

Aus der Klinik für Zahnärztliche Prothetik, Propädeutik und Werkstoffkunde

(Direktor: Prof. Dr. M. Kern)

Universitätsklinikum Schleswig-Holstein, Campus Kiel
an der Christian-Albrechts-Universität zu Kiel

**EFFECT OF AIR-ABRASION ON THE RETENTION OF ZIRCONIA CERAMIC CROWNS LUTED
WITH DIFFERENT CEMENTS BEFORE AND AFTER ARTIFICIAL AGING**

Inauguraldissertation

zur

Erlangung der Würde eines Doktors der Zahnheilkunde
der Medizinischen Fakultät
der Christian-Albrechts-Universität zu Kiel

vorgelegt von

RAMEZ SHAHIN

aus **Damaskus, Syrien**

Kiel **2010**

1. Berichtstatter: Prof. Dr. Matthias Kern
 2. Berichtstatter: Prof. Dr. Birte Grössner-Schreiber
- Tag der mündlichen Prüfung: 06.06.2011
Zum Druck genehmigt, Kiel, den 06.06.2011

gez.:

Index

1	Introduction	5
1.1	Historic overview	5
1.2	Ceramics.....	6
1.2.1	Silicate ceramics	6
1.2.2	Oxide ceramics	6
1.3	Cements.....	7
1.3.1	Water-based cements	7
1.3.2	Resin composite cements	8
1.4	Previous studies on retention and bond strength to zirconia ceramic	8
1.4.1	Evaluation methods	9
1.4.2	Retention and bond strength	9
1.4.3	Surface treatment	10
1.4.4	Taper mode	10
1.4.5	Cements and surface treatments tests	10
1.5	Statement of the problem	12
1.6	Objectives.....	13
2	Materials and methods	14
2.1	Cements.....	15
2.2	Particle air-abrasion	16
2.3	Crowns surface determination	16
2.4	Luting procedures	16
2.4.1	Zinc phosphate cement	17
2.4.2	Glass ionomer luting cement	17
2.4.3	Adhesive resin	18
2.5	Artificial aging	18

2.6	Retention test.....	19
2.7	Failure mode	19
2.8	SEM images	20
2.9	Statistical analysis	20
3	Results	21
3.1	Dislodgment force.....	21
3.2	Retention	21
3.2.1	Statistical analysis of the retention	22
3.3	Failure mode	24
3.4	SEM images	24
4	Discussion.....	27
4.1	Discussion of the methods	27
4.1.1	Preparation design	27
4.1.2	Artificial aging	27
4.2	Discussion of the results	27
4.3	Influence of the luting cement on crown retention	28
4.4	Influence of air-abrasion on crown retention.....	30
4.5	Influence of artificial aging on crown retention.....	31
4.6	Failure modes	32
5	Conclusions	33
6	Summary	34
7	Zusammenfassung.....	35
8	Literature	37
9	Appendix.....	44

1 Introduction

With the increased improvement of the community oral health care, preventive dentistry and the development of the dental treatments, the concern of both dentist the patient is more focusing today on aesthetic treatment, better appearance and quality of life [1]. Supported by advanced technology, which joins forces of many science disciplines, the dental restoration science has achieved a large step in materials as well as in the techniques [2].

1.1 Historic overview

Since their introduction in the late 1950's, metal-ceramic restorations were commonly used restorations to restore esthetics and structural integrity of discolored, heavily restored, fractured or worn teeth [3]. However, many disadvantages of metal-ceramic restorations such as discoloration, allergy caused by released metal ions and limited esthetics led to the development of all-ceramic restorations [4-5].

Dental restorations should fulfill the following criteria: strength, fit, esthetics, and biocompatibility. In spite of the advantages of all-ceramic restoration including life like appearance, biocompatibility, and durability, for decades there were still disadvantages of their use. Disadvantages like limited fracture strength, inadequate marginal fit, and adhesive wear of the opposing dentition, as well as relative lack of the retention [6-8].

Continuous development of ceramic materials including manufacturing techniques and luting materials, allowed for the introduction of various new all-ceramic crown systems [9-10]. The need for stronger and more durable all-ceramic restorations led to the introduction of ceramics with an increased alumina and zirconia content [11]. Computer Aided Design/Computer Aided Manufacturing (CAD/CAM) is among the recent advances in dental technology for direct and indirect fabrication of all-ceramic restorations [12-15].

1.2 Ceramics

Ceramics are defined as non-metallic, inorganic, man made solid objects, formed by baking raw materials (minerals) at high temperature [3-4, 16]. According to their chemical composition dental ceramics can be classified into two major categories, silicate ceramics and non-silicate (high strength oxide ceramics) [16-17].

1.2.1 *Silicate ceramics*

In dentistry silica-based ceramics have been used for dental restorations reinforced with metal substructure and luted to the teeth with conventional cements for decades [11, 18].

Silicate ceramics are formed of clay or kaolin (Al_2O_3 , SiO_2 , $2\text{H}_2\text{O}$), feldspar (K_2O , Al_2O_3 , 6SiO_2) and quartz (SiO_2). However due to their inherent low mechanical properties, clinically when used as all-ceramic restorations they had a high tendency to fracture during mastication. Recently, modified silicate ceramics, i.e. lithium disilicate ceramics, with improved strength properties have been introduced [19-21].

1.2.2 *Oxide ceramics*

These ceramics contain only a minimal amount of silicate or no silicate at all [17, 19]. By definition oxide ceramics contain less than 15 wt% silica and only a small or no glass phase. Current dental oxide ceramics consist mostly of alumina, magnesia, zirconia and yttria [21]. Based on technique of fabrication, they can be classified in two systems [15]:

Sintered ceramics: Sintering is defined as a process of fusion by point contact to particles resulting in densification by viscous flow of a ceramic or glass powder, produced by heating or heat and pressure [20]. Examples include conventional powder slurry porcelain, In-Ceram, Procera All-Ceram and zirconia ceramics [16, 22].

Machinable ceramics: CAD/CAM (computer-assisted design/computer-assisted manufacturing) machinable ceramics and adhesive cementation are new technologies in dentistry [9]. Machinable ceramics are used with CAD/CAM and copy milling techniques. These ceramics are manufactured under optimized industrial conditions. Therefore, the risk of laboratory defects such as pores, flaws, and stress cracks are minimized which results in ceramic materials with improved properties compared to their equivalent laboratory fabricated ceramics [15, 23-24].

1.2.2.1 *Zirconium oxide (ZrO₂)*

Zirconium oxide is an oxide ceramic with many attractive properties, such as its white opaque color and outstanding biocompatibility. In addition to this it features a high degree of crack resistance which distinguishes it among oxide ceramics. The latter is a result of the ability of zirconium dioxide to be stabilized in its tetragonal high-temperature phase by means of suitable additives, e.g. yttrium oxide. Only when applying an external source of energy, as for example in the case of a beginning crack individual zirconium oxide grains are transformed, localized and accompanied by an increase in volume, to their stable monoclinic form at room temperature.

This procedure is described as transformation strengthening. The compressive stresses arising within the structure prevent the unhindered growth of a crack and hence the failure of the ceramic. This behavior results in a so-called tension expansion, a phenomenon otherwise known only in the case of steel. For this reason zirconium oxide is also referred to as "ceramic steel". This property is also reflected in the long life of zirconium dioxide under permanent loading.

Depending on the specific composition, fracture strength of sintered zirconia can exceed 1000 MPa. Zirconium-oxide ceramic is indicated for conventional and resin-bonded FPDs "Fixed Partial Dentures", full coverage crowns, implant abutments, and endodontic posts [17].

1.3 Cements

1.3.1 *Water-based cements*

The most commonly used water-based permanent luting agents are zinc phosphate and glass-ionomer cements. Zinc phosphate cement has served for decades as the universal cement for different applications in restorative dentistry relying on retention and resistance form of the tooth preparation and an adequate marginal fit.

Because of its long history of successful clinical use, associated with cast and metal-ceramic restorations, zinc phosphate cement was considered as the 'reference' or 'gold standard' [25-26].

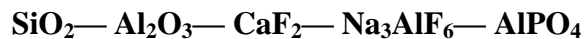
1.3.1.1 *Zinc phosphate cement*

Zinc phosphate cement is one of the oldest and widely used cements. It is a high-strength cement base, mixed from zinc oxide powder and phosphoric acid liquid. Due to its low initial pH, it may cause pulpal irritation, especially where only a thin layer of dentin exists between the cement and the pulp; thus is especially important to follow the correct procedures and precautions when using zinc phosphate cement. Zinc phosphate cement is a traditional crown and bridge cement used for alloy restorations. It is supplied as a powder and a liquid, both of which are carefully compounded to react with one another during mixing to develop a mass of cement possessing desirable physical characteristics.

1.3.1.2 *Glass ionomer cement*

Glass ionomer cements are supplied as a powder and a liquid or as a powder that is mixed with water. Several products are encapsulated. The liquid typically is a 47.5% solution of 2:1 poly-acrylic acid/itaconic acid copolymer (average molecular weight 10,000) in water. The itaconic acid reduces the viscosity of the liquid and inhibits gelation caused by intermolecular hydrogen bonding; D (+) tartaric acid (5%, the optically active isomer) in the liquid serves as an accelerator by facilitating the extraction of ions from the glass powder.

The powder of glass ionomer cement is a calcium fluoroaluminosilicate glass with a formula of:



The maximum grain size of the powder appears to be between 13 and 19 μm .

The powder is described as an ion-leachable glass that is susceptible to acid attack when the Si/Al atomic ratio is less than 2: 1. Barium glass or zinc oxide may be added to some powders to provide radiopacity.

1.3.2 *Resin composite cements*

With advances in the field of polymerizing cements with added advantages of luting to dental tissues and to indirect restorations, conservative preparation designs could be achieved with reduced need for macro-retention. The reliable adhesion to enamel achieved with the adhesive techniques has had a major impact on saving the remaining tooth structure and has led to a crucial change in the existing paradigms in tooth preparation. Nevertheless, adhesive luting techniques for oxide ceramics can provide significant clinical advantages over conventional cementation of dental restorations. An adhesive luting can provide gap-free

restoration margins, minimizing microleakage and thereby reducing the risk of secondary caries [27]. In addition, esthetics might be improved by using tooth colored transparent resin luting agents as compared to opaque conventional cements. Most importantly, adhesive luting does not require retentive tooth preparation, which often causes invasive removal of sound tooth structure. So adhesive bonding of oxide ceramics allowed the introduction of new non-invasive treatment modalities for tooth replacement by resin bonding all-ceramic fixed partial dental prostheses to lingual surfaces of teeth adjacent to an edentulous area [28-29]. Furthermore, adhesive bonding might provide stabilization of the remaining tooth structure when oxide ceramics are used for post and core restorations of structurally severely compromised teeth after endodontic treatment [21, 30].

1.4 Previous studies on retention and bond strength to zirconia ceramic

1.4.1 *Evaluation methods*

Material selection and clinical recommendations on luting to ceramics are based on mechanical laboratory tests which show great variability in materials and methods [31-32]. Chemical [33-34], thermal [35-37], and mechanical [38] influences under intraoral conditions were generally used.

The simulation of such influences in the laboratory is compulsory to draw conclusions on the long-term durability of a specific luting procedure and to identify superior materials and techniques. Long-term water storage [39] and thermal cycling of bonded specimens are accepted methods to simulate aging and to stress the luting interface. Most studies that apply these methods reveal significant differences between early and late bond strength values [40-43]. Also application of mechanical cyclic loading (fatigue load) might cause significant reduction of bond strengths [44-45].

1.4.2 *Retention and bond strength*

The most common bond strength tests are the 3-point bending test, the tensile and micro-tensile test, and the shear and micro-shear test [46]. The most frequent testing method is the shear bond test; however, some researchers prefer modified tensile tests to eliminate the occurrence of non-uniform interfacial stresses typical to conventional tensile and shear bond tests [47].

1.4.3 Surface treatment

A strong bond relies on micromechanical interlocking and chemical bonding to the ceramic surface, which requires roughening and cleaning for adequate surface activation. Common treatment options are grinding abrasion with diamond rotary instruments [48-50], airborne particle abrasion with aluminum oxide [51-53], acid etching [53-55], silica coating and silanization [48, 56-58] and combinations of these methods.

1.4.4 Taper mode

Luting to zirconium oxide ceramic was the subject of a number of studies. The details of these studies have been contradictory with regard to cementation mode, preparation geometry of the margin, angle of convergence, and extent of tooth removal. Some studies showed that comparing to metal-based restorations, all-ceramic restorations should not involve any primary retention, as this would produce crack-inducing tensile stresses from the inner surface of the restoration. Certain preparation guidelines that differ from recommendations for metal-supported systems have to be taken into consideration for all-ceramic FPDs. Nevertheless all-ceramic restorations exhibit an inferior overall fit compared with cast metal or metal-ceramic restorations [59-61].

1.4.5 Cements and surface treatments tests

Comparing the different types of cements, Tinschert et al. found that full-coverage zirconium-oxide ceramic restorations and FPDs may not require adhesive cementation [62]. However, a sufficient resin bond has the aforementioned advantages and may become necessary in some clinical situations, such as compromised retention and short abutment teeth [63].

Osman et al. compared the film thickness and rheological properties of zinc phosphate cement with different polymerizing cements, including Panavia 21, Superbond, All Bond C&B Cement, and Variolink. An initial film thickness of 25 μm was observed and was not significantly different between the cements [64].

Zirconia ceramic surface treatment was also subject to many studies. Derand and Derand evaluated different surface treatments and resin cements and found that an auto-polymerizing resin cement (Superbond C&B) exhibited the significantly highest retentions regardless of surface treatment (silica coating, airborne particle abrasion, HF etching, or grinding with a diamond bur). Water storage for 60 days had mixed effects on retentions [55].

Kern and Wegner evaluated different adhesion methods and their durability after long-term storage (150 days) and repeated thermal cycling. As surface treatment they used air-abrasion alone and the additional use of a silane or acrylizing. Only the phosphate-modified resin cement after airborne particle abrasion provided a long-term durable resin bond to zirconia ceramic. These findings were confirmed by a long-term study in which specimens were subject to 2 years water storage and repeated thermal cycling [65]. Wegner and Kern found also that restorations made of yttrium-oxide-partially-stabilized zirconia ceramic (YPSZ) can be cemented non-adhesively or adhesively with self-curing composite Panavia 21 or the dual curing composite Panavia F (Kuraray). They found that durable bonding could be achieved with the resin composite products with the air-abraded surface of the zirconia substructures without the need for silication and silanization of the surfaces [58].

1.5 Statement of the problem

As the use of conventional cements is strongly dependent on a retentive tooth preparation and excellent marginal fit of the restoration, polymerizing cements can confer adhesive properties to both the tooth tissues and the restorative material. They also present negligible solubility and significantly improved optical properties. The use of adhesive cements for translucent restorative materials enables an outstanding esthetic outcome and simultaneously reduces the need for retentive tooth preparation. This can be considered as a high impact on the preservation of sound tooth tissues [66].

Although clinical studies showed considerable failure rates related to loss of retention when using conventional cements [67], manufacturers claim that zirconia ceramic restorations can be cemented successfully with either conventional or adhesive cements. Also, various authors suggested the use of conventional cements for zirconia ceramic restorations and stated that retention of conventional cements can be equal or even better than with adhesive resins [22, 52, 57].

Nevertheless, some manufacturers do not recommend air-abrasion prior to cementation taken into consideration that air-abrasion might affect the ceramic surface by creating micro cracks which might reduce the fracture strength of the ceramic [68]. Some authors suggested that the machining process generated a surface roughness what was at least as rough as a machined and air-abraded surface in some machinable all-ceramic systems [22, 55, 57]. Other authors suggested that resin luting agents providing durable resin bonds significantly strengthen ceramic materials by “healing” minor surface defects caused by air-abrasion [69-70]. However, many studies showed improved bond strength to zirconia ceramic after air-abrasion [48-50, 71]. Even more, there are authors who showed that air-abrasion might even strengthen zirconia ceramics [72-73]. Nevertheless, the question arises whether air-abrasion can be omitted when cementing crowns in order to avoid weakening of the ceramic. Some studies tested the zirconia crowns retention like Palacios et al. [52] and Ernst et al. [74], but both studies did not test air-abrasion versus no air-abrasion. Besides, no long-term aging was performed.

However, data on the retention of zirconia ceramic crowns when luted with conventional or adhesive cements and whether crown retention is influenced by air-abrasion are missing. In addition the effect of artificial aging on crown retention is not known.

1.6 Objectives

The purpose of this in vitro study was to evaluate the influence of air-abrasion on the retention of zirconia ceramic crowns luted with zinc phosphate cement, glass ionomer cement, and composite resin to human teeth. Furthermore, the durability of the retention over 150 days storage time with artificial aging should be tested.

The null hypotheses of the study were:

1. Crown retention is not influenced by luting materials.
2. Air-abrasion does not affect crown retention.
3. Artificial aging has no influence on crown retention.

2 Materials and methods

Ninety six caries free extracted human maxillary premolars were collected from orthodontic clinics in Damascus, Syria. Approval was obtained from the local ethical committee. They were mostly extracted from young patients for orthodontic reasons. Only carious free premolars were selected, cleaned with scalpel, and stored in thymol 0.1% solution (Caelo, Hilden, Germany) at room temperature.

The roots of the teeth were embedded in custom made standard copper cylinders (25 mm long, *outer* Ø 15 mm, *inner* Ø 13 mm) positioned along their long axis, with auto polymerizing acrylic resin material (Technovit 4000, Heraeus Kulzer, Wehrheim, Germany). The margins of the crowns were located 1 mm above the level of the embedded resin, the roots inside the cylinders were retained by a thin steel bar (10 mm long, Ø 1 mm) inserted in the apical third of the root. After embedding, the teeth were stored in 0.1% thymol solution at room temperature [75].

All the premolars were prepared for all-ceramic crowns in standard manner (12 degree taper, approximately 3mm axial length, and 0.8 mm right angle rounded shoulder). The shoulder depth was defined with wheel diamond bur (Meisinger 908032, Neuss, Germany), the axial surfaces of the tooth were prepared using the hand piece secured in a parallelometer (Amann, Vienna, Austria) with 6° (one side to the long axis) standard cylindrical diamond bur at a speed of 55,000 r/m. A fine bur with the same angle was used for finishing of the preparation at the speed 10,000 r/m. The occlusal surface was prepared with occlusal double cone diamond bur (811.314.037, Komet Dental, Lemgo, Germany). Then the specimens were stored in 0.1% thymol solution. The preparation was done 3 days after the embedding of teeth [75].

CAD/CAM zirconia ceramic crowns (Vita In-Ceram YZ for inLab, Vita, Bad Säckingen, Germany) were manufactured in the dental laboratory of the prosthetic department at Kiel University using a CAD/CAM machine (Cerec 3, Sirona, Bensheim, Germany). The upper occlusal surface of the crowns was designed with 60° (to the long axis of the tooth), and with two wings on the distal and mesial surfaces of the crown to provide retention for the dislodgment apparatus (Fig. 1).

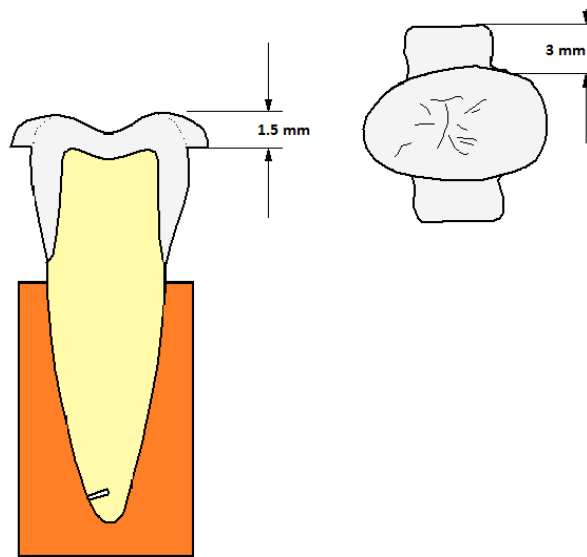


Fig.1 The tooth preparation and design of the crown with two wings on the sides.

2.1 Cements

Two kinds of conventional cements (zinc phosphate cement, Hoffmann quick setting, Hoffman, Berlin and glass ionomer cement; Ketac Cem Maxicap, 3M Espe, Seefeld, Germany), and one adhesive resin cement (Panavia 21 TC, Kuraray, Osaka, Japan), were used in this study (Table 1).

Table 1. Used Materials.

Material	Manufacturer	Batch No.
Zirconia ceramic (Vita In-Ceram YZ for inLab)	Vita, Bad Säckingen, Germany	19033
Zinc phosphate cement (Hoffmann quick setting)	Hoffmann Dental, Berlin, Germany	<i>P.</i> 1104 A 16 <i>L.</i> 1116 B 04
Glass ionomer cement (Ketac Cem Maxi Cap)	3M ESPE, Seefeld, Germany	249006
Resin composite cement (Panavia 21 TC)	Kuraray, Osaka, Japan	41279

2.2 Particle air-abrasion

The ninety-six uniformly prepared human premolars were divided by random into three cement-bonded groups of 32 teeth each. Half of the crowns in each group were air-abraded (Al_2O_3 particles of size 50 μm , 0.25 MPa for 15 seconds, Harnisch-Rieth P-G400, Winterbach, Germany) one hour before cementation. To assure that the complete surface was sufficiently air-abraded, the intaglio surface of each treated crown was dyed with a blue marker before air-abrasion. The other half of the specimens was left as received.

In standard clinical manners, the tooth coronal part was cleaned with a hand piece dental brush and fine powder (Bimsstein) on low speed of 10,000 rpm then rinsed with water and air-dried for 5 seconds directly before luting [76].

All the crowns were cleaned in the ultrasonic device (Sonorex Super RK 102H, Germany) for 5 minutes in 99% ethanol directly before cementation.

2.3 Crowns surface determination

Prior to cementation the surface area of each preparation including the occlusal surface was determined by adapting a plastic foil (Adapta 0.5 mm, Bego, Bremen, Germany) to the master die and cutting foil excess at the line angles (Fig. 2a). This measurement foil was cut into pieces and was digitized on a flat bed scanner (Epson Expression 1680 pro) (Fig.2b). The digitalized images were used to determine the surface of the foil with a computer program (Photoshop CS3 extended, San Jose, Ca. USA) [75].

The measured surface area ranged from 44.9 mm^2 to 99.2 mm^2 . To minimize the effect of different surface areas, teeth were randomly allocated to groups.

2.4 Luting procedures

For cementation, the setting time of the cements as stated by the manufacturer was doubled in each group before further manipulation of the specimens to ensure unaltered setting/ polymerization of the luting material.



(a)

(b)

Fig. 2 (a) Adapting a plastic foil (Adapta) to the master die and cutting foil excess at the preparation margins. (b) The plastic foil cut and flattened on in order to be digitalized.

2.4.1 Zinc phosphate cement

For zinc phosphate cement groups, prepared dentin was dried, the cement was mixed according to the manufacturer's instructions, and the copings were placed with finger pressure, then the specimen was mounted under pressure of 20 N for 10 minutes in a special device (Fig. 3) at room temperature.



Fig. 3 Special device used to load the crowns with 20 N during setting of the cements.

2.4.2 Glass ionomer luting cement

In glass ionomer groups, the prepared dentin was dried using cotton pellets, the Ketac capsules were mixed for 8 seconds in a mixing machine (EspeRoto Mix, 3M Espe, Seefeld Germany), the copings were placed with finger pressure, and then mounted under pressure of 20 N for 14 minutes in a special device at room temperature.

2.4.3 Adhesive resin

For Panavia 21, ED primer (Kuraray, Osaka, Japan) was mixed for 3-5 seconds and applied to the tooth surface, left for 60 seconds and lightly air-dried, Panavia 21 two pasts were mixed for 25 seconds, the cement was evenly distributed on the intaglio crown surface with a pen brush, and the copings were placed with finger pressure. The excess of the luting resin was removed from the margins with plastic pellets; Oxyguard II (Kuraray) was applied around the margins and the specimen. Using setting force of 20 Newton the specimens were placed into an oven at 37°C for 10 minutes. Oxyguard II was removed by water spray.

2.5 Artificial aging

All the specimens were stored in a 37° C water bath after cementation [77-78]. Subgroups of eight specimens each were stored in distilled water at 37°C either for 3 or for 150 days. During the storage time the teeth in the 150-day groups were thermal cycled between 5 and 55°C in distilled water for 37,500 cycles with a 30 s dwell time at each temperature.

After 150 days the specimens were placed in a masticatory simulator. The masticatory simulation was applied with a 6-mm steatite ceramic ball for 300,000 cycles at 1.5 Hz frequency with load of 50 N and a speed of 30 mm/s and lateral movement of 0.3 mm. The load was applied 2 mm eccentrically to the occlusal surface (Fig. 4).



Fig. 4 (a) Specimens mounted in the masticatory simulator. (b) Schematic drawing of the load application, red indexes refer to movement direction.

2.6 Retention test

The crowns of all specimens were dislodged along the long axis of the teeth (Fig. 5) with a universal testing machine (Zwick Z010/24, FRG, Ulm, Germany) at a crosshead speed of 2 mm/minute.

The force at dislodgement was recorded. The force was converted to stress using the surface area of each preparation. The total surface including the occlusal surface was considered.

Retention in MPa was calculated by dividing the crowns dislodgment force in N by the total surface size of the tooth preparation in mm²:

$$\text{Retention (MPa)} = \frac{\text{Dislodgement force (N)}}{\text{Total crown surface size (mm}^2\text{)}}$$

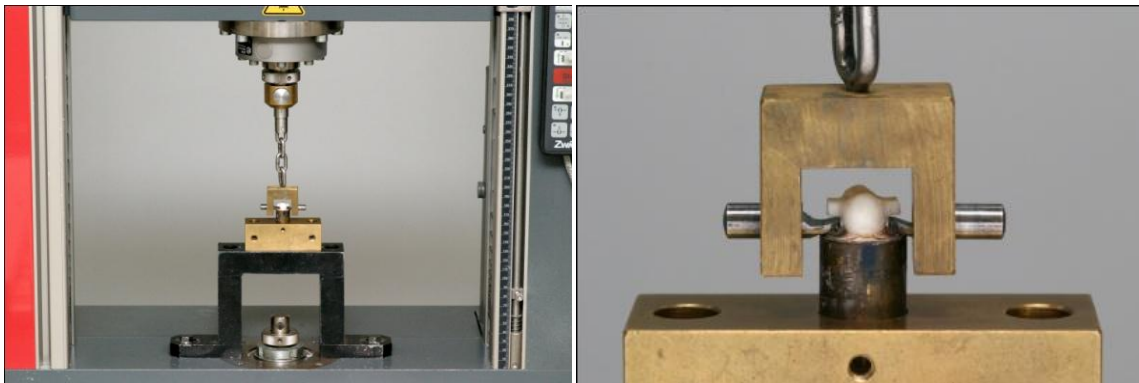


Fig. 5 Dislodgement of the crown along the long axis of the tooth.

2.7 Failure mode

The failure mode was determined as descriptive information with the use of light microscope (Carl Zeiss Micro Imaging, Jena, Germany) at x10 magnification. All prepared teeth and the intaglio surface of the crowns were investigated.

The type of failure after crown removal was divided into four different categories according to Johnson et al. 1998 (Table 2).

Table 2. Categories for characterization of type of failure after crown removal.

Category	Description
1	Cement mainly on prepared tooth (over 75%)
2	Cement on both crown and tooth (between 25 and 75%)
3	Cement mainly on crown (over 75%)
4	Fracture of tooth root without crown separation

2.8 SEM images

The intaglio surface of 2 zirconia crowns were examined using scanning electron microscopy (SEM, XL 30 CP, Philips, Kassel, Germany) operating at x500 magnification to characterize the as-received condition and to visually evaluate the effect of airborne-particle abrasion with 50 μm alumina particles for 15 seconds.

SEM photos were made also operating at $\times 800$ and $\times 1600$ magnification to investigate the dentin after the dislodgment of the crowns in each group.

2.9 Statistical analysis

Statistical analysis was performed with three-way ANOVA. Pair-wise comparison of groups was made with the Tukey test. The mode of failure was analysed with Pearson's chi-square test ($\alpha = 0.05$).

The statistical analysis was done with the support of the Institute of Medical Information and Statistic, Christian-Albrechts University, Kiel.

3 Results

3.1 Dislodgment force

Mean dislodgment force of crowns in N presented in Fig. 6.

Mean dislodgment force ranged between 102 N to 296 N after 3 day and 85 N to 231 N after 150 day storage with aging. The force was converted to stress using the surface area of the entire preparation of each tooth.

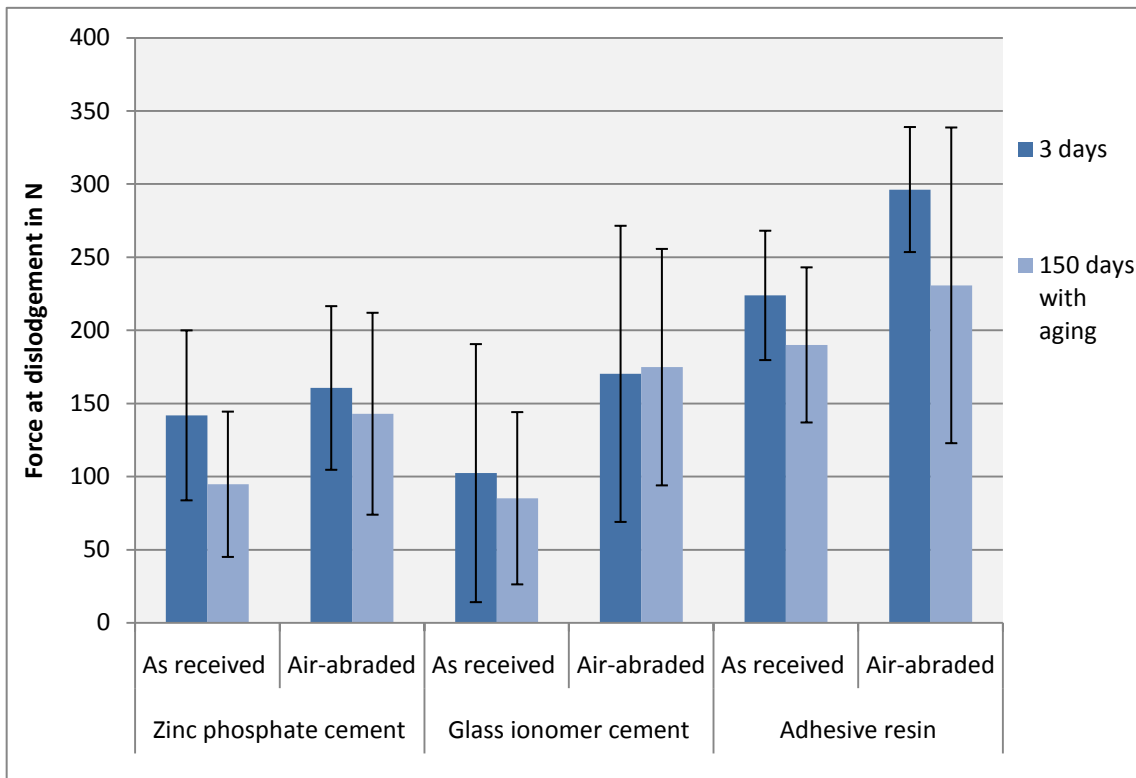


Fig. 6 Crown dislodgment force, means and standard deviations in N.

3.2 Retention

Mean crown retention and the standard deviation in MPa presented in Fig. 7.

For zinc phosphate cement as received groups, the mean dislodgment stress after 3 days was 3.0 ± 1.0 MPa and decreased to 1.6 ± 1.3 MPa with aging. By air-abraded crowns it was 3.9 ± 1.4 MPa and remained nearly the same after aging with 3.5 ± 1.3 MPa. For glass ionomer as received, it was 2.8 ± 1.9 MPa and 2.1 ± 1.0 MPa over storage time. Air-abraded it was plainly higher with 4.8 ± 2.3 MPa and 4.4 ± 1.5 MPa.

For the adhesive resin cement the mean retention stress after 3 days for as received groups was 6.1 ± 1.2 MPa and after aging 4.7 ± 0.7 MPa. It was 7.1 ± 1.1 MPa to and 6.1 ± 1.9 MPa with particle air-abrasion.

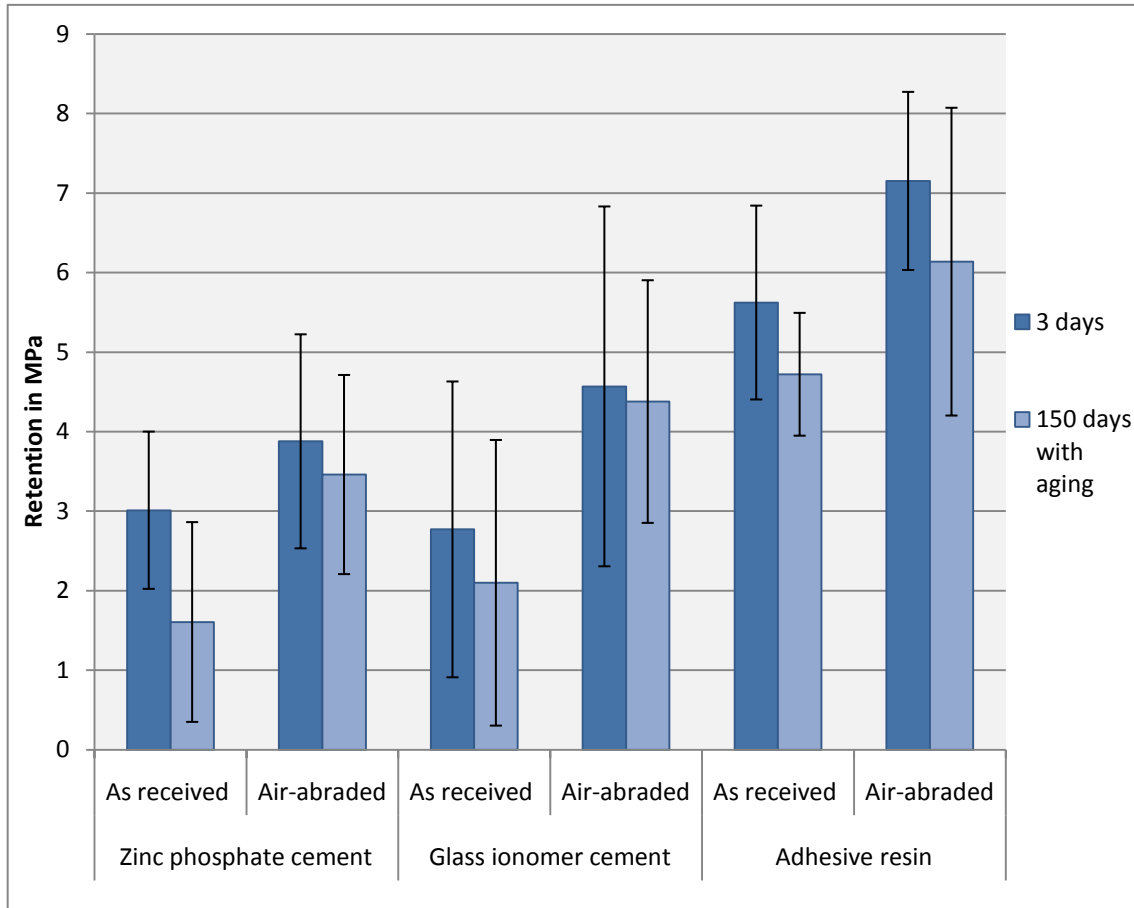


Fig. 7 Crown retention, means and standard deviations in MPa.

3.2.1 Statistical analysis of the retention

Three-way ANOVA showed a significant influence of the three tested factors on crown retention without a significant interaction of factors. Multiple comparison of cement groups with Tukey test showed that the phosphate monomer containing adhesive resin (Panavia 21) provided statically significantly higher retention than the conventional luting cements ($p < 0.001$). Zinc phosphate and glass ionomer cements showed no significant differences in retention ($p = 0.549$). Air-abrasion increased retention ($p < 0.001$), while artificial aging reduced retention ($p = 0.017$) (Tables 3-4).

Table 3. Summary of three-way ANOVA on retention.

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	214.873	11	19.534	7.060	.000
Intercept	1651.491	1	1651.491	596.913	.000
Air-abrasion	40.239	1	40.239	14.544	.000
Aging	16.501	1	16.501	5.964	.017
Cement	153.721	2	76.860	27.780	.000
Air-abrasion × Aging	.010	1	.010	.004	.951
Air-abrasion × Cement	.762	2	.381	.138	.871
Aging × Cement	.547	2	.274	.099	.906
Air-abrasion × Aging × Cement	3.093	2	1.546	.559	.574
Error	232.405	84	2.767		
Total	2098.769	96			
Corrected Total	447.278	95			

Table 4. Multiple comparison of cement groups with Tukey test.

(I) Cement	(J) Cement	Mean Difference (I-J)	Std. Error	Sig.
Zinc phosphate	Glass ionomer	-.5582	.41584	.376
	Composite resin	-2.9196	.41584	.000
Glass ionomer	Zinc phosphate	.5582	.41584	.376
	Composite resin	-2.3613	.41584	.000
Composite resin	Zinc phosphate	2.9196	.41584	.000
	Glass ionomer	2.3613	.41584	.000

3.3 Failure mode

The results for characterization of the failure mode are presented in Fig. 8.

For zinc phosphate cemented groups, the cement mainly remained on the tooth (category 1) and on the crown and the tooth (category 2). For glass ionomer cemented groups, the failure mode was generally in category 2 followed by cement was mainly on the crown (category 3) and category 1. For the groups luted with adhesive resin the category 3, followed by category 2. Only in two cases a root fracture occurred (category 4). Differences in failure mode between cement groups were highly significant (Pearson's chi-square test: $p < 0.001$). All air-abraded groups showed more cement remains on the crown or mixed than as-received groups luted with the same cement. Aging did not show a significant influence on the mode of failure.

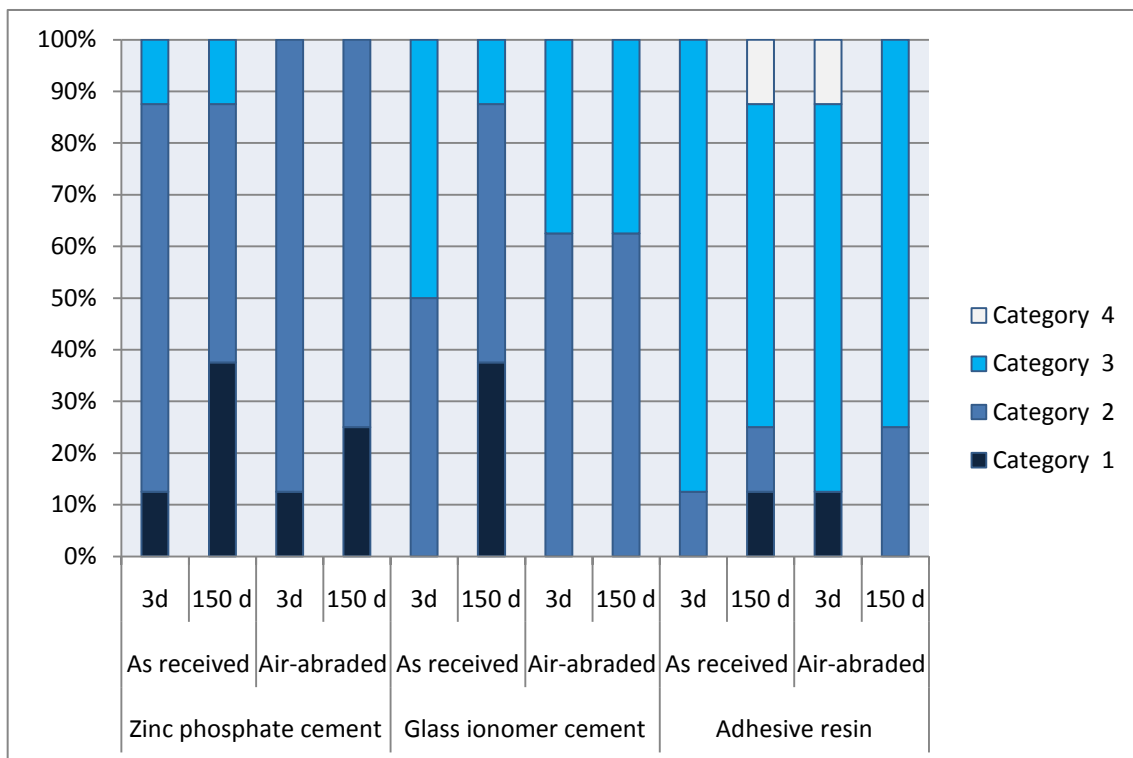


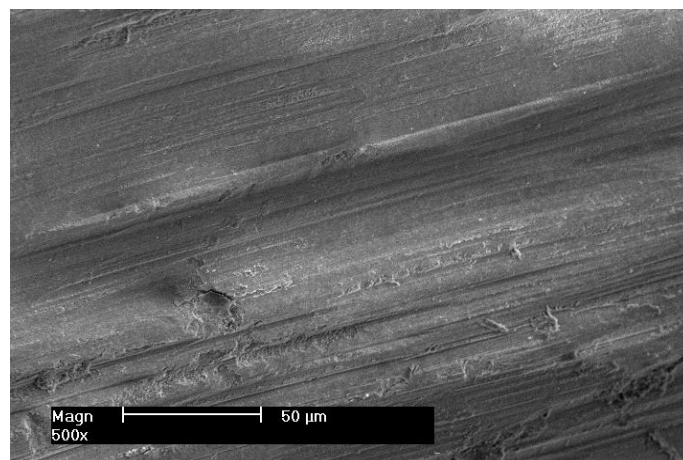
Fig. 8 Failure mode [%], for categories description see table 2.

3.4 SEM images

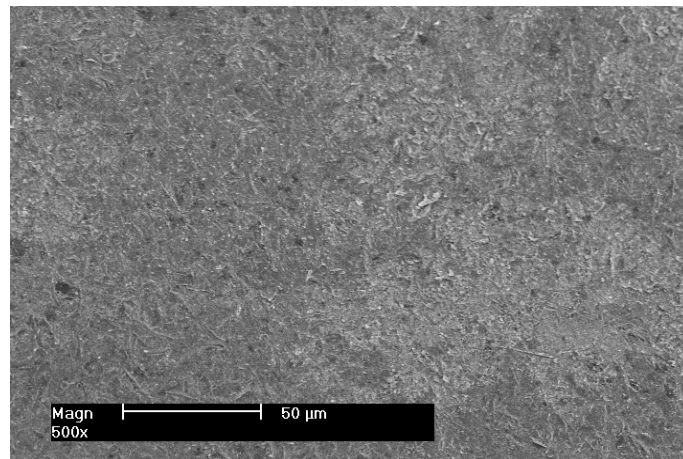
The SEM image of the intaglio surface of a zirconia ceramic crown is shown in Fig. 9a for the “as- received” condition. In this untreated condition, densely sintered particles of

high-purity zirconia ceramic providing some undercuts were observed. Fig. 9b shows the intaglio surface after airborne air-abrasion with 50 μm alumina particle for 15 seconds. This treatment produced a roughened surface grooving and sharp edges. There are also some clusters of sintered particles unaffected by the abrasive.

SEM images of the dentin after the dislodgment of zinc phosphate and glass ionomer cemented groups exhibited areas with a relative thick cement layer on some areas of the tooth surface (Fig. 10). Adhesive resin cemented crowns showed microscopic remnants of the adhesive material especially in the dental tubules (Fig. 11 a, b).



(a)



(b)

Fig. 9 (a) SEM image of the as-received surface of zirconia (machined) at x500 magnification. (b) SEM image of the air-abraded surface of zirconia at x500 magnification.

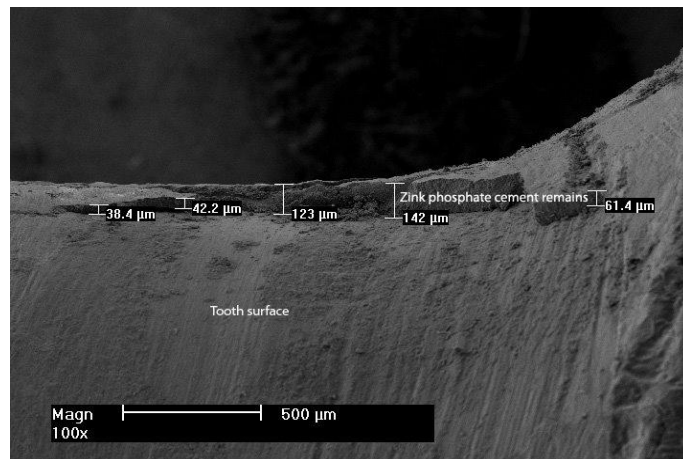
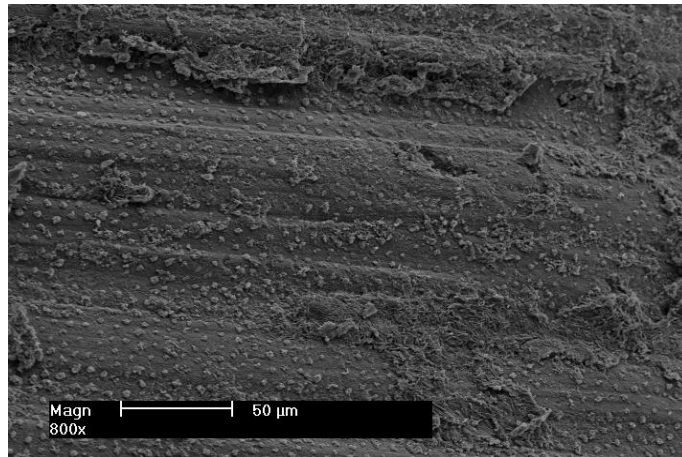
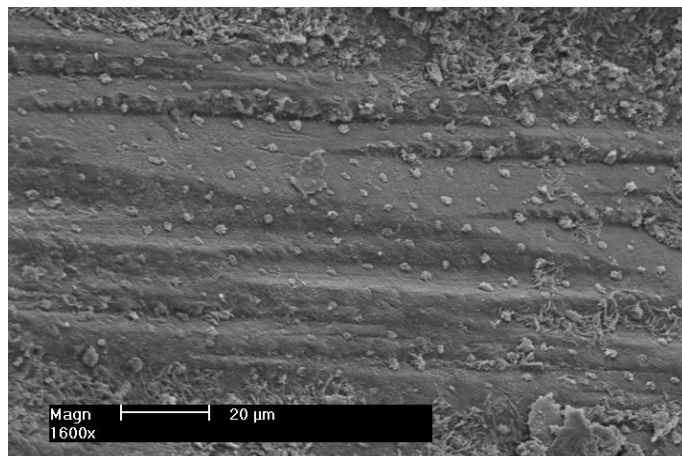


Fig. 10 SEM image of the surface of prepared tooth in the marginal area after dislodgment of a crown cemented with zinc phosphate cement. Cement remnants and the thickness of its layers are indicated.



(a)



(b)

Fig. 11 (a) SEM image of remnants of composite resin on the dentin surface after dislodgment at x800 magnification. (b) The remnants of composite resin on the dentin surface (especially in the dental tubules) after dislodgment at x1600 magnification.

4 Discussion

4.1 Discussion of the methods

In-vitro evaluation of retention of ceramic crowns with prepared human teeth and artificial aging simulating the mouth environment are able to provide some indications about the clinical durability of these restorations. Several other factors influence the retention of all-ceramic crowns, such as the fabrication technique, and the intaglio surface machining of the crowns.

4.1.1 Preparation design

A circumferential shoulder finish line of 0.8-1.5 mm depth was the finishing line usually used for all-ceramic crown preparation in several in-vivo and in-vitro studies [2]. Therefore, in this study a circumferential shoulder finish line with 0.8 mm depth was used. Different tapering angles were used for all-ceramic crown preparation, ranging from 4-20 degree [2]. However, an axial taper of 12 degree was used in this study as a very commonly used preparation taper. This degree of convergence was selected because a smaller preparation angle may have increased the retention resistance to crown removal regardless of the type of cement [79]. It might have also resulted in crown fracture instead of crown removal by the retention test.

4.1.2 Artificial aging

Long -term water storage and long-term thermal cycling are widely accepted conditions for testing the durability of luting systems in vitro. However, different luting systems are influenced differently by these two parameters [80]. In Addition, application of mechanical cyclic loading (masticatory simulation) might cause significant reduction of retention [44-45]. Therefore, a combination of these methods was used for artificial aging.

4.2 Discussion of the results

Crown retention in the current study was in a similar range as published in previous studies with zirconia ceramic crowns. Palacios et al. (2006) evaluated composite resin cement with adhesive cement (Panavia F 2.0), resin-modified glass ionomer cement, and a self-adhesive modified composite resin for luting air-abraded zirconia crowns to human molars. But neither non-abraded crowns nor long-term artificial aging were tested. Also, they used

only the axial surface of the preparation without the occlusal surface to calculate and convert the crown retention force in N to stress in MPa. However, the luting agents had a mean retention of 5.1, 6.1, and 5.0 MPa respectively. In the present study similar results were obtained after using air-abrasion and adhesive resin or glass ionomer cement after artificial aging, which were 6.1 and 4.8 MPa [52]. Ernst et al. (2005) used 12 different cement-surface treatment groups but did not test the influence of air-abrasion with alumina particles. The medians of retention found in this study were similar in range to the present study for two of the cements assessed in both studies (Panavia F with ED primer and Ketac Cem without conditioner) as the removal stresses for not air-abraded crowns were 4.0 and 1.8 MPa compared to 4.7 and 2.1 MPa in the present study. Also, Ernst et al. used the entire preparation surface to convert dislodgment force into retention stress in MPa [74].

In a more recent study (2009), Ernst et al. used similar methods to their investigation in 2005, but used different cements and one year water storage without thermal cycling for artificial aging. The median of retention of crowns ranged between 2.1 MPa to 7.5 MPa, which are also in the same range of our present study [81].

In the current study, due to the differences in shape and substance of the natural premolars, and the type of cements used, means of group retention showed varying standard deviations. In groups with adhesive resin cement standard deviations were smaller than in groups with conventional cements, while standard deviations increased after artificial aging. However, due to using natural teeth with their inherent variations, standard deviations were rather high in most of the study groups.

4.3 Influence of the luting cement on crown retention

ANOVA showed that retention was influenced significantly by the cement used. The adhesive resin showed a significantly higher retention than glass ionomer and zinc phosphate. However, no significant difference was found between the two conventional cements. The results are in agreement with Ernst et al. (2005) who found that composite resin cement demonstrated the highest median retentive strength. Also, Ernst et al. (2009) evaluated the retention of two resin cements (Panavia F 2.0, Multilink), two self-adhering cements (Rely X Unicem Aplicap, Maxcem), and a resin-modified glass-ionomer cement (Fuji Cem). After cementation to zirconium-oxide ceramic crowns on extracted human teeth and one year of

water storage, Ernst et al. found that the adhesive resin cements (Rely X Unicem Aplicap and Multilink) showed the highest median retentive strength values [81].

Using tensile bond strength test, Kern and Wegner (1998) evaluated different adhesion methods and their durability after long-term storage and thermal cycling. In their study, air-abrasion with 110 μm aluminum oxide, an application of silane, and silica coating with the Rocatec system were insufficient for achieving durable resin bond to zirconia ceramics. Only a phosphate-modified composite resin cement provided long-term durable bond and resulted in the high bond strengths values after air-abrasion, which agrees with the results of the present study [65].

Clinically, Sailer et al. (2007) in a systematic review of studies addressing the issue of loss of retention (fracture of the luting cement) showed that the standard Poisson distribution was with high failure rates when using conventional cements for luting zirconia crowns [67].

The findings of the present study are in agreement also with studies on luting to zirconia ceramic with different methods. Lüthy et al. (2006) used shear test specimens prepared by luting small cylindrical stainless steel rods (3 mm in height and diameter) of tribochemically silica-coated with the Rocatec system to air-abraded ceramic disks made of zirconia. A glass ionomer cement, a conventional BisGMA resin composite, a 4-META adhesive resin, and three adhesive resin composites containing phosphoric acid monomers, were used as the luting agents. The results of this study showed that retention for glass ionomer cement was quite low. The four adhesive resin cements gave the best results. The highest retention to air-abraded zirconia was obtained with one adhesive resin (Panavia 21) [36].

Uo et al. (2006) investigated the shear bond strength of zirconia ceramic (Denzir) specimens machined into a rectangular shape, luted together with 3 cements: Zinc phosphate cement, glass ionomer cement, and adhesive resin composite cement. Uo et al. found that the resin composite cement exhibited significantly higher bond strength than the glass ionomer cement. However, glass ionomer cement showed significantly higher bond strength than zinc phosphate [82].

In disagreement with the present study, Palacios et al. (2006) using crowns luted with 3 cements, Rely X Unicem, Rely X Luting, and Panavia F 2.0 with ED Primer, found that the

use of composite resin cement with a dentin bonding system did not yield greater retention as comparative to modified glass ionomer cement [52]. However, Palacios et al. used prepared human teeth with a relative small taper of 10° which can boost the mechanical retention of the crowns. This mechanical retention can specially improve the retention of the conventional luting agents [83-85], which explains the difference of the current study.

As well, with two systems of all-ceramics, Söderholm et al. (2003) evaluated the retention of two all-ceramic crown systems (aluminum oxide (Procera) and zirconium oxide (Denzir)) cemented on metal dies with zinc phosphate cement as one luting agent in the study. Based on good clinical results with zinc phosphate cemented Procera crowns and comparable results for both ceramic crowns, Söderholm et al. predicted that zinc phosphate cement luted Denzir crowns are likely to perform well clinically. However, Söderholm et al. used for their study metal dies with 3° taper and very high mechanic-retentive surface, which can boost the crown retention regardless of the cement used [22]. That explains again the differences to the current study.

In conclusion, the hypothesis, that zirconia ceramic crown retention is not influenced by the luting material must be rejected.

4.4 Influence of air-abrasion on crown retention

ANOVA showed that air-abrasion influenced crown retention regardless of the cement used. The air-abraded groups showed significantly higher retention than as received groups ($p < 0.001$). The increase of retention corresponds to the increase of micro-roughness on the surface of the air-abraded zirconia [53, 82]. Surface roughness enhances the micromechanical interlocking of luting agents to ceramic surfaces [86].

Blatz et al. (2007) evaluated in an in-vitro study, short- and long-term shear bond strengths of four luting agents to zirconia following different surface treatments: left untreated, air-abraded, Rocatectribochemical silica/silane coated, or ground and polished. They concluded that air-abrasion combined with a resin composite containing adhesive phosphate monomers provides superior long-term shear bond strength values [48]. Guazzato et al. (2005) [49] investigated the influence of air-abrasion (110 μm alumina particles at a pressure of 5 MPa for 20 seconds), grinding, grinding orientation, polishing and heat treatment on the flexural strength of a Y-TZP ceramic using beams from a block of DC-

Zircon cut with a diamond blade. Their results suggest that air-abrasion and grinding may be recommended to increase the strength of dental Y-TZP. Fine polishing may remove the layer of compressive stresses and therefore, lower the mean flexural strength.

In contrast to our data, in a recent study Derand et al. (2008) [57] stated that air-abrasion reduced crown retention of alumina and zirconia copings. The author reported that untreated zirconia copings cemented with zinc phosphate showed significantly higher retention than the copings with air-abraded surfaces, and illustrated that air-abrasion decreased the retention of zirconia and alumina copings. However, Derand et al. performed their experiment with air-abraded roughened steel dies with a surface area of 112 mm², which will provide very high mechanical retention. This mechanical retention can specially improve the retention of conventional luting agents (zinc phosphate cement) [83-85]. Also, the pressure used for air-abrasion of zirconia was relatively high (5 bar) compared to the pressure used in the present study (2.5 bar). High pressure air-abrasion might alter the luting surface of zirconia and lead to a weakening of the retention properties.

In conclusion, the hypothesis that air-abrasion does not influence crown retention has to be rejected.

4.5 Influence of artificial aging on crown retention

Artificial aging with thermocycling and masticatory simulation decreased crown retention regardless of the cement used. These results can be compared to other studies only to a limited extent as the authors of this investigation did not find any published scientific studies evaluating the long-term artificial aging effect on the retention of zirconia ceramic crowns cemented to human teeth. Our results conflict with the findings of a recent study of Ernst et al. (2009) in which the retention of zirconia ceramic crowns was not reduced during one year of water storage without long-term thermocycling and masticatory simulation [81]. However, stresses induced by long-term thermal cycling and masticatory simulation might be responsible for the reduction of retention in the current study which simulated the clinical conditions more closely than one year of water storage alone as used in the other study.

Our results are in agreement with many other studies on luting to zirconia ceramic [48, 87-89] which showed a decrease in bond strength after artificial aging. This decrease in the retention can be explained by material fatigue as result of microleakage, changes in the elastic

modulus, and plastic deformation over the time under thermal cycling and mechanical loading [90-91].

Therefore, also the third hypothesis that artificial aging does not influence crown retention has to be rejected.

4.6 Failure modes

In the present study, the failure type showed significant differences between the three luting cements. Also it was partially affected by air-abrasion in the three luting materials, in which the mode of failure shifted slightly towards “cement on both crown and tooth” or “on crown”, while aging did the opposite effect in some groups.

With the zinc phosphate cement the failure was mostly on both crown and tooth, followed by cement remaining on the tooth. In the glass ionomer cemented groups the cement was mostly on both crown and tooth, followed by cement on crown. With adhesive resin cement the mode of failure for 75% of specimens was cement residing principally on the crown, followed by cement found principally on the tooth and the crown for 12.5% of the specimens. Type of tooth substrate, enamel or dentin, seems to have an impact on the type of failure. In clinical long-term studies with adhesively luted glass-ceramic crowns, Kinnen et al. (2006) found that loss of retention was caused mainly by loss of adhesion at the dentin–resin interface [92]. The mode of failure with adhesive resin corresponds well to the high bond strength between zirconia surface and the phosphate-modified resin cement [48, 58, 65].

SEM evaluation of the failure mode showed that there are still remains of the adhesive resin across the entire dentin surface and especially filled in the dentin tubules, which can be considered as cohesive failure in the resin itself. In conclusion higher crown retention corresponded to more cohesive failures within the luting material.

5 Conclusions

The results of this study may have important clinical implications, since there has been a lack of clear information for standard procedures for the cementation of zirconia ceramic restorations.

However, the limitations of laboratory studies, which may not completely simulate in vivo performance, must be considered. Further research is needed to examine various surface treatments and luting cements. In addition, long-term prospective and randomized clinical trials are needed to evaluate the benefits of certain clinical procedures, including this innovative type of all-ceramic restoration.

Within the limitations of this study, the following conclusions can be drawn:

1. Within this common luting agents (zinc phosphate, glass ionomer, and adhesive resin), a phosphate monomer containing luting resin showed a significantly higher crown retention than conventional cements.
2. Air-abrasion of the crown surface improves the crown retention regardless of the luting material used. Roughening and activating the surface by air-abrasion with alumina particles prior to adhesive bonding and the use of a phosphate monomer containing resin composite is required to achieve durable bond to zirconia ceramics.
3. Long-term artificial aging with mechanical loading decreased crown retention regardless of the luting material used.

6 Summary

Objectives:

The purpose of this in vitro study was to evaluate the effect of intaglio surface air-abrasion on the retention of CAD/CAM produced zirconia ceramic crowns cemented with three different types of cement. In addition, the influence of artificial aging in masticatory simulator and thermocycling was tested.

Methods:

Extracted human premolars were prepared for all-ceramic crowns (12° taper, 3mm axial length). CAD/CAM zirconia crowns were manufactured. Half of the crowns were air-abraded with 50 µm alumina particles at 0.25 MPa, the rest was left as machined. The crowns were luted with zinc phosphate cement (Hoffmann, Hoffmann, Berlin, Germany), glass ionomer cement (Ketac Cem Maxicap, 3M Espe, Seefeld, Germany), or composite resin (Panavia 21 TC, Kuraray, Osaka, Japan), subgroups were either stored for 3 days in 37° water bath or stored for 150 days in 37° water bath, with additional 37,500 thermal cycles (5°-55°) and 300,000 cycles dynamic loading with 5 kg in a masticatory simulator. Then crown retention was measured in tension at a crosshead speed of 2 mm/min using a universal testing machine. Statistical analysis was performed with three-way ANOVA.

Results:

Mean retention values ranged from 2.8 to 7.1 MPa after 3 days and from 1.6 to 6.1 MPa after artificial aging. Air-abrasion significantly increased crown retention ($p < 0.001$), while artificial aging decreased retention ($p = 0.017$). In addition, the luting material had a significant influence on retention ($p < 0.001$) with the adhesive luting resin providing the highest retention.

Conclusions:

A phosphate monomer containing luting resin provided significantly higher retention of zirconia ceramic crowns than conventional luting cements. In addition, air-abrasion of the crown surface improved the crown retention regardless of the luting material used. Also, long-term artificial aging with mechanical loading decreased crown retention regardless of the luting material used.

7 Zusammenfassung

Einfluss von Korundstrahlung und Zement auf die Retention von Zirkonoxidkeramik-Kronen vor und nach künstlicher Alterung.

Ziele:

Zweck dieser In-vitro-Studie war es, den Einfluss des Abstrahlens der Innenflächen von Zirkonoxidkeramik-Kronen auf deren Retention unter Verwendung von drei verschiedenen Zementen zu untersuchen. Zusätzlich wurde der Einfluss einer künstlichen Alterung in einem Kausimulator und durch Thermocycling getestet.

Methode:

Sechsendneunzig extrahierte menschliche kariesfreie Prämolaren wurden für Vollkeramik-Kronen nach klinischen Parametern präpariert (12° Präparationswinkel, 3 mm Stumpfhöhe, 0,8 mm Hohlkehle). Vollkeramische Kronen aus Zirkonoxidkeramik wurden hergestellt (Vita In-Ceram YZ for inLab, Vita, Bad Säckingen) unter Benutzung eines CAD/CAM-Gerätes (Cerec 3, Sirona, Bensheim).

Die Hälfte der Kronen wurde mit 50 µm Aluminiumoxid-Partikeln bei 0,25 MPa für 15 Sekunden abgestrahlt, der Rest wurde nicht weiter konditioniert.

Die Kronen wurden mit Zinkoxidphosphat-Zement (Hoffmann, Berlin), Glasionomer-Zement (Ketac Cem Maxicap, 3M Espe, Seefeld) oder einen Phosphatmonomer haltigen Kompositkleber (Panavia 21 TC, Kuraray, Osaka, Japan) gemäß der Anweisungen der Hersteller zementiert.

Untergruppen jeder Zement-Gruppe wurden entweder für 3 Tage in einem 37° Wasserbad gelagert oder für 150 Tage gelagert und zusätzlich mit 37.500 Thermozyklen (5°-55°) und weiteren 300.000 Zyklen dynamischer Belastung mit 5 kg in einem Kausimulator künstlich gealtert. Die statistische Analyse wurde mittels dreifaktorieller ANOVA durchgeführt.

Ergebnisse:

Die Mittelwerte der Kronen-Retention reichten von 2,8 bis 7,1 MPa nach 3 Tagen und von 1,6 bis 6,1 MPa nach künstlicher Alterung. Abstrahlen der Retentionfläche mit Aluminiumoxid erhöhte signifikant die Kronen-Retention ($p < 0,001$), während künstliche Alterung die Retention verringerte ($p = 0,017$). Außerdem hatte der Zementtyp einen signifikanten Einfluss

auf die Retention ($p < 0,001$), wobei der Phosphatmonomer haltige Kompositkleber die höchste Retention aufwies.

Schlussfolgerung:

Die Verwendung von Phosphatmonomer erdhaltigen Kompositkleber auf abgestrahlter Zirkonoxidkeramik kann als zuverlässigste Befestigungsmethode klinisch empfohlen werden.

8 Literature

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9 Appendix

Curriculum Vitae

Personal Data

Name: Ramez Shahin

Address: Frerichsstr. 10
D-24116 Kiel

Tel. +49 431 2375397

E-mail: rshahin@proth.uni-kiel.de

Date of Birth: 31.03.1971

Place of Birth: Damascus, Syria

Nationality: Syrian

Family Status: Married

Languages

English: Fluently, German: Fluently, Arabic: Mother language.

Professional Experience

2006- 2010 Visiting research fellow in the department of Prosthodontics, Propaedeutic and Dental Materials, School of Dentistry in Christian-Albrechts University at Kiel.

2002-2006 Dentist in the Aleimes policlinic, Aleimes Medical Group, Gizan, Saudi Arabia.

2000- 2001 Assistant professor in the Department of Prosthodontics, Tishreen University, Lattakia, Syria.

1999- 2000 Resident in the Department of Oral Surgery in Zahi-Azrak military hospital at Lattakia, Syria.

1996- 2000 General practitioner in private dental clinic, Lattakia, Syria.

Academic Qualifications

- 1990- 1995 DDS “Doctor in Dental Science” in Tishreen University, Lattakia, Syria.
1990 High School in Lattakia, Syria.

Memberships

- Since 2009 Member of the International Association for Dental Research IADR, and its European division CED-IDAR.
2007 Member of the General Dental Board (Zahnärztekammer) in the State of Schleswig-Holstein, Germany.
2002 Member of the Saudi Council for Health Specialties, Saudi Arabia.
2000 Member of the Teaching Board of the Tishreen University, Lattakia, Syria.
1996 Member of the Syrian Dental Board.

Scholarships

- 2006- 2008 DAAD “Deutscher Akademischer Austausch Dienst” for Doctoral Studies.

Publications

Shahin, R., Kern, M.: Effect of air-abrasion on the retention of zirconia ceramic crowns luted with different cements before and after artificial aging, *Dental Materials* 26, 922-928 (2010).

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Danksagung

Mein Dank für die Unterstützung bei der Erstellung meiner Doktorarbeit geht vor allem an meinen Doktorvater Professor Dr. Kern, der die Durchführung dieser Studie ermöglicht und mir viele hilfreiche Anregungen und wissenschaftliche Beratung gegeben hat. Darüber hinaus möchte ich auch den Mitarbeitern des wissenschaftlichen Labors in der Klinik für zahnärztliche Prothetik: Prof. Ludwig, Dr. Steiner, Herr Lehman, Herr Gostomsky und Herr Möller, sowie den Mitarbeitern des dentalen Labors für die Unterstützung danken.

Auch möchte ich mich bei meiner Frau bedanken, die mich nicht nur tatkräftig unterstützt hat, sondern mich stets aufbaute und für die erforderliche Abwechslung sorgte.