Use of zinc phosphate cement as a luting agent for Denzir™ copings: an in vitro study
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Abstract

Background: The clinical success rate with zinc phosphate cemented Procera crowns is high. The objective with this study was to determine whether CADCAM processed and zinc phosphate cemented Denzir copings would perform as well as zinc phosphate cemented Procera copings when tested in vitro in tension.

Methods: Twelve Procera copings and twenty-four Denzir copings were made. After the copings had been made, twelve of the Denzir copings were sandblasted on their internal surfaces. All copings were then cemented with zinc phosphate cement to carbon steel dies and transferred to water or artificial saliva. Two weeks after cementation, half of the samples were tested. The remaining samples were tested after one year in the storage medium. All tests were done in tension and evaluated with an ANOVA.

Results: Sandblasted and un-sandblasted Denzir copings performed as well as Procera copings. Storage in water or artificial saliva up to one year did not decrease the force needed to dislodge any of the coping groups. Three copings fractured during testing and one coping developed a crack during testing. The three complete fractures occurred in Procera copings, while the partly cracked coping was a Denzir coping.

Conclusion: No significant differences existed between the different material groups, and the retentive force increased rather than decreased with time. Fewer fractures occurred in Denzir copings, explained by the higher fracture toughness of the Denzir material. Based on good clinical results with zinc phosphate cemented Procera crowns, we foresee that zinc phosphate cement luted Denzir copings are likely to perform well clinically.

Background
CADCAM technologies have found increased use in dentistry during the past 15 years. Cerec, a system invented by Mörmann and Brandistini [1,2], was the first commercially available CADCAM system. Cerec was designed for making ceramic inlays and veneers, and these should be etched and bonded to the tooth with resin based luting agents [3,4]. Resin bonding was promoted because it improved retention and sealed gaps around Cerec restorations. Such gaps were often wider around the early Cerec restorations than they were around cast restorations. In addition, clinical experience evolving at that time suggested that the fracture rate of ceramic restorations decreased if they were resin bonded rather than cemented with traditional zinc phosphate or glass ionomer cements [5]. However, because of high equipment cost and a not yet...
During the past few years, ZrO₂ has been introduced to dentistry [14,15]. The partially stabilized ZrO₂ has a fracture toughness twice that of Al₂O₃ [16] suggesting that ZrO₂ based copings could become a major competitor to Procera in the future. One such ZrO₂ based system is the Decim system that makes ZrO₂ copings (Denzir™) by milling zirconium dioxide rods. These ZrO₂ copings, like the Procera copings, cannot be etched because of the acid resistance of ZrO₂. Although a resin-based cement such as Panavia is the recommended luting agent for Denzir at the present time, there is an interest in determining whether zinc phosphate and glass ionomer cements are acceptable alternatives. That interest relates primarily to properties such as simplicity of use, easiness of removing excess from marginal regions after cementation, and last, but not least important, easiness of removing a previously cemented crown if so needed. As we know from the previously quoted Procera study [13], 6.5 % of the crowns were remade because of coping/dental porcelain fractures and caries (6.5%). Another 5% suffered from acceptable chipping [13]. These findings are important, because they suggest that fractures and caries may require removal of the ceramic restoration. If the coping is well bonded to the tooth surface, the old unit must be cut away. Such a removal is not easy to do with strong ceramics, and there is a potential risk that incomplete cooling during cutting could cause pulp irritations. Because of the latter aspects, a clinically important question to address is whether zinc phosphate cemented Denzir crowns compare as well clinically regarding low ceramic fractures as resin based cemented Denzir crowns do. However, before such clinical trials can be justified ethically, in vitro tests must prove that the retention of Denzir crowns is as good as that of Procera crowns. If the retention of Denzir is as good as that of Procera, one would expect that Denzir crowns will

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provide as good or even better clinical results than those reported with zinc phosphate or glass ionomer cemented Procera crowns [11,13].

Because of the above considerations, the objectives of this study was to determine in vitro whether Denzir copings cemented with zinc phosphate cement to metal dies could provide as good retention strength as Procera crowns cemented to similar metal dies. We also wanted to determine if sandblasting would improve the retention of the Denzir copings, or if retention over time would behave differently if the cemented crowns were stored in water or artificial saliva.

**Methods**

**Metal dies**

Thirty-six metal dies were machined out of carbon steel to dimensions shown in Figure 1. During the machining procedure all the surfaces to which the zinc phosphate cement would be attached were finished to roughness values around 6.3 µm. The reason we used the 6.3 µm surface roughness was that a preliminary evaluation of dies with surface roughness values of 3.2, 6.3, 8.0 and 12.5 µm had revealed that a surface roughness value of 6.3 µm was ideal for our study. With such surface texture, the cement did not separate from the cement-model surface. Instead it fractured within the cement or at the crown-cement interface.

To verify the surface roughness values, the finished die surfaces were recorded with a profilometer (Federal Surf analyzer System 5000, Federal Products Co, Providence, RI). The surface roughness value, Rs, represents the arithmetic average of the absolute values of the measured roughness profile height deviations taken within the scanned length and measured from the mean line. These scanned recordings were made in a cervical to occlusal direction over a length of 3 mm on each metal die. The surface roughness value for that distance was then used to determine the average value for all the dies.

**Impressions and gypsum dies**

Before impressions were made of the metal dies, a 1.6 mm thick ring was inserted and located to the marginal part of the simulated crown preparation (Figure 2). Impressions were then made in a polyvinylsiloxane impression material (Light Body, President, Coltène AG, Altstätten, Switzerland) supported by an individual tray. The tray had been covered with an adhesive to secure a reliable tray-impression attachment, and the space between the tray and the die was 2 mm. One hour after the impression had been made it was poured with a Type IV gypsum (Silky-Rock, Whip-Mix Corporation, Louisville, KY) and allowed to set during the night. After impression removal and die inspection, the 36 gypsum dies were sent to laboratories making Denzir and Procera copings.

**Ceramic copings**

Twenty-four Denzir and twelve Procera copings were ordered from Denzir and Procera certified laboratories. All ceramic copings were made 0.6 mm thick and with a cement space corresponding to 60 µm. That spacing started 0.8 mm from the cervical margin and reached its maximal...
thickness after 1.2 mm from that margin. When the copings arrived from the laboratories, all 36 copings were checked regarding their fit.

**Sandblasting**

Twelve of the Denzir copings were sandblasted on the inner surfaces with Al₂O₃ (particle size = 50 µm) using an air pressure of 2 bars (200 kPa). The sandblasting process was done with the sandblasting tip located at a distance of 10 mm from the ceramic surface. The centre of the sandblasting stream targeted the transition from the occlusal to the proximal inner surfaces. The entire inner surface was then sandblasted by rotating the coping four times, each time 90 degrees. Each of these locations was sandblasted for 5 s.

**Inner surface roughness**

The inside of each coping was scanned with the profilometer. The scans were collected within the 0.5 to 1.5 mm interval from the cervical margin. From these scans the R₄ values were calculated.

**Cementation**

The 1.6 mm ring, located in the cervical region of the preparation when the silicone impression was made was removed and replaced with a 1.55 mm thick washer with an outer diameter of 18 mm (Figure 3). A zinc phosphate cement (Phosphate Cement, Heraeus Kulzer, Dormagen, Germany) was mixed on a room tempered glass plate. For each portion, 1.2 g powder was mixed with 0.5 mL liquid. The powder was divided into six portions (two 1/16th, one 1/8th, and three 1/4th portions). First, one 1/16th portion was mixed for 10 s, then the second 1/16th portion for 10 s, followed by the 1/8th portion for another 10 s. A 1/4th portion was then added and mixed for 15 s followed by another 1/4th portion, also mixed for 15 s. The final 1/4th was then added and mixed for 30 s. Thus, a total mixing time of 1 min and 30 s was used. The mixed cement was then placed inside the ceramic coping, which was rotated 90 degrees as the coping was seated on the metal die. Thirty seconds after completed mixing, a load of 2 N was placed on the crown, and the load acted on the crown for 5 min. Excess material was removed after 7.5 min counted from the time the coping was loaded with the 2 N load.

**Retention force**

Fourteen days after cementation, half of the specimens (3 Denzir as received, 3 Denzir being sandblasted, and 3 Procera, all stored in distilled water; and 3 Denzir as received, 3 Denzir being sandblasted, and 3 Procera, all stored in artificial saliva) were tested in tension (Figure 4) until failure using a specially designed testing device in an Instron Universal Testing machine at a load rate of 0.5 mm/min. After one year (3 Denzir as received, 3 Denzir being sandblasted, and 3 Procera, all stored in distilled
water; and 3 Denzir as received, 3 Denzir being sandblast-ed, and 3 Procera, all stored in artificial saliva), the re-maining 18 specimens were also tested as described earlier.

Fifteen minutes after the initiation of the cementation process the cemented copings with the steel dies and washers were transferred to distilled water or artificial saliva and then stored in an oven at 37°C. The artificial saliva [17] was of the following composition: 0.1 L each of 25 mM K2HPO4, 24 mM Na2HPO4, 150 mM KHCO3, 100 mM NaCl, and 1.5 mM MgCl2. To this were added 0.006 L of 25 mM citric acid and 0.1 L of 15 mM CaCl2. The pH was then adjusted to 6.7 with NaOH or HCl and the volume made up to 1 L. To avoid bacterial growth, we added 0.05% by weight thymol to the artificial saliva. All chemicals were ACS-grade (American Chemical Society).

Statistical evaluation
The force values, needed to dislodge the copings, were used for the statistical evaluation. One-way and two-way ANOVA:s were used to determine significant differences between materials, storage medium and storage time as well as interactions of these (ANOVA, SAS Institute, Cary, NC, USA). Comparisons between the individual groups were also conducted using Duncan’s multiple range tests. All test were conducted on the 95% significance level.

Results
Metal dies
The profilometer readings of the metal die surfaces gave an average surface roughness value (R_a) of 5.49 ± 0.98 µm. These roughness values were primarily based on wave shaped surface where the distance between the peaks was around 200 µm and where the main peak-to-main valley distance was around 20 µm (Figure 5).

Ceramic copings and effect of sandblasting
The surface roughness values of the insides of the different coping groups are shown in Table 1. No significant differ-ence existed between the three combinations (p = 0.2239).

Retention force
The statistical analysis revealed that the most important factor affecting the retention force was storage time (Ta-bles 2 and 5). There was no difference between the two main materials or whether the Denzir copings had been sandblasted or not (Table 3).

Comparing the storage media could not prove whether such a difference existed (p = 0.082) (Table 4). In this comparison, no consideration was taken for the different material groups and storage times. When storage time only was compared there was a significant increase in re-tention force with time (Table 5).

As seen in the Tables 6, 7 and 8, there are large differences among the different test groups (standard deviation ~30% of the mean value). From Table 2, we can also see that there are no significant interactions, although the material/storage and storage/time interactions are pretty close.

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Table 1: R_a mean values (Mean) and standard deviations (SD) for the different coping materials expressed in microns.

| Material | Treatment   | Mean ± SD  |
|----------|-------------|------------|
| Denzir   | Sandblasted | 2.01 ± 0.65|
| Denzir   | Untreated   | 2.13 ± 0.52|
| Procera  | Untreated   | 2.47 ± 0.76|

Table 2: Results of ANOVA evaluation

| Source          | F  | Anova SS | Mean Square | F Value | Pr>F  |
|-----------------|----|----------|-------------|---------|-------|
| MATER           | 2  | 261306.722| 130653.361  | 0.29    | 0.7522|
| STORAGE         | 1  | 1827002.778| 1827002.778| 4.03    | 0.0553|
| TIME            | 1  | 2628721.778| 2628721.778| 5.79    | 0.0235|
| MATER*STORAGE   | 2  | 2620642.389| 1310321.194| 2.89    | 0.0737|
| MATER*TIME      | 2  | 299347.722 | 149673.861  | 0.33    | 0.7220|
| STORAGE*TIME    | 1  | 1814409.000| 1814409.000| 4.00    | 0.0561|
Figure 4
The die with the cemented coping was inserted into a specially designed testing device (left drawing). The washer was located under the horizontal top bar shown above and the die protruded through that bar. A metal pin (P) was inserted through a metal band and a hole drilled through the die. That attachment is shown in the central drawing. The 300.0 mm long metal band was attached to the universal testing machine that generated a recordable force (in the direction of the arrow shown in the figure). The metal band and the attachments are shown in the reduced drawing to the right.
Of the Procera copings, two copings fractured during testing after 14 days of storage and one coping fractured after one year. Of all tested Denzir copings, not a single coping fractured. However, careful inspection with transilluminating light revealed that one of the Denzir copings tested after 1 year had a crack that extended from the cervical region to the occlusal region.

**Discussion**

**Effect of coping composition and sandblasting**

Table 1 shows that there was no significant difference in surface roughness between the three evaluated ceramic coping groups. The low value of the sandblasted Denzir copings suggest that the machining process generated a surface roughness that was at least as rough as a machined and sandblasted Denzir surface. Because of these findings, sandblasting conducted under the conditions evaluated in this study is not recommended for Denzir copings.

**Effect of storage**

The statistical evaluation of variables such as material, storage and time as well as interactions of these variables revealed that storage time was significant regarding retention force (Table 2). Storage medium and interaction between time and storage medium were almost significant on the 95% significance level.

There was no difference between the three material groups regarding retentive force (Table 3). That finding most likely relates to the similarities in surface roughness values among the three groups (Table 1). The similarities in retentiveness among the three material groups are important. Because of the published success rate of Procera after 10 years in clinical service [13], our *in vitro* results suggest that Denzir copings, sandblasted or not, and cemented with zinc phosphate cement are likely to perform equally well as Procera crowns, at least regarding retention.

Based on the lower fracture frequency identified among the Denzir copings, our findings suggest that Denzir copings might perform even better than Procera crowns. Of the twelve tested Procera copings, three complete fractures occurred in the Procera copings, while of the twenty-four tested Denzir copings only one had a detectable crack that did not even result in a clear fracture during testing. At the present time, though, one cannot exclude that these differences are coincidental. However, the higher fracture toughness of Denzir, almost twice as high as that of Procera, probably explains the lower fracture tendency of Denzir identified in this study.
Future studies regarding CADCAM technologies need to focus on flaw formation that might be induced during manufacturing. One may suspect that a milling process like the one used to make the Denzir copings, induce more flaws than a pressing and sintering technique as the one used to make Procera copings. However, there is no proof available supporting that assumption at the present time. In the case of Procera, one cannot exclude the possibility that flaws are induced when copings are pressed and that these flaws may not heal completely during sintering. Besides, during sintering and cooling, thermal stresses may be induced that trigger crack formation in the future.

Table 3: Mean retentive forces (Mean) and standard deviations (SD) of the different material groups (DECBL = Denzir sandblasted; DECUNBL = un-sandblasted; PROCERA) expressed in Newtons. The values are based on pooling the values generated at the two storage times and the two storage media.

| Material  | Mean ± SD       | N  | Duncan Grouping |
|-----------|-----------------|----|-----------------|
| DECUNBL   | 1870.4 ± 683.2  | 12 | A               |
| DECBL     | 1733.8 ± 1044.7 | 12 | A               |
| PROCERA   | 1665.5 ± 591.4  | 12 | A               |

N = number of samples

Table 4: Mean retentive forces (Mean) and standard deviations (SD) of the different storage groups (AS = artificial saliva; WATER) expressed in Newtons. The values generated by pooling the material group values and the two storage times.

| Storage | Mean ± SD       | N  | Duncan Grouping |
|---------|-----------------|----|-----------------|
| AS      | 1981.8 ± 886.4  | 18 | A               |
| WATER   | 1531.3 ± 597.3  | 18 | A               |

N = number of samples

Table 5: Mean retentive forces (Mean) and standard deviations (SD) of the two different time groups (Months) expressed in Newtons. The values are based on pooling the material groups and storage media results for the two time groups.

| Months | Mean ± SD       | N  | Duncan Grouping |
|--------|-----------------|----|-----------------|
| 0.5    | 1486.3 ± 547.7  | 18 | B               |
| 12     | 2026.8 ± 891.8  | 18 | A               |

N = number of samples

Table 6: Mean retention forces (Mean) and standard deviations (SD) for the different material groups (DECBL = Denzir sandblasted; DECUNBL = un-sandblasted; PROCERA) and storage groups (AS = artificial saliva; WATER) expressed in Newtons. The results are based on pooling the values for the two storage times.

| Material  | Storage | N  | Mean ± SD |
|-----------|---------|----|-----------|
| DECBL     | AS      | 6  | 2184.2 ± 1313.8 |
| DECBL     | WATER   | 6  | 1283.3 ± 433.7  |
| DECUNBL   | AS      | 6  | 1716.3 ± 728.1  |
| DECUNBL   | WATER   | 6  | 2024.5 ± 663.3  |
| PROCERA   | AS      | 6  | 2045.0 ± 526.0  |
| PROCERA   | WATER   | 6  | 1286.0 ± 383.6  |

N = number of samples
From the above argumentation, flaws may very well be induced during manufacturing of both Denzir and Procera copings. Thus, differences in either fracture toughness or flaw sizes/densities, or a combination of the two, would explain why the Procera copings had higher fracture tendency. The higher fracture toughness of zirconia favours Denzir and would explain the lower fracture frequency seen in these copings. However, whether the flaws introduced in Denzir copings are smaller or bigger than those present in Procera copings is not known and needs to be investigated further. Flaw formation during manufacturing becomes very important when we compare different zirconia crowns that now are available on the market. Some of them are made by milling industrially sintered and processed zirconia, while other are made by milling presintered zirconia that is then sintered.

Storage of the copings in artificial saliva resulted in force values almost significantly higher than those stored in water (Table 4). A possible explanation is that some of the ions, for example phosphate ions, diffused into the cement and pushed the setting reaction toward an increased precipitation reaction. Such an explanation can be related to the setting reaction of zinc phosphate cements. As the storage time increased, the required force needed to dislodge the copings also increased (Table 5). A likely explanation is that as time passed the setting reaction became more complete. There is also a possibility that corrosion of the steel dies and release of iron ions from the dies affected the setting reaction of the zinc phosphate cement. Such a corrosion process might also have increased the surface roughness at the cement-dye interface and thereby also increased the mechanical retention.

Even though time improved the retention of the cemented copings, one should not extrapolate that value to the clinical situation. Clinically, the coping would be exposed to different loads during the entire observation time. In our study, no such forces acted on the cemented coping from time of cementation to time of testing. However, the improved results with time shows that storage media such as water and artificial saliva by themselves do not decrease the retention force. This finding is important, because it implies that other factors are more important when we try to explain why the retention of zinc phosphate cemented

| Material  | Months | N  | Mean ± SD       |
|-----------|--------|----|----------------|
| DECBL     | 0.5    | 6  | 1358.8 ± 486.5 |
| DECBL     | 12     | 6  | 2108.7 ± 1351.6|
| DECUNBL   | 0.5    | 6  | 1587.3 ± 558.8 |
| DECUNBL   | 12     | 6  | 2153.5 ± 722.8 |
| PROCERA   | 0.5    | 6  | 1512.8 ± 662.2 |
| PROCERA   | 12     | 6  | 1818.2 ± 524.5 |

N = number of samples

| Storage | Time | N  | Mean ± SD       |
|---------|------|----|----------------|
| AS      | 0.5  | 9  | 1487.1 ± 521.6 |
| AS      | 12   | 9  | 2476.6 ± 920.2 |
| WATER   | 0.5  | 9  | 1485.6 ± 604.5 |
| WATER   | 12   | 9  | 1577.0 ± 622.9 |

N = number of samples
crowns sometimes fail over time. Loading conditions and fracture toughness of the luting agent are such factors.

Our results suggest that a clinical evaluation of Denzir crowns cemented with zinc phosphate cement are likely to perform as well as Procera crowns cemented with zinc phosphate cement. However, based on Burke et al.’s [5] review, which supported the use of resin cements, one can question the rational of even considering using zinc phosphate cement as a luting agent in a clinical study. There are at least two reasons justifying such a clinical study. First, by assuming that the high success rate of zinc phosphate cemented Procera crowns is likely to be equally high with Denzir copings, justifies such a study ethically. Second, the simplicity of using zinc phosphate cements, their ease of removal from marginal regions after setting, and the ease with which a zinc phosphate cemented crown can be removed if remake is needed, are beneficial clinical advantages that cannot be neglected.

Having justified the use of zinc phosphate cement in a clinical study, it is also important to emphasize that such an evaluation should consider retention and solubility of the luting agent too. In the Procera study conducted by Ödman and Andersson [13], retention failures requiring recementation were not included in their impressive success rate. Present evidences suggest that resin bonding improves the results with ceramic restorations [5], even though these claims are not conclusive [18,19]. There is no doubt that retention is an important factor to consider, but one must also accept that strong bonding can also be a drawback if the crown needs to be removed. In the latter case, a well-bonded ceramic restoration can be a bigger clinical challenge than the need for recementing a less well-bonded restoration.

One often hears the claim that resin cements decrease the fracture frequency of ceramics. Such a claim is justified for some ceramic systems, but is may not be valid when we are dealing with high strength ceramic copings like the ones used in both Procera and Decim. Instead, some clinical studies dealing zinc phosphate cemented alumina copings are so good that one can question whether resin bonded copings will outperform these results. It is first when comparative studies take all these pros and cons into consideration, as we know whether resin bonded alumina or zirconia copings outperform zinc phosphate cemented alumina or zirconia copings.

Conclusions
Denzir copings, cemented with zinc phosphate cement to steel dies, perform at least as well as Procera copings cemented with the same zinc phosphate after storage in water and artificial saliva for one year when tested in vitro. The use of sandblasting under the conditions given in this study does not enhance the internal surface roughness or the retentiveness of the Denzir copings. During a one-year storage time in water or artificial saliva, the retentiveness did not decrease. Instead, the retentiveness of the samples increased.

Competing interests
Decim AB, Skellefteå, Sweden, funded this study. The contract was supervised by the University of Florida through University Project Number 00032218 and covered all expenses associated with this project.

Authors’ contributions
Decim supplied us with the steel dies. Mr. E Mondragon made the impressions and the stone dies. A Procera laboratory and a Decim laboratory made the copings. Mrs. I Garcea did the surface measurements and prepared the artificial saliva. Dr. K-JM Söderholm contributed with all other research components related to this project (experimental design, sandblasting, cementation, testing, statistical evaluation and final report).

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