr>
<td>Cercon</td>
<td>0.951/0.861</td>
<td>0.752/0.597</td>
</tr>
</tbody>
</table>

Prior to aging, more than 95% perfect margins were found for all conventionally cemented FPDs, without significant differences among the three ceramics. TCML caused a max. 5% decrease of the median perfect margin for all materials, but at the cement-crown interface, Lava and Cercon FPDs showed a wider distribution of the results. At the cement-crown interface, a spreading between 85% (1st quartile) to 98% (2nd quartile) was found for conventionally cemented Cercon FPDs.
6.5 Discussion

The results of the zirconia restorations were twice and thrice as high as the values of comparable FPDs made of pressable ceramic (Empress2: 387 N) [19] and exceeded the loading requirements of 500 N for replacing posterior teeth [20]. The broad distribution of the fracture results restricts the significance of our results, but it represented the individuality of the clinically relevant FPDs. Differences in fracture resistance of the three zirconia materials with identical adhesive bonding may be attributed to the milling process (milling traces) or material properties (grain size distribution). The Digizon FPDs, which were milled of the hipped sintered ceramic, showed no advantages compared to the two other systems that were fabricated in pre-sintered state. Milling of high-strength hipped zirconia is supposedly for inducing cracks and superficial roughness, reducing the strength of the FPDs [21, 22]. For decreasing superficial stress, annealing of the cores is proposed [23] but not recommended by all manufacturers.

Adhesive bonding with resin-composite cement did not improve the fracture strength of the zirconia FPDs. In contrast, the median fracture force in tendency was higher with the conventional cementation. A reason may be a limited chemical conversion caused by restricted light activation of the dual curing composite cement through the opaque zirconia ceramic. It was found that not all resins polymerize adequately under different polymerization conditions [24]. It is supposed that hydrophilic conventional cements may show a better wetting capability of the zirconia surface. Phosphoric acids/monomer derivates were discussed for improving the wetability and bonding quality of the zirconia surface further [9, 10, 25]. Laboratory tests showed a higher bonding strength of zinc phosphate or glass ionomer compared to adhesive bonding [26], but pull-off tests of zirconia crowns demonstrated no different retentive strength using different types of cements [12]. Varying properties (Young’s modulus, compression strength etc. [5-7]) or bonding of the cement contribute to the fracture resistance of glass-ceramics [27], but an influence on the stability of high-strength zirconia may be excluded. Resilience of the cement may raise the mobility of the restoration, increasing torsion and the danger of fracture. In SEM marginal analysis, we found no failures in the matrix or between matrix and particles in glass-ionomers and micro-fractures of the zinc phosphate cement, both are damages which might contribute to a deterioration of especially highly loaded margins [5]. Polymerization stress, which is caused by thickness variations of the cement layer [28], is described, but we found sufficient marginal adaptation of the resin cement before and after TCML. An insufficient fitting of the FPDs as a result of the framework configuration [29] may cause resilience [30] and strength reduction in the long term. SEM analysis showed that the adaptation between the zirconia and cement was good, with 95% perfect margin. Nevertheless, a small decrease was found after
aging, indicating a deterioration of this interface. The adhesively bonded Lava restorations showed a lower but sufficient share of a perfect margin compared to the other zirconia systems. The application of alternative adhesive bonding systems may provide significantly better marginal adaptation [31]. Superficial ceramic treatments, such as hydrofluoric etching, do not activate the inert zirconia surface, whereas soft air- abrasion with small Al₂O₃-particles, tribochemical treatments [10] or pyrosil technologies [32] were supposed to improve bonding properties. The clinical consequences of the lower marginal adaptation were estimated as small, but the ultimate importance of this deterioration must be cleared in clinical tests. Caution is required when conical, low retentive preparation designs were used as contribute to the digitizing process. A separation of the interface may be supported under tensile load, e.g. taking off impressions. 
The results indicated that adhesive bonding, as required for other non-zirconia ceramic systems, is not necessary for the cementation of zirconia. An easy application of zirconia FPDs even under subgingival or moisture conditions with conventional cementation seems possible. The use of three different conventional cements confined the comparability of the results, although clinical relevance was improved using the recommended cements. Summarizing the results, all tested zirconia FPDs showed good to sufficient fracture resistance and marginal adaptation and might not be restricted for clinical application.
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