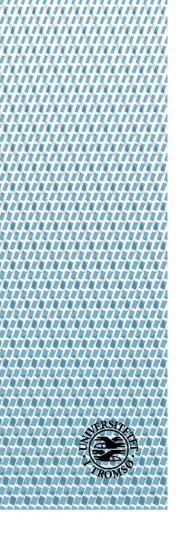


Institutt for klinisk odontologi, Det helsevitenskapelige fakultet

Fracture resistance of monolithic zirconia crowns: The importance of the compressive strength of the dental cements used.

John Magnus Nergård, Solveig J. Lægreid Veiledere: Ulf Örtengren, Keisuke Nakamura Masteroppgave i Odontologi Mai 2014



ABSTRACT

Background: Monolithic zirconia crowns have not been used for a very long period in dentistry even though zirconia with veneering porcelain, as crown and bridge material has been used for several years with great clinical success. Several different luting materials have been purposed for zirconia but concerning monolithic zirconia the knowledge of the influence of the cement seem to be limited. The aim of the study was to investigate three different cements (one phosphate, one self adhesive and one dual cured resin based cement) and their influence on fracture strength of monolithic zirconia crowns in vitro with the null hypothesis tested that the compression strength of the cement would have no statistical influence on the fracture strength of the monolithic zirconia crowns tested.

Materials and methods: Eighteen CAD-CAM produced monolithic zirconia crowns (Th=0.5 mm) divided into 3 groups (n=6) and cemented with 3 different cements were tested. In advance the Poissons ratio, modulus and flexural strength of the composite abutment used, fit of the crowns and their quality was evaluated.

Two tests were then conducted for the cement study: Compressive strength of the cements used were tested and recorded as well as load to failure test. The 3 groups of crowns cemented with the three different cements were tested until fracture after water storage for 24 hours at 37°C±1. The tests were done with guidance of ISO 9917:1 and comparative test procedures for load to failure test. Light microscope and SEM analysis were conducted and differences between the groups tested were statistically evaluated. Results and conclusion: The composite abutment showed similar Poissons ratio and modulus values as wet dentin. Even though the phosphate cement showed significantly less compressive strength, no significant difference in load to failure for the crowns tested were found. The compressive strength of the cement used seemed to be of no importance for the fracture resistance of monolithic zirconia crowns.

INTRODUCTION

Zirconia (stabilized zirconium dioxide (ZrO_2)) has become widely used as a dental ceramic material for full coverage crowns (1). All-ceramic constructions initially gained popularity due to both their biological and aesthetic properties. Zirconia is an oxide ceramic and natural compound of the element zirconium that occurs in nature (Fig 1). It has a principal crystalline phase, has high fracture toughness and is considered as biocompatible. It is currently the strongest available dental ceramic (2 , 3).

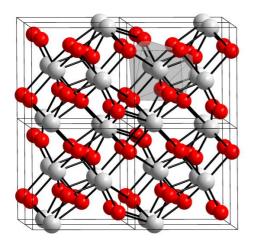


Figure 1: The three-dimensional structure of zirconia, white being zirconium and red being oxygen $\binom{4}{}$

Pure zirconia is monoclinic in its structure at room temperature, and adopts a tetragonal structure during sintering at high temperatures. During cooling there is a transition from the tetragonal to the monoclinic phase $(^{5,6})$. This behavior will create a volume expansion, inducing high compression stresses. These stresses make the material brittle. To stabilize the crystalline phase small amounts of yttria (Y_2O_3) , magnesia (MgO), calcia (CaO) or ceria (CeO) are added. Addition of these oxides fully or partially suppresses the transformation from the tetragonal to the monoclinic structure during cooling after sintering, thereby inhibiting crack formation $(^{7,8})$. This will make the mechanical behavior controllable and desirable. When a crack occurs in the material, the tetragonal phase close to the crack undergoes transformation to a monocline phase due to the stress initiated by the crack formation. The transformation will give a volume expansion of the crystals due to the phase transformation resulting in the generation of compression stress around the crack that will obstruct further crack propagation (i.e. transformation toughening) $(^{9,10})$.

Even though zirconia has a high fracture toughness on its own, the preparation of the tooth is still of importance for the stress state within the crown-tooth complex (11). An adequate preparation will give higher mechanical retention to the zirconia crown and decrease the risk of breakage, especially in the posterior part where the bite forces are higher. Eliminating sharp edges will also help in reducing the risk for cracks in the crown. Recommendations are therefore given by the manufacturers concerning preparation and minimal requirements concerning removal of tooth substance.

Until recently, ceramic crowns made of zirconia were constructed with an inner core of zirconia and an outer layer with sintered porcelain as for ordinary metal-ceramic (MC) crowns. This will give the crown a good aesthetic appearance together with an assumed high mechanical strength (¹²). The problem, however, has been fractures in the veneering porcelain due to adhesion difficulties between zirconia and the veneering material (^{13,14}). During the last years manufacturers have developed a monolithic zirconia for fixed dental prosthesis (FDP) system utilizing a tooth-colored zirconia. In this system, full ceramic restorations of zirconia without the veneering porcelain are fabricated using a dental CAD/CAM system for the process in total without the adhesion difficulties between the zirconia and the veneering porcelain. The aesthetic properties of monolithic zirconia are however poorer as compared to zirconia cores with veneering porcelain. This may be due to the fact that monolithic zirconia crowns are fabricated as one homogenous color (¹²).

Different cements on the market such as resin-based cements have been recommended for cementation of monolithic zirconia crowns for improved marginal aesthetics and increased strength (^{15,16}). Ceramics based on zirconia are considered difficult to treat for an optimal micromechanical adhesion to composite resin-based cement because of the oxidic structure (^{17,18}). Sandblasting has been recommended as a suitable method (¹⁹). Cements containing monomers with phosphate groups or equivalent can be used to achieve chemical adhesion. Still, zirconia FDPs rely to a large extent on the macro mechanical retention (i.e preparation design) and is often recommended by the manufacturer to be cemented to the tooth using conventional cement (i.e. zinc phosphate or glass-ionomer cement).

Ceramics, including zirconia are considered brittle materials even though zirconia will achieve high strength due to transformation toughening, as written, $(^{20,9})$. Therefore, it is suggested that the cement by its compression strength will support the reconstruction $(^{21})$.

To achieve mechanical support for reconstructions made of ceramics both oxidic (e.g. zirconia) and especially silica based (e.g. glass-ceramics) often polymer resin-based cements are used. Polymer resin-based cements initially gained popularity due to their mechanical properties, adhesion due to the acid-etch technique to enamel and dentin, and low solubility (²²).

Panavia (Kuraray, Japan) as an example of polymer resin-based cement is well-used and studied. Its self-etching primer system combines micromechanical adhesion and chemical adhesion to both the tooth substance and the inner surface of the ceramic (^{23,24}). The latter have also been supported scientifically and is due to a methacrylate monomer within the cement that contain phosphate bonds and has therefore an ability to chemically bond to inorganic molecules (²⁵). It has also been reported as a suitable cement in clinical studies (^{26,27,28}). It is methacrylate based and contains both chemical and light initiators (e.g. dual cure).

RelyX unicem (3M/ESPE) is a dual-cure dental cement. It is claimed to be a self-adhesive cement based on polymer resin with modified glass-ionomer properties (²⁹). Resin modified glass-ionomers are formed by replacing part of the polyacrylic acid in conventional glass-ionomer-cements with hydrophilic methacrylate monomers (³⁰). During the mixing, the pH will fall to approximately 2, making the material acidic. This makes the cement demineralize the tooth as for acid etch and creating a surface with both micro mechanical and chemical bonding features. During the procedure, the pH will rise because of formation of water, thus neutralizing the cement and also making the ion-movement of the glass-ionomer reaction possible. The result is cement with hydrophobic features, making it more able to resist water uptake from the oral environment (³¹).

Zinc phosphate dental cement is one of the oldest and most widely used and studied cements, and is commonly used for luting permanent restorations with good macro-mechanical retention (^{31,32}). Zinc phosphate cement has a well-documented longevity and is used for cementation of for example gold inlays/onlays, MC-crowns and bridges. It consists of a powder, zinc oxide with a 2-10% additive of magnesium oxide, and a liquid consisting principally of phosphoric acid, water, and buffers. During the setting, the reaction between positive zinc ions and negative phosphate groups results in a crisp cement with a reasonable compression strength (³³).

All of the cements mentioned are recommended by the different manufacturers to be used in cementation of zirconia FDPs. Even though adhesion has been brought to attention, knowledge of the influence of compression strength of the cement for fracture strength on zirconia FDPs, especially monolithic zirconia crowns, seems limited to the knowledge of the authors. To support zirconia, as a ceramic material, the compressive strength of the cement may be of importance to give the tooth-cement-crown complex the ability to withstand higher forces.

The purpose of the present study was, therefore, to investigate three different cements and their influence on fracture strength of monolithic zirconia crowns in vitro.

The null hypothesis of the present study is that the compression strength of the cement would have no influence on the fracture strength of the monolithic zirconia crowns tested.

MATERIALS AND METHOD

Preparation of abutments and crowns

A plastic model of a right mandibular first molar (46) (A5A-500, NISSIN, Kyoto, Japan) was prepared by one operator according to a preparation protocol made by Nakamura et al (³⁴) at Tohoku university/Japan for single crowns made of zirconia (Fig. 2).

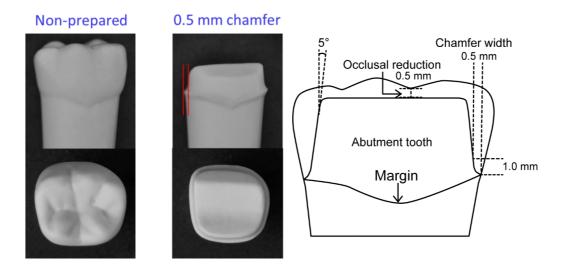


Figure 2. a) Non-prepared and prepared tooth models. b) Schematic drawing of abutment reduction and crown

The preparation protocol followed descriptions for all-ceramic crowns made for in-vitro testing according to Kelly (³⁵). The protocol was finally established after a pilot study made by Tohoku University. One tooth crown was manually prepared with a cervical chamfer shaped preparation line and a V-shape of the occlusal surface with an angle of 135±2°C. The chamfer width and the reduction of the occlusal surface were both 0.5 mm. The taper of the tooth abutment model was finally finished using a milling machine (F3 ergo, DeguDent GmbH, Hanau-Wolfgang, Germany) to be 5°. The roughly prepared tooth abutment model was scanned by a dental CAD system (Lava Scan ST, 3M/ESPE, St Paul, US). The dimensions of the prepared tooth abutment model were measured using the software of the CAD system after scanning. The measurements and fine adjustments of the abutment models were repeated until the defined reduction was obtained and the final model was used as a master model. Subsequently, the master model was scanned and replicas were milled from hybrid polymer resin-based block (Lava Ultimate, 3M/ESPE; St Paul, US) using a dental CAM system (Lava system, 3M/ESPE; St Paul, US).

It has been reported that the modulus of elasticity of abutment teeth can affect the results of strength test and should be in agreement with the modulus of dentin (36,37,38). Therefore, the modulus of elasticity of the polymer resin used was measured according to ISO 10477:2004, Dentistry – Polymer-based crown and bridge materials (MOD). The test was performed in advance by Tohoku University using a three-point bending test equipment (AG-IS Universal testing machine, Shimadzu, Japan). In addition a test for evaluation of Poisson's ratio of the abutment material was performed at Tromsø University. Six rods of the abutment material were prepared and polished until equal dimensions were obtained. The Poisson's ratio was evaluated in compression using a universal testing machine with video extensiometer (Zwick/Roell, Ulm, Germany).

Manufacturing procedure of the monolithic zirconia crowns tested

For the production of the monolithic zirconia crowns the replica model was scanned using the same dental CAD/CAM scanner as above. The outer design of the crown was performed by scanning of a non-prepared tooth model (Tooth 46, A5A-500, NISSIN, Kyoto, Japan). This procedure enabled to fabricate the monolithic zirconia crowns with the same outer design. The cement space was fixed at 70 µm for all samples according to the default setting of the CAD software. The data of the crown design was transferred to the 3M Lava milling center (Digital Dental Operation, Osaka, Japan) for fabrication of the monolithic zirconia crowns with the shade of A2 (Lava Plus Zirconia, 3M/ESPE; St Paul, US). Margin adjustment of the milled crowns was, then, performed manually using a grinding point (CeraPro, Edenta, AU/SG, Switzerland). Then, polishing was performed using a series of polishing point (StarGloss, Edenta) and a wheel brush together with polishing agent (Zircon-Brite, Dental Ventures of America Corona, CA, USA).

For evaluation of the thickness of the crowns, a microCT evaluation was performed at Tokohu University using the following method and conditions. A microCT Cone beam scanning (ScanXmate-D2225RSS270, Comscantecno, Japan) was used with a voltage of 200 kV and current of 200 μ A. The number of projections was 1200 with a resolution was 14.9 μ m and a 360° rotation.

Compression test of the cement

Since the compressive strength of the cements could influence the fracture strength of the crowns tested it was of importance to test the compression strength of the cement used in advance. In the present study, the compressive strength test of three different cement (Zink phosphate cement TM, Dentsply/DeTrey, Germany; Panavia 2.0®, Kuraray, Japan and RelyX Unicem I®I, 3M/ESPE, Germany) was performed for comparison with the information given by the manufacturer and for evaluation of the results with the cemented monolithic zirconia crowns. The ISO standard of 9917-1:2004 was used as guideline for performance and evaluation of the results achieved.

A number of 10 samples of each of the cement tested were produced according to the manufacturer's instructions in a polytetra-fluoroethene (PTFE) mold with the inner dimension of 4 mm in diameter and a height of 6 mm.

The zinc phosphate cement was mixed on a glass plate in room temperature. The mix consisted of 1 scoop of powder and 7 droplets of the liquid phase to get cement with sufficient consistency. The mixed cement was introduced into the PTFE mold with a Jiffy tube (Produits Dentaires SA, Vevey, Switzerland), the end surfaces were covered by a polyethylene (PPC) film (NKV, Umeå, Sweden) and the cement left to set for 12 minutes.

For the test with RelyX Unicem the PTFE mold was filled with a mix of the two phases. The end surfaces were covered by a PPC-film and a glass plate was put on top of the mold. The cement was light cured (Bluephase®, Ivoclar, Lichtenstein) through the glass plate for 2 seconds, the plate was then removed and light curing continued for 40 seconds on one side.

For the Panavia cement, equal amounts of base and catalyst were mixed. A droplet consisting of a mixture of primer A and B was added to the mix to get proper dual cure and the cement was filled into the mold with a Jiffy tube. A PPC-film was put on each end surface, and the same procedure as for RelyX was followed.

The end surfaces were after curing polished with SiC paper (400 grit) to ensure a surface perpendicular to the force applied. After storage in $37\pm1^{\circ}$ C for 24 H, the dimensions of the specimens were controlled using a digital micrometer (IP 65, Mitutoyo, Japan). The cements were tested for compression until fracture using a Zwick/Roell universal testing machine (Ulm, Germany) at a speed of 0.75 ± 0.1 mm/min and the compressive strength was recorded.

Crown test procedure

Eighteen crowns (Lava Plus Zirconia, 3M/ESPE; St Paul, US), manufactured as described above, were used for the load-to-failure test. After production, the crowns with their abutments were divided into 3 groups with 6 samples in each group. The crowns were cemented on the abutments using one of Zink phosphate cementTM, Panavia 2.0®, or RelyX Unicem II®. Before cementation, the abutments were placed in the test jig with a light-bodied A-silicon impression material (Flexitime Correct flow, Heraeus). That was done to ensure that the abutment was not moved during the cementation.

The inner surface of the crowns and the prepared sites of the abutments were cleansed with a solution of 99% ethanol to remove any grease and debris. The alcohol was left to dry/vaporize for 60 seconds.

The Zink phosphate cement was mixed as described above on a glass plate in room temperature. A thin layer of the cement was applied to the crown, and the crown was placed on the abutment with finger pressure. Excess was removed with a carver after setting (described below).

RelyX base and catalyst pastes were mixed together according to the manufactures instructions in a mixing syringe and applied to the inner surface of the crown. The crown was then placed on the abutment with finger pressure. The excess of cement was removed as described above.

For the cementation with Panavia, the manufacturer's instructions were followed. Equal amounts of primer A and B were blended and applied to the abutment. Thereafter, equal amounts of base and catalyst pastes were blended for 20 seconds and a thin layer applied to the crown. The crown was placed on the abutment with finger pressure, and the excess was removed with Oxyguard (Kuraray, Japan) on a quick stick (Dentsolve AB, Huddinge, Sweden).

To ensure equal conditions during cementation, the crowns and their abutments respectively were placed in a Zwick/Roell universal testing machine (Ulm, Germany) and a force of 20 N was applied until set of the cement has occured. Between the load stylus with a diameter of 10 mm and the crown a sheet of 2mm polyurethane was used to ensure that the stylus surface should not affect the crown surface. For the zinc phosphate cement, the crown was put under pressure for 15 minutes to ensure that it set properly. For the crowns cemented with RelyX

and Panavia, the pressure was held for 4 minutes while the crowns were light-cured on all sides for 40 seconds each. It has been reported that a controlled pressure during cementation seems vital to achieve optimal cementation (³⁹).

After storage in distilled water (grade 2, $37\pm1^{\circ}$ C) for 24 ± 1 H, the specimens were mounted in the testing jig with a vinylpolysiloxane material (Flexitime Bite, Heraeus), covered with a sheet of 2 mm polyurethane and subjected to compression stress with a rounded stylus with a diameter of 10mm until fracture of the zirconia crown was registered, using a Zwick/Roell universal testing machine (Ulm, Germany) at a speed of 0.5 ± 0.1 mm/min. The testing procedure followed the same procedure described by Nakamura et al. (36) for testing different preparation reductions for monolithic zirconia crowns. By this, the studies made at IKO/Tromsø and Tokohu University, the results achieved can be compared.

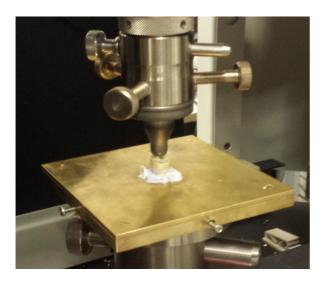


Figure 3. Abutment-crown complex mounted in testing jig for compression until fracture

The force at failure was recorded and differences between the groups concerning type of cement used accounted for. The stress behavior was analyzed both by recording the breakage on high speed video and by stress calculation. In addition, one randomly selected sample from each group was examined by light microscope.

Scanning electron microscopy was performed on one randomly chosen sample (A crown cemented with Panavia) for evaluation of the crown after fracture. The sample was sputter coated (Polaron Sputter coater E 5000, Quarum technologies Ltd, UK) with gold palladium at

1,2kV 10⁻¹ bar. The SEM investigation was performed in a Ziess-sigma scanning electron microscope (Carl Zeiss Microscopy GmbH, Oberkochen, Germany).

Statistical analysis

For comparison between groups tested, non-parametric methods were used (Wilcoxon Rank sum test and Bonferroni statistical analysis). The statistic evaluation of crown thickness was done by Tukey-Kramer HSD multiple comparison test. The level of significance was set to 5%.

RESULTS

The flexural strength, the E-modulus and the Poisson's ratio of the abutment material were 196 (SD: 10) MPa, 10.73 (SD: 0.28) GPa and 0.43 (SD: 0.03), respectively.

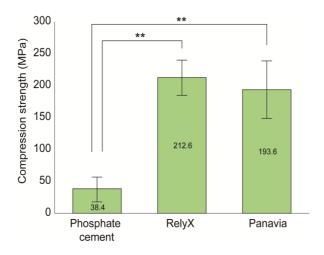


Figure 4. Compression test of cement. **: p<0.01

The compression test of the cement used displayed significant lower values for zinc phosphate cement (p<0.01) than for the two other cement tested (Figure 4). Given the numbers from the manufacturers of the different cement, the results were expected, with RelyX having slightly higher compression strength than Panavia and the zinc phosphate cement having the lowest.

The microCT analysis confirmed that the occlusal thickness was 0.5 ± 0.1 mm at any measurement points. Furthermore, it was observed that the zirconia crowns had no internal defects.

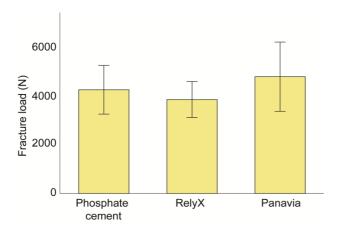


Figure 5. Fracture strength of monolithic zirconia crowns cemented using different cements

For the fracture test of the cemented crowns, high values were displayed and no significant differences between the three cements tested were recorded (see table 2). Compared to the compression test of the cements the differences between the cement were no longer as obvious.

Even though the cement space was fixed at 70 μ m in the CAM for all the crowns manufactured, light microscopy showed that the space was actually >70 μ m for all the samples. That was also confirmed by micro-CT analysis of the cement space before cementation and by SEM analysis showing a cement space occlusal above 130 μ m. The space seemed to have slightly increased more occlusal when the cement was applied.

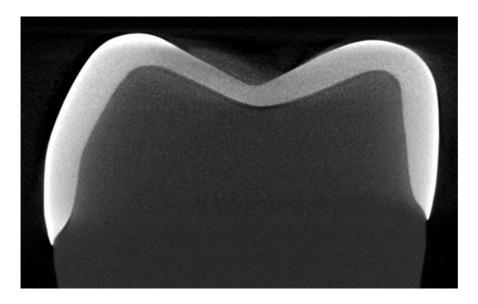


Figure 6. MicroCT image of crown and abutment before cementation. The dark line underneath the crown showing the cement space.

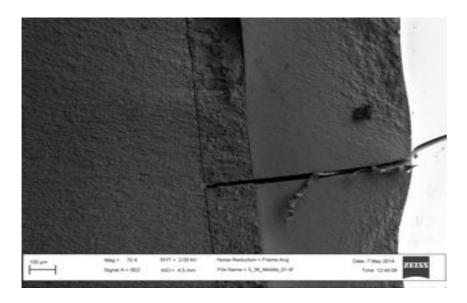


Figure 7. SEM analysis (x70 magnification) of abutment-cement-crown complex occlusal. No visible cracks were seen at the surface.

DISCUSSION

The null hypothesis of the present study was that the compressive strength of the cement would not influence the fracture strength of monolithic zirconia crowns. The results from this study showed that the hypothesis was confirmed; there was no statistical evidence showing any influence on the fracture strength.

One of the limitations with this type of study was to make the study design as optimal as possible to achieve clinical relevant results. The present study seemed to be no exception from that since it is difficult to reproduce a clinical situation in in-vitro testing (34,40). According to Kelly, traditional failure tests of single unit all-ceramic prostheses are inappropriate because they do not mimic the clinical situation to a satisfactory degree. In a clinical situation, the cyclic load over time will result in fracture of the crown (i.e. fatigue). In addition, the cracks may originate from the cementation surface in a clinical situation (41). In the present study that seemed not to be the case according to the microscopic analysis (light microscope and SEM) performed. On the other hand there were no sign of Herzian cracks at the occlusal surface. Kelly at al. (1995) suggested presence of Herzian cracks to be a sign of inappropriate loading on the surface making the test clinically less relevant. As this was not the case in the present study as well as the use of a dentin like abutment material with a flexural modulus and Poissons ratio comparable to wet dentin (Kinney et al 2003, 2004) it can

still be suggested that the results achieved can be clinically relevant. A high Poissons ratio may result in crack formation close to the crown margins due to compression-induced expansion of the abutment material used. That was experienced in the present study (figure 7). A logic conclusion must therefore be that even though the crowns tested as well as the compression strength of the cement were able to withstand high forces, the developed stress at the margin caused by the abutments expansion gave crack formation at those parts subjected to tensile stress. That may also happen in the clinical situation since the abutment used have a Poissons ration close to wet dentin.

It has also been proposed that the presence of saliva will be of importance, due to the contents ability to chemically aid in crack propagation, and physically because it will be present in the crack and over time lead to fatigue failure. In the present study the crowns were placed in a container with water for 24 hours, which is a very short period and will not properly reflect a clinical situation. Still, the corrosion of ceramics is a slow process often also depending on heat over 80°C (not clinically relevant) and an acidic environment (^{42,43}). Moreover, the aim of the present study was not to test the aging of the crowns but to test the importance of the cement for fracture strength of monolithic zirconia crowns under standardized conditions.

In vitro tests are considered of importance due to their ability of using standardized methods where different parameters can be evaluated and the differences between materials therefore more equal analyzed.

One of the strengths of the present study was that equal conditions were ensured for all the crowns in the test, thus eliminating bias. An equal study has been conducted in Japan showing similar results (³⁶).

According to the technical data sheet of RelyX Unicem Automix (3M Espe) the compressive strength of RelyX Unicem is 291 MPa. The producers of zinc phosphate cement (Dentsply/DeTrey, Germany) stated that the compressive strength of the cement is ≈ 117 MPa, and the producers of Panavia 2.0 stated that the compression strength for this material was 236 MPa.

Panavia is a dual-cure polymer resin -based cement and has been recommended by the manufacturer for all types of prosthodontic restorations, including adhesive techniques. The recommendations are due to the high bonding strength and low degree of micro leakage (⁴⁴).

The result of the compressions test in the present study showed comparable results for Panavia.

RelyX unicem (3M/ESPE) is a dual-cure dental cement. It is claimed to be a self-adhesive cement based on polymer resin with modified glass-ionomer properties. It has been recommended by the manufacturer for all types of prosthodontic restorations. The manufacturers promote it as being time saving because of the convenient dispensing and since it is self-etching. There is virtually no post-operative sensitivity and it is strong and moisture-tolerant (⁴⁵). The result of the compressions test in the present study showed that RelyX unicem slightly less compression strength.

Zinc phosphate cement consists of a powder, zinc oxide with a 2-10% additive of magnesium oxide, and a liquid consisting principally of phosphoric acid, water, and buffers. It has been recommended by the manufacturer for all types of prosthodontic restorations. The recommendations are due to good binding strength. It has also been used for a very long time in dentistry and there have been reported good results. The result of the compressions test in the present study showed that zinc phosphate had the lowest compression strength.

The variables working with phosphate cement are many; room temperature, temperature of the glass plate, the fact that the cement was not compressed during the setting, mixing time and consistency of the finished product. All this could have had an impact on the result of the test, given that the reported mean value of the phosphate cement from the manufacturer was significantly higher than the results shown in Figure 4.

Even though the cements evaluated are very different in their structure, each manufacturer has recommended their cement for all types of prosthetic restorations. The results of the present study showed that there were no significant differences between the various types of cement in the fracture test, even though the minimal thickness of the crowns was as little as 0.5 mm. This may indicate that the compression strength of the cement alone will not suffice in showing how strong the crown-cement-tooth complex will be, because a lot of other factors will influence upon this. One important factor could be the design of the preparation (³⁴). Given that the macro mechanical retention is present the cement may be of less importance to the fracture strength due to a large supporting area. Because of the relative high values achieved in the fracture test in the present study it might be suggested that crowns with thickness of 0.5 mm may have good resistance against fractures.

Microscopic analysis of the specimens showed that the crowns had one main crack in the occlusal surface of the crown-abutment complex, compared to a clinical situation, where the crack typically originates from a defect on a surface of the crown (46,47). In a clinical situation, the tooth will have physiological movement within the PDL when forces are applied, e.g mastication. In the present study, the crown-abutment complex, although not being entirely stiff because of the Poissons ratio being dentin-like, had no such ability to move when mounted in the testing jig. In this study the surfaces of the crowns were without any flaws, but they did not have a naturally physiological movement like mentioned above. When the crown-abutment complex was put under pressure, the fact that there was no physiological movement of the complex, may have forced initiation of a crack on the occlusal surface even though there were no flaws in the surface of the ceramic.

During microscopy of the fractured specimens, it was obvious that the cement space was >70 μ m for all the crowns even though the default settings of the CAD system was set at 70 μ m for all crowns. It has been indicated that a cement thickness above 70 μ m may reduce the fracture strength of crowns (⁴⁸). This seems not to be the case in the present study, where the analyses with microCT and SEM showed cement spaces over 130 μ m.

It is difficult to achieve high clinical relevance in an in vitro study. A lot of factors will be of importance in deciding whether a study has clinical relevance or not. Some of these factors have been discussed above. Even though not all of them have high relevance compared to a clinical situation, the present study may have indicated how the tooth-crown complex will react in a clinical situation.

CONCLUSION

Within the limitations of this study, the following conclusions were drawn: The compressive strength of the cement used seemed to be of no importance for the fracture resistance of monolithic zirconia crowns. The compressive strength of the cement differed significantly. The microCT analyses done showed no defects on the crowns tested, and analyses of the specimens showed that the cement space was larger than was set by the CAM setting. Because of these things, other factors seem to be of higher importance when it comes to fracture strength.

ACKNOWLEDGEMENTS

Special thanks to Dentsply/DeTrey, Germany, Kuraray, Japan and 3M/ESPE, Japan for their support and to Mathieu Mouhat for all his laboratory assistance.

REFERENCES

¹ Vagkopoulou T, Koutayas SO, Koidis P, Strub JR. Zirconia in dentistry: Part 1. Discovering the nature of an upcoming bioceramic. Eur J Esthet Dent. 2009 Summer;4(2):130-51.

² Denry I, Kelly JR. State of the art of zirconia for dental applications. Dent Mater. 2008 Mar;24(3):299-307.

³ Manicone PF, Rossi Iommetti P, Raffaelli L. An overview of zirconia ceramics: Basic properties and clinical applications. J Dent. 2007 Nov;35(11):819-26.

⁴ http://en.wikipedia.org/wiki/Zirconium_dioxide

⁵ Subbarao EC. Zirconia-an overview. In: Heuer AH, Hobbs LW, editors. Science and technology of zirconia. Colombus, OH: The American ceramic society; 1981. p 1-24.

⁶ Kisi E., Howard C. Crystal structures of zirconia phases and their interrelation. Key Eng Mater 1998;153/154:1-35.

⁷ Garvie RC, Nicholson PS. Phase analysis in zirconia systems. J Am Ceram Soc 1972;55:303-5.

⁸ Heuer AH, Lange FF, Swain MV, Evans AG. Transformation toughening: an overview. J Am Ceram Soc 1986;69:i-iv.

⁹ Hannink R, Kelly P, Muddle B. Transformation Toughening in Zirconia-Containing Ceramics J. Am. Ceram. Soc., 83 [3] 461–87 (2000).

¹⁰ Giordano R, Sabrosa C. Zirconia: Material Background and Clinical Application. Compendium November/December 2010, Volume 31, Issue 9. Published by AEGIS Communications.

¹¹ Sharhbaf S, Vannoort R, Mirzakouchaki B, Ghassemieh E, Martin N. Effect of the crown design and interface lute parameters on the stress-state of a machined crown-tooth system: A finite element analysis. Dent Mater. 2013 Aug;29(8):e123-31.

- ¹² Long H, Monolithic Zirconia Crowns and Bridges: New all-ceramic, CAD/CAM-fabricated crowns and bridges are unbreakable and less expensive than traditional full-coverage PFM restorations. Inside Dentistry, January 2012, Volume 8, Issue 1. Published by AEGIS Communications.
- ¹³ Kim JH, Lee SJ, Park JS, Ryu JJ. Fracture load of monolithic CAD/CAM lithium disilicate ceramic crowns and veneered zirconia crowns as a posterior implant restoration. Implant Dent. 2013 Feb;22(1):66-70.
- ¹⁴ Zahran M, El-Mowafy O, Tam L, Watson PA, Finer Y. (2008) Fracture strength and fatigue resistance of all-ceramic molar crowns manufactured with CAD/CAM technology. J Prosthodont. Jul;17(5):370-7.
- ¹⁵ http://solutions.3m.no/wps/portal/3M/no_NO/3M_ESPE/Dental-Manufacturers/Products/Digital-Dentistry/Dental-Crowns/Lava-Zirconia/#tab3

- ¹⁷ Mattiello R, Coelho T, Insaurralde E. et al. A Review of Surface Treatment Methods to Improve the Adhesive Cementation of Zirconia-Based Ceramics. ISRN Biomaterials Volume 2013 (2013), Article ID 185376, 10 pages.
- ¹⁸ Metha D, Shetty R. Bonding to Zirconia: Elucidating the confusion. International dentistry SA vol. 12, no. 2.
- ¹⁹ Lava Crowns and brigdes sandblasting or Rocatec treatment of Lava zirconia. http://multimedia.3m.com/mws/mediawebserver?mwsId=SSSSSuH8gc7nZxtUm8_SP8mGev Uqe17zHvTSevTSeSSSSS--&fn=lava_insight_bulletin_02.pdf.

¹⁶ http://www.bruxzir.com/features-bruxzir-zirconia-dental-crown/

²⁰3M ESPE Lava Zirconia Ageing Stability. http://multimedia.3m.com/mws/mediawebserver?mwsId=66666UgxGCuNyXTtoXTXL8TyE VtQEcuZgVs6EVs6E666666--.

- ²¹ Kanie T, Kadokawa A, Nagata M, Arikawa H. A comparison of stress relaxation in temporary and permanent luting cements. J Prosthodont Res. 2013 Jan;57(1):46-50.
- ²² Margeas R. Self-adhesive resin cements: Convenient, strong and nearly universal. Dental Town, 64-68.
- ²³ Papia E, Zethraeus J, Ransbäck PÅ, Wennerberg A, Vult von Steyern P. Impaction-modified densely sintered yttria-stabilized tetragonal zirconium dioxide: methodology, surface structure, and bond strength. J Biomed Mater Res B Appl Biomater. 2012 Apr;100(3):677-84.
- ²⁴ De Munck J, Vargas M, Van Landuyt K, Hikita K, Lambrechts P, Van Meerbeek B. Bonding of an auto-adhesive luting material to enamel and dentin. Dent Mater. 2004 Dec;20(10):963-71.
- ²⁵ http://www.kuraray-dental.eu/scientific-area/core-technologies/mdp-monomer/
- ²⁶ Triolo PT, Kelsey WP, Barkmeier WW. Bond strength of an adhesive resin system with various dental substrates. J Prosthet Dent. 1995 Nov;74(5):463-8.
- ²⁷ Sasse M, Eschbach S, Kern M. Randomized clinical trial on single retainer all-ceramic resin-bonded fixed partial dentures: Influence of the bonding system after up to 55 months. J Dent. 2012 Sep;40(9):783-6.
- ²⁸Dias de Souza GM, Thompson VP, Braga RR. Effect of metal primers on microtensile bond strength between zirconia and resin cements. J Prosthet Dent. 2011 May;105(5):296-303.
- ²⁹ Technical data sheet RelyX Unicem 2 Automix.
 http://multimedia.3m.com/mws/mediawebserver?mwsId=66666UgxGCuNyXTtnxf2l8TXEVt
 QEcuZgVs6EVs6E666666--&fn=rx_u2_auto_tds.pdf

³⁰Zhang L, Tang T, Zhang ZL, Liang B, Wang XM, Fu BP. Improvement of enamel bond strengths for conventional and resinmodified glass ionomers: acid-etching vs.conditioning. J Zhejiang Univ Sci B. 2013 Nov;14(11):1013-24.

³¹ Pameijer CH. A Review of Luting Agents, Int J Dent. 2012;2012:752861.

³² Lindquist E, Karlsson S.Success rate and failures for fixed partial dentures after 20 years of service: Part I. Int J Prosthodont. 1998 Mar-Apr;11(2):133-8.

³³ https://www.dentsply.co.uk/Products/Restorative/Cements/DeTrey-Zinc.aspx

³⁴ Nakamura K, Harada A, Inagaki R, Kanno T, Niwano Y, Milleding P, Örtengren U.
Fracture strength of full-contour zirconia molar crowns in relation to crown thickness. JDR,
2014 (submitted).

³⁵ Kelly JR (1999). Clinically relevant approach to failure testing of all-ceramic restorations. *J Prosthet Dent* 81(6):652-661.

³⁶ Rosentritt M, Plein T, Kolbeck C, Behr M, Handel G (2000). In vitro fracture force and marginal adaptation of ceramic crowns fixed on natural and artificial teeth. *Int J Prosthodont* 13(5):387-391.

³⁷ Yucel MT, Yondem I, Aykent F, Eraslan O (2012). Influence of the supporting die structures on the fracture strength of all-ceramic materials. *Clin Oral Investig* 16(4):1105-1110.

³⁸ Scherrer SS, de Rijk WG (1993). The fracture resistance of all-ceramic crowns on supporting structures with different elastic moduli. *Int J Prosthodont* 6(5):462-467.

³⁹ Zortuk M. Bolpaca P. Kilic K. Ozdemir E. Aguloglu S. Effects of Finger Pressure Applied By Dentists during Cementation of All-Ceramic Crowns.Eur J Dent. 2010 October; 4(4):383–388.

⁴⁰ Øilo M, Kvam K, Tibballs JE, Gjerdet NR. Clinically relevant fracture testing of all-ceramic crowns.Dent Mater. 2013 Aug;29(8):815-23.

- ⁴¹ Øilo M, Gjerdet NR. Fractographic analyses of all-ceramic crowns: a study of 27 clinically fractured crowns. Dent Mater. 2013 Jun;29(6):e78-84.
- ⁴² Milleding P, Wennerberg A, Alaeddin S, Karlsson S, Simon E. Surface corrosion of dental ceramics in vitro. Biomaterials 20 (1999) 733-746.
- ⁴³ Dhima M, Assad D, Volz J, An K, Berglund L, Carr A, Salinas T. Evaluation of Fracture Resistance in Aqueous Environment of Four Restorative Systems for Posterior Applications. Part 1. Journal of Prosthodontics Volume 22, Issue 4, pages 256–260, June 2013.
- ⁴⁴ Panavia F 2.0 Brochure. http://www.kuraraydental.com/product/cements/panavia-f2-0
- ⁴⁵ RelyXTM Unicem 2 Automix Self-Adhesive Resin Cement.
 http://solutions.3m.com/wps/portal/3M/en_US/3M-ESPE-NA/dental-professionals/products/espe-catalog/~/RelyX-Unicem-2-Automix-Self-Adhesive-Resin-Cement?N=5145460+3294798215&rt=rud
- ⁴⁶Oilo M, Kvam K, Gjerdet NR. Simulation of clinical fractures for three different all-ceramic crowns. Eur J Oral Sci. 2014 Jun;122(3):245-50.
- ⁴⁷ Øilo M. High-strength dental ceramics. Potential failure indicators for dental zirconia. Dissertation for the degree of philosophiae doctor.
- ⁴⁸ Tuntiprawon M, Wilson PR. The effect of cement thickness on the fracture strength of all-ceramic crowns. Aust Dent J. 1995 Feb;40(1):17-21.