Resin-based cements and substrate surfaces –

an evaluation of critical factors for bond strength

Doctoral thesis for the degree of Philosophiae Doctor

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"Above all, don´t fear difficult moments. The best comes from them."

Rita Levi-Montalcini

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List of publications

Paper I

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Paper II

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Paper III

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Abbreviations

Al_2O_3	Aluminum oxide
bis-EMA	Ethoxylated bisphenol-A dimethacrylate
bis-GMA	Bisphenol-A glycidyldimethacrylate
BPDM	Bisphenyl dimethacrylate
CAD	Computer-aided design
CAM	Computer-aided manufacturing
HEMA	Hydroxyethyl methacrylate
HF	Hydrogen fluoride
KHF_2	Potassium hydrogen difluoride
LDS	Lithium disilicate glass ceramic
MDP	10-Methacryloyloxydecyl dihydrogen phosphate
Sa	Arithmetical mean height
SEM	Scanning electron microscope
SiC	Silicon carbide
SiO_2	Silica
TEGDMA	Triethyleneglycol dimethacrylate
UDMA	Urethane dimethacrylate
Zir A	Zirconia airborne particle abraded with aluminum oxide
Zir E	Zirconia hot etched with potassium hydrogen difluoride

1 Introduction

1.1 Adhesion or bonding?

Creating a strong, lasting bond of indirect dental restoration to tooth substance is crucial for the durability of the treatment. The bonds between cement and indirect restoration, and cement and tooth substance, have been popular research subjects for decades (1-8). When cementing ceramic restorations to tooth substance using resin cement, challenges relate to the structure and chemistry of both the ceramic and the tooth surface, and also to the mechanical and chemical properties of the cement systems. Bond strength testing is widely used for studying the effect of different interventions in ceramic-cement and cement-tooth substance interfaces, and in the cement itself (9). In addition, determining fracture morphology after bond strength testing gives an indication of the weakest link and where effort should be placed for increasing bond strength.

Adhesion and bonding are terms commonly used in research describing the bond of indirect restoration to tooth substance—even though their meaning is different, they are often used interchangeably about the same procedure. There is a need to bring clarity to this terminology in scientific writing.

Adhesion is defined as the interfacial contact between two dissimilar surfaces which accrues because of forces between them. The forces that cause adhesion might be 1) mechanical, 2) chemical, 3) dispersive, 4) electrostatic or 5) diffusive (10). When cementing indirect restorations, adhesion occurs in the interface between cement and restoration and between cement and tooth substance. It is mostly mechanical, in the establishment of interlocking, and chemical in the presence of covalent, van der Waals, acid-base or hydrogen bonds (10).

Cohesion is also a term often used in research concerning the bond of dental restorations to tooth substance and should be defined to distinguish it from adhesion. Where adhesion concerns forces between dissimilar surfaces, cohesion covers forces between similar or identical surfaces. It is the intermolecular forces that hold the particles in the material together (11). When considering failure in the bonding of materials, it is useful to determine differences between adhesive or cohesive fractures to establish the best course for intervention.

Bonding can be defined as surfaces attached together using an adhesive, which is responsible for joining materials by forming bonds with each surface (10, 11). When cementing ceramic restorations, the result is bonding of the restoration to tooth substance where the cement serves as an adhesive.

In this thesis, the term adhesion is used when interfaces between resin cement and ceramic and resin cement and tooth substance are described and discussed. The term bonding is used to describe and discuss the retention established when ceramic restorations are cemented to tooth substance using resin cement.

1.2 Tooth substance

When restoring teeth using an indirect technique it is preferable to place the preparation in enamel to ensure retention. Enamel contains about 96% minerals, mainly crystalline hydroxyapatite, which makes it the hardest tissue in the body (12). Etching of enamel using phosphoric acid selectively dissolute crystalline minerals to create a roughness in the surface for mechanical adhesion of cement (12, 13). When restoring teeth with extensive substance loss, the preparation must be placed in dentin, which is a more complex tissue compared to enamel. Dentin consists of 50-70% apatite, 20-30% organic material (mainly as collagen type 1), and 10-20% water (14, 15). The main structure in dentin consists of the tubules, which represent the tracks taken by dentin forming cells (odontoblasts) from the dentin-enamel-junction to the pulp. Tubules are lined with apatite crystals in a so-called peritubular dentin layer, and apatite-reinforced collagen in intertubular dentin separate tubules. The density and dimension of the tubules increase through the dentin, from 20 000 tubules/mm^2 at the dentin-enamel junction to 45 000 tubules/mm^2 closer to the pulp (13). Due to the tubules, dentin is a highly permeable tissue and its permeability increases towards the pulp and dentinal fluid and intra pulpal pressure are moistening the prepared dentin surface. After primary dentin formation, odontoblasts line the pulp chamber and are responsible for a life-long formation of secondary and tertiary dentin. These gradual changes in dentin affect properties like permeability, moisture and the area available for adhesion (16).

1.3 Dental cements

Water-based cements—for instance, zinc phosphate—were previously the first choice when cementing indirect restorations (17). An acid-base-reaction takes place when zinc oxide powder and a eugenol-containing liquid is mixed, and a zinc phosphate chelate is formed (18). Glass ionomer cement, which is another extensively used water-based cement, also undergoes an acid-base-reaction where the calcium fluoro aluminosilicate glass is brought in contact with a polyacrylic acid forming a hydrogel. Some glass ionomer cements are modified with resin monomers for improved mechanical properties (18). Water-based cements are still in use today but should be limited to cementing high-strength materials such as cast metal, metal-ceramic and polycrystalline ceramic restorations. Low-strength all-ceramic materials have shown reduced fracture strength when cemented with water-based cements compared to resin-based cements (17, 19).

Resin-based cements are composite materials mainly consisting of an organic resin matrix, filler particles and adhesive monomers. Their composition is similar to composite for direct restorations, but viscosity, filler distribution and initiator content are adjusted to obtain a thin cement layer and optimal handling time (18). The resin matrix is composed of monomers—most often dimethacrylate monomers like bisphenol-A glycidyldimethacrylate (bis-GMA), ethoxylated bisphenol-A dimethacrylate (bis-EMA) or urethane dimethacrylate (UDMA)—because of their high strength and low polymerization shrinkage (20, 21). Other monomers, like triethyleneglycol dimethacrylate (TEGDMA), are added to modify the viscosity and degree of conversion (22). Monomers in the resin matrix are responsible for cohesion of the composite components, but their shrinkage during polymerization affects the mechanical properties and marginal sealing (23). In addition, adhesive monomers—like10-methacryloyloxy-decyl-dihydrogen-phosphate (MDP) and 4-methacryloxyethyl trimellitic anhydride (4-META)—are added to resin cements because of their ability to chemically adhere to hydroxyapatite and zirconia (18). The fillers are inorganic quartz, glass or ceramic particles covered by a coupling agent that promotes the bond to the organic resin matrix. The filler content is 30-70% of the cement volume and affects its viscosity and ability to penetrate into irregularities in the substrate surface (24). Size, shape and distribution of the particles vary greatly and influence properties like flexural strength and fracture toughness of the cements (25).

Resin cements cure by free-radical polymerization and are available in light-curing and combined light- and chemically curing modes, dependent on the initiators added (18, 26). In exclusively light-cured cements, free radicals are generated through light activation of camphorquinone and aliphatic amines, and a densely cross-linked polymer is formed (26). Light-curing cements are only used for cementing restorations with a thickness up to 1 mm because of their dependency on light transition for a high degree of conversion. For thicker restorations, cements with dual-cure mode are recommended. These cements have a selfcuring activator—benzoyl peroxide—added to the catalyst part of the cement, which undergoes a chemical reaction with tertiary amines, in addition to light-curing initiators (26). The degree of conversion of dual-cure cements increases when both curing modes are applied (18, 26).

Resin cements support the overlying glass ceramic restoration and increase fracture strength by chemically uniting the ceramic restoration and the cement (24, 27, 28). Their translucency and shade matching also make them suitable for cementing thin ceramic restorations in the aesthetic zone.

1.4 Ceramics in dentistry

Ceramics were introduced to the dental market in the 18th century when porcelain replaced ivory and wood in denture teeth. Later, in the 19th century, porcelain was used for fixed restorations but, because of the brittleness of the material, its application was limited (29). Restoration with porcelain fused to metal (PFM) expanded and revolutionized the field of prosthetic dentistry in the 1950s after problems related to thermal expansion had been solved by adding leucite (30). PFM represented the first choice and gold standard for fixed prostheses for decades because of acceptable aesthetics and improved mechanical properties (31). Several improvements—such as inclusion of filler particles and stabilizers and better production methods—have revolutionized and expanded the use of all ceramic restorations. Today, all ceramic materials with different properties and areas indicated for use are available (32, 33). High-strength polycrystalline ceramics are used for both single-tooth restorations and fixed partial dentures, but when restoring teeth in the anterior region, glass ceramics are often preferred for aesthetic reasons (34, 35).

Reasons for the extensive use of ceramics in dentistry are numerous. The most obvious advantage is aesthetic, especially for the glass ceramic restorations. Translucency, light transmission and colour matching give restored teeth a natural look (31, 36). In addition, ceramics are biocompatible and have desired mechanical properties such as high elasticity modulus, low thermal conductivity and good wear resistance (32, 37, 38). Ceramics can be bonded to tooth substance using resin cement, which reduces the need for substance removal to create retention and resistance properties of the prepared tooth. When restorations are made one-layered—so-called monolithic—even less substance removal is needed. This lowers the incidence of biological complications, such as pulpal infection, that could lead to the need for further treatment (39).

Alongside the advantages of ceramic as a restorative material, there are some disadvantages. Because of the ionic and covalent atomic bonds that hold the ceramic together, the material is brittle and prone to fractures, especially when exposed to tensile forces (40). During production, cracks and flaws are incorporated in the material and on the surface, which might initiate crack propagation and potentially catastrophic failures (41).

Classification of ceramics is useful considering education and communication between parties involved in patient treatment and restoration production (42). Numerous systems have been suggested but, as new materials with new properties are introduced to the market, classifying becomes complicated. Aesthetic, indication for use, ability to be etched by hydrofluoric acid, mechanical properties and production method are just some ways of classifying ceramics (42). But according to Kelly and Benetti (43) there are only three main classes of ceramics, and they suggest a classification system based on structure; 1) predominantly glassy materials; 2) particle-filled glasses; 3) polycrystalline ceramics. A classification based on structure is useful in understanding material quality, area indicated for use and clinical handling. Since the ceramic materials used in the three studies in this thesis fall in categories 2 and 3, Kelly and Benetti´s classification system is used.

The atomic structure in ceramics is partly glassy (unorganized) and crystalline (organized), and the proportions of the two structures determine mechanical and optical properties (38). The predominantly glassy materials consist of a mixture of the minerals feldspar, silica (SiO_2) and alumina (Al_2O_3)—arranged mainly in an unorganized, amorphous structure—and some part crystalline (38). These types of ceramics are brittle and have low flexural strength but are highly aesthetic because of their colour and translucency (44). To improve the mechanical properties, leucite—or more commonly today, lithium disilicate—particles are added to the glass ceramics. The particles are arranged in a crystalline structure, which improves the mechanical properties, for instance increases the fracture strength (38).

The polycrystalline ceramic used today is mainly zirconia. The atomic structure is up to 99% crystalline, with no glass content (38). These ceramics have high flexural strength and high fracture toughness but are rather opaque, which limits their use in the aesthetic zone unless they are covered with a glass ceramic (45). The introduction of translucent polycrystalline zirconia with altered crystal structure and increased amount of stabilizing oxides has extended the range of use and reduced amounts of substance removal needed to restore teeth (46). Oxides, such as calcium oxide, magnesium oxide, cerium oxide or, more often, yttrium oxide, are added to stabilize zirconia by reducing transformation between crystal phases in the ceramic (37). The three different crystal phases—monoclinic, tetragonal and cubic—are stable at different temperatures and exhibit different mechanical properties, with the tetragonal phase being superior. Transformation of the crystal phase in stabilized zirconia might still accrue due to external stresses, like grinding or airborne particle abrasion (47). During transformation from the tetragonal to the monoclinic phase, up to 3-4% volume expansion takes place, which contributes to inhibiting both crack propagation and weakening of the material. This is known as transformation toughening and is one of the reasons for the favourable mechanical properties of zirconia (48, 49). But phase transformation might also accrue spontaneously in humid environments, which is known as low temperature degradation or ageing (50). Mechanical properties are deteriorated by phase transformation initiated on the surface creating micro cracks which are further penetrated by water, and the phase transformation penetrates the bulk of the material. Exposing restorations to further stress, like chewing, might result in catastrophic failure (37, 50, 51).

The increased translucency of novel zirconia materials is due to decreased grain size and higher amounts of yttrium oxide (up to 8 mol%) leading to an increase in cubic crystal structure (45, 52, 53). A higher resistance to phase transformation and ageing has been shown for translucent zirconia in some studies (52, 54), which would be an advantage concerning the stability of the material (47). However, a drawback is reduced transformation toughening, resulting in decreased flexural strength and fracture toughness compared to zirconia with 3 mol% yttrium oxide (53, 55).

1.5 Adhesion to dentin

For creating stable retention of indirect restorations cemented with water-based cements the tooth must be prepared with retentive and resistant properties. In addition, micro mechanical adhesion is established by roughness in the tooth surface that creates an interlocking of the cement (56). An advantage with glass ionomer cement compared to zinc phosphate, is its ability to adhere to tooth substance through interaction with hydroxyapatite, even though the adhesion created is not strong enough to significantly increase retention of the restoration (56).

The adhesion between resin-based cement and dentin is mainly mechanical, with resin infiltration of exposed collagen fibrils creating a so-called hybrid layer and forming tags in open tubules (57). Creating a stable adhesion between dentin and resin cement is, however, challenging due to the complexity and variation of the dentin structure (13). Moisture deteriorates the resin bond and, in proximity to the pulp, dentin permeability is higher due to the tubule structure, therefore creating a stable bond to the deeper section of dentin is difficult (58, 59). Roughness after preparation of the dentin surface also contributes to the interlocking of the cement. Different grit dental burs result in different roughness, which affects both bond strength and fit of the restorations. Still, there is no consensus about the optimal grit for tooth preparation for indirect restorations (60, 61).

Resin cements are commonly grouped according to their adhesive method. Etch-and-rinse refers to three- or two-step systems where the tooth substance is first etched by phosphoric acid (30-40%) and thoroughly rinsed with water to create a retentive surface with smear layer removed. Thereafter, primer and bonding are applied in one or two steps before placement of cement and restoration (62). Primers contain hydrophilic monomers in evaporative solvents that wet the tooth surface and penetrate into dentin tubules (13), while bonding is a hydrophobic adhesive resin that forms tags in tubules and roughness and further co-polymerizes with the applied cement. The three-step etch-and-rinse method has been the gold standard for adhesion to tooth substance for decades. It creates a strong, durable interface with both enamel and dentin (13, 63), but the method is time-consuming and prone to error. Also, phosphoric acid etching might demineralize several micrometres of dentin, and if this layer is not completely infiltrated by the resin, adhesion might be compromised (64). Less time-consuming adhesive methods consist of two or one steps pre-treatment of tooth substance as a part of so-called self-etching systems. Acidic methacrylates in solvents are either applied prior to an adhesive resin consisting of dimethacrylates, or all components are combined in an all-in-one method.

Monomers in adhesives are mainly cross-linkers that form polymers and functional monomers that ensure wetting of dentin and chemical adhesion to calcium in hydroxyapatite (20). 2-hydroxyethyl methacrylate (HEMA), bis-GMA and 4-META monomers are often added to adhesives due to their adhesion-promoting properties (20, 65), but in recent years MDP has become a popular monomer because of its strong bond to calcium and formation of low-dissolution calcium salt (20), and the belief that its adhesive stability is higher than for other monomers (64).

Even cements with self-adhesive properties that require no pre-treatment of the tooth substance are available for cementing indirect restorations (66). These cements consist of two-compartment capsules—with one-part acidic monomers and one-part resin monomers and filler particles—which are activated and auto-mixed to form a homogenous cement. The acidic monomers etch the tooth substance and create a chemical bond with calcium in hydroxyapatite (18), while the resin monomers polymerize, crosslink and bind to filler particles.

Scanning electron microscopy (SEM) studies have shown that, when using a self-etching adhesive method, the etching pattern in dentin is shallow and more irregular compared to etching with phosphoric acid (67). For self-adhesive cements, the interaction with tooth substance is even more superficial (68). One would expect that this affects the retentive properties of the cement system (13), but comparative laboratory studies have shown that less time-consuming methods can perform equally as well or even better than the three-step gold standard in bond strength testing (56).

1.6 Adhesion to ceramics

A common feature of glass ceramics and particle-filled glass ceramics is their sensitivity to hydrofluoric acid (HF) (69). Etching with HF dissolves glass on the ceramic surface and results in a roughness that promotes mechanical adhesion (18, 70). HF concentration, etching time, liquid or gel, and characteristics of the ceramic all affect the adhesive strength to resin cement. An increase in concentration should be balanced by a reduction in etching time (71). After etching, the surface is left with high energy, which promotes wetting by the silane coupling agent applied before cementation (71). Silane is available in nonhydrolyzed form, consisting of two components mixed before application, or as one component (prehydrolyzed silane). When applied to the ceramic surface, silane forms covalent siloxane bonds with oxides, and carbon double bond with the resin cement, which form the basis for adhesive cementation (18).

The crystal structure and lack of glass particles in zirconia make the material resistant to etching with HF. Micro roughness in the surface for mechanical retention must therefore be established in a different manner. Surface treatment of zirconia is a popular field of research but there is still no consensus about the optimal method. Airborne abrasion using Al_2O_3 particles has most evidence in the literature (47, 72, 73). Particles sized in the range 30 to 110 μm are used in performed studies (73), but it is argued that the particles should preferably be between 30-50 μm to reduce damage to the material. Also, the pressure should be kept low, with a maximum of 2.5 bar. The nozzle should be held at a 10 mm distance to the ceramic surface and moved in circular motions, all this to avoid damages to the ceramic material (74). Tribochemical SiO_2-coating is another established method that creates micro roughness using Al_2O_3, and in addition leaves a layer of SiO_2 on the surface for chemical adhesion (73, 75). Other roughness creating methods are grinding using diamonds or silicon carbide (SiC) papers of different grit, laser irradiation and hot etching (72, 73, 76, 77).

A concern when using all types of abrasive techniques on zirconia is possible structural damage (from tetragonal to monoclinic phase transformation) and the introduction of flaws that might compromise the material integrity and reduce fracture strength (47, 77, 78). Because of this, and the desire to create chemical adhesion between zirconia and resin cement, inclusion of glass particles in the surface, either by coating, heat infiltration or sputtering, has been tested—but the adhesion is unstable (18, 73). Phosphate monomer containing primers, adhesives and cements have been presented as a solution to challenges related to chemical adhesion between zirconia and resin cement. It is believed that chemical adhesion is established between the zirconia surface and acidic monomers, and that the monomers further copolymerize with resin cement due to methacrylate groups. MDP is the most used phosphate monomer and is incorporated in ceramic primers, dental adhesives and resin cements (18). The monomer is adsorbed on the zirconia surface forming Zr-O-P bonds. Results from in vitro testing show increased adhesive strength when using MDP primer. Still, the adhesive strength is concentration-dependent, reflected in a reduced frequency of adhesive fractures between ceramic and cement with increased MDP concentration (79). Artificial ageing also affects the adhesive strength, which has been shown to be reduced after both thermocycling and water storage (80).

Creating surface roughness and the potential for chemical adhesion to resin cement by hot etching of the zirconia surface using potassium hydrogen difluoride (KHF_2) was first studied by Ruyter et al. (76). Dry in vitro testing resulted in considerably higher bond strength of KHF_2 etched zirconia compared to an airborne particle abraded surface, even though the differences were insignificant. Hjerppe et al. (81) performed both dry and wet bond strength testing of KHF_2, which led to results equivalent to more established methods like particle abrasion with and without silica.

Quantitative crystallite analysis has shown that the fraction of monoclinic and cubic crystal structure is lower for KHF_2-etched than particle-abraded zirconia, indicating that the etching method could be favourable for the mechanical properties (76). Oilo et al. (55) also observed a higher fraction of tetragonal crytal structure after KHF_2-etching compared to particle abrasion. Akazawa et al. (82) also showed that hot etching results in higher surface energy and wettability of zirconia compared with particle abrasion. With this in mind, hot etching of the zirconia surface using KHF_2 might be a method for reducing stress-induced flaws in the material but still attain sufficent adhesion to resin cements. Surface treatment with KHF_2 should be further adressed to evaluate the bond strength when different resin cements are used for cementing zirconia to tooth substance.

1.7 Bond strength testing

In vitro bond strength testing of ceramics cemented to tooth substance is a common method for evaluating the effect of interventions in the different substrates—for example, surface treatment of ceramics and dentin and choice of cement. Limitations of the in vitro test method are mostly related to low comparability to in vivo conditions like vitality of teeth, working conditions and directions of applied forces, and lack of standardized testing protocols (83). Despite the caution that must be exhibited when drawing a conclusion from in vitro testing, the method has many advantages—quick results, stable sample size, the possibility to measure and compare specific parameters and an easy technique being some of the main benefits (9, 83). In bond strength testing specimens are either applied tensile forces perpendicular to the bonded substrates or, more commonly, shear forces horizontal to the bonded substrates. Further, the test methods are divided into micro and macro, reflecting the diameter of the test specimens. Arguments for using micro (cross-sectional area 3 mm^2 or less) bond strength tests include lower occurrence of flaws that affect bond strength and more control of differences in tooth substance and economy. But specimens must be processed after cementations to obtain micro size, which is technique sensitive and might induce flaws or damages and even pre-test failures (83). Macro specimens (cross-sectional area >3 mm^2) are easier to handle and demand no further processing, which is why macro bond strength methods have been preferred by many researchers (84, 85). Optional methods for evaluating bond strength are push-/pull-out tests where the substrate—for example, a post—is enclosed by cement in a prepared cavity in dentin and further pushed or pulled out. Swelling of the resin cement during artificial ageing has been a drawback for this test method and is one of the reasons for the limited application

(83). Recently, a four-point bending flexural bond strength test was suggested as an alternative to tensile bond strength (86). The flexural bond strength method resulted in a higher frequency of adhesive failures, which is an advantage when studying interventions on substrate surfaces.

Lack of consensus regarding the most reliable test method and standardized protocols leads to a lack of comparability of results in different studies, as all testing variables—substrates, geometry, loading, cement layer thickness, elasticity modulus and storage—seem to affect the results (83, 87). Adding a variation of ageing methods—for example, thermocycling and water storage—further contributes to test results that are exclusive for each study (87).

2 Aim

The primary aim of this thesis was to investigate the different aspects of ceramics resin cemented to dentin in order to improve bond strength.

The secondary aims of the thesis were to investigate:

- Bond strength in relation to different surface treatments of ceramics.
- Cement layer thickness of dual-cure resin cements in relation to bond strength.
- Dentin surface roughness in relation to bond strength of zirconia cemented to dentin.

3 Material and method

This thesis comprises three papers with an in vitro study design. All test specimens were created in the same laboratory, under similar conditions, with an identical protocol by the same operator.

3.1 Test specimens

Bovine dentin, five dual-cure resin cements and three different ceramic materials were selected for production of test specimens. Of the ceramics, lithium disilicate glass ceramic (LDS) was considered control material, whereas zirconia was experimental.

3.1.1 Bovine dentin

Bovine mandibles (3-6 years of age) were obtained from Nortura (Rudshøgda, Norway). Four to eight incisors were extracted from each mandible and kept in distilled water until further processing.

Teeth were cut in the cervical area to remove the root. The tooth crowns were mounted in epoxy resin (EpoFix, Struers, Denmark) with the buccal surface exposed. Thereafter, teeth were ground using P500 SiC paper on a universal grinding machine (Planopol, Struers, Denmark) to expose a >5 x 5 mm dentine surface.

3.1.2 Ceramic rods and surface treatment

Zirconia (Starceram Z, H.C. Starck Ceramics GmbH, Germany, n= 340) and LDS (IPS e.max CAD, Ivoclar Vivadent, Liechtenstein, n=150) circular rods (Figure 1) were produced by CAD/CAM technique in cooperation with a dental technician.

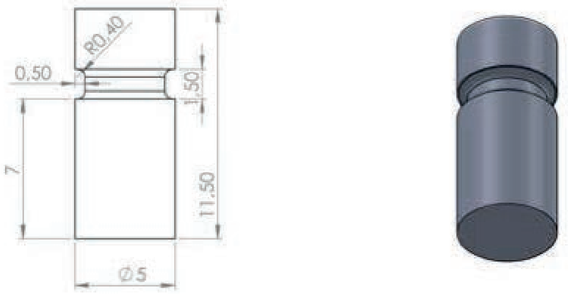


Figure 1. Configuration of ceramic rods.
The rods were 5 mm in diameter and 11.5 mm high. For identical test conditions in the three papers, all rods were produced with a notch in the circumference, which was used to facilitate grip during tensile testing.

Rods were ground at both ends using P500 SiC paper on a universal grinding machine (Planopol, Struers, Denmark) for equal surface roughness. Zirconia rods (n=300) were randomly assigned to two different surface treatment groups:

1) Zir A: Airborne particle abrasion with 50 µm aluminum oxide (Al_2O_3, Korox, Bego, Québec, Canada) at 2.5 bar perpendicular to the surface from a 10 mm distance for 10 s. The rod had a rotational movement.

2) Zir E: Hot etching with potassium hydrogen difluoride (KHF_2, Honeywell, North Carolina, USA) at 280 °C for 10 min.

After surface treatment, rods were steam cleaned and ultrasonically cleaned in distilled water for 10 min, and then thoroughly air-dried.

LDS rods were etched with 4.5% hydrofluoric acid (HF, IPS Ceramic Etching Gel, Ivoclar Vivadent) for 20 s, cleaned in running water >20 s and thoroughly air-dried.

3.1.3 Cement

The five dual-cure resin cements listed in Table 1 were selected for cementing ceramic rods to dentin.

Table 1. Dual-cure resin cements used for creating test specimens and pre-treatment of ceramics and tooth substance.

Cement	**Filler content**	**Chemical pre-treatment of ceramic**	**Pre-treatment of tooth substance**
Variolink Esthetic DC (Ivoclar Vivadent)	60-68%	Monobond Plus*	Two-step etch-and-rinse: Phosphoric acid etchant, Adhese Universal
Multilink Automix (Ivoclar Vivadent)	61%	Monobond Plus*	One-step self-etching: Multilink Primer A and B
Duo-Link (BISCO Dental)	62%	Z-Prime Plus**, Bis-Silane Parts A & B and D/E Resin***	Three-step etch-and-rinse: Phosphoric acid etchant, All-Bond 2 primer A and B, Pre-Bond Resin
Panavia F 2.0 (Kuraray Noritake Dental Inc.)	76%	Clearfil Ceramic Primer Plus*	One-step self-etching: ED Primer 2 Liq. A and B
RelyX Unicem (3M)	70%	No pre-treatment of zirconia, Bis-Silane Parts A & B***	Self-adhesive: No pre-treatment

*Universal primer for both zirconia and glass ceramics, ** selective zirconia primer, ***selective pre-treatment of glass ceramics.

3.2 Cementation

For each cement, 10 ceramic rods of each surface treatment group were created—giving a total of 150 test specimens in each paper.

Before cementing, dentin was cleaned with pumice powder dispensed in water, thoroughly rinsed in running water and dried.

All cementation was performed under similar conditions regarding room temperature and humidity, and according to the producers´ manuals. In addition, 8.7 N seating load was applied using a cementation jig. Excess cement was removed using a micro brush before light curing 20 s from 4 directions. Following cementation, all specimens were kept dry at room temperature for 15 min and thereafter immersed in 37 °C distilled water for 24 h.

3.3 Thermocycling

In Paper I and III, airborne particle abrasion with Al_2O_3 was used for removing cement remnants outside the rod. Complete removal was confirmed in light microscope (American Optical Stereo Star/Zoom, model 570, American Optical Corporation, Buffalo NY, USA. Magnification 10X–63X). Thereafter, test specimens were thermocycled 5000 cycles of 45 s in 5 °C and 55 °C water baths.

3.4 Paper I

3.4.1 Surface evaluation of ceramics

Randomly selected rods from all three ceramic groups were selected for surface evaluation in a scanning electron microscope (Hitachi Analytical Table Top Microscope /Benchtop SEM TM3030). Surface roughness was measured using a confocal microscope (Sensofar S Neox, Barcelona, Spain) and the mean surface roughness (Sa-value) in µm was calculated for each ceramic group.

3.4.2 Tensile bond strength

Test specimens were mounted in a universal mechanical test machine (Lloyd LRX, Lloyd Instruments Ltd, Leicester, UK). Tensile force was applied until breakage using a centred wire with a crosshead speed of 1 mm/min. Tension force (N) at breakage was recorded and tensile bond strength (MPa) calculated in Nexygen DF Force Measuring Software (Ametek Chatillon, California, USA).

3.4.3 Fracture morphology

After tensile bond strength testing, both dentin and ceramic rods were studied in light microscope (American Optical Stereo Star/Zoom, model 570, American Optical Corporation, Buffalo NY, USA. Magnification 10X–63X) to determine fracture morphology. Fractures were classified as: adhesive between dentin and cement; adhesive between ceramic and cement; cohesive in dentin; cohesive in cement; and combination of adhesive and cohesive fractures.

3.4.4 Statistical analysis

Microsoft Excel (version 14.2.3) was used for calculating mean tensile bond strength and standard deviation.

The Kolmogorov–Smirnov test was used for calculating normality. Differences were evaluated using ANOVA followed by Tukey’s HSD test, both between rod materials for each cement and between cements for each rod material. $p<0.05$ was regarded as a statistically significant difference.

3.5 Paper II

3.5.1 Cutting of specimens

Test specimens were further embedded in epoxy resin after 24 h of storage in 37 °C distilled water. The epoxy embedded specimens were mounted in a Micracut Precision (Kemet International, Kent, United Kingdom) cutting machine and two vertical slices of 2 mm were cut from each specimen with the ceramic rod centred. The slices were kept moist in closed containers.

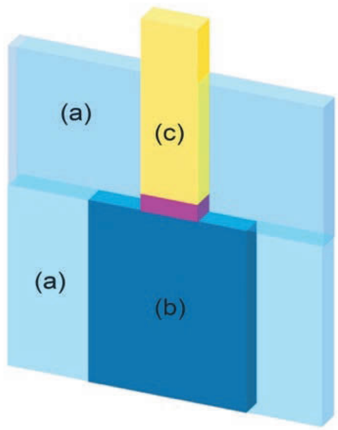


Figure 2. Illustration of the specimen after being cut into slices: epoxy resin (a), embedded dentin (b), ceramic rod (c) with resin cement (red).

3.5.2 Cement thickness measurement

One slice of each specimen was selected for SEM study and coated using a combination of platinum (80%) and palladium (20%).

SEM (HITACHI SU1510 Variable Pressure SEM, Hitachi High-Tech Corporation, Tokyo, Japan) was used for studying the cement layer between the ceramic rod and bovine dentin. Each cement layer was imaged in back-scattered or secondary electron mode and the thickness measured at five evenly distributed points at 300 times magnification.

For some of the test specimens, it appeared that the ceramic rod surface was oriented with a slight inclination angle to the dentin surface. The thickness at the central point of the cement layer (called cement thickness) and the inclination angle were both estimated for each specimen by linear regression (Thickness = constant + β*distance of measurement) based on the five measuring points. In addition, the thinnest part of the cement layer was derived from the regression parameters and located at the periphery of the rods due to the inclination angle. This was called peripheral cement thickness.

3.5.3 Finite element analysis

Finite element analysis (FEA) was performed to examine the uncertainty in the measured strength introduced by variation in the cement thickness and to see whether correlation between the average variation for a test specimen and the outcome of the experiments could be explained. A 1292-element and a 21168-element model of a rod-cement-dentine-epoxy mounted tensile-test specimens were created in Lisa (version 8.0.0, Lisa-Finite Element Technologies) with refinement of element size down to 1.5 µm for the outer edge of the cement layer in the later model. In a half-section model (Figure 3), the components were divided into sixteen segments of 11° and four segments of 1° about the cylinder axis. To validate the test conditions, the circumference of the epoxy moulding was constrained to zero displacement along the axial direction. The origin along this axis was set at the cement-rod interface.

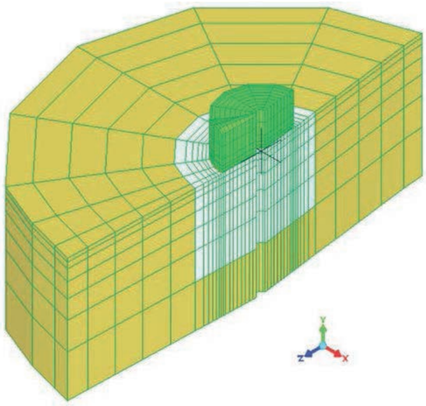


Figure 3. Finite element model of the tensile specimens. The components visible are: yellow, epoxy mould; white, dentin; green, ceramic rod. The cement is too thin to resolve. The black cross is at the origin of the axes with direction denoted in the inset. The apparent angle in YZ-plane is an artefact from checking the integrity of the model.

3.5.4 ISO cement film thickness

ISO cement film thickness was measured according to ISO 4049-2019 (88). Cement was placed between two glass-plates and loaded with 150 N for two min before light curing. ISO cement film thickness was measured using a micrometer (Mitutoyo, Kawasaki, Japan). The procedure was repeated five times for each cement.

3.5.5 Statistical analysis

Regressions analysis was performed using STATA version 16 (STATACorp, Texas, USA). The following models were chosen for the regression analysis: Model 1**:** Mean cement

thickness = $\alpha 0 + \alpha 1$ Material + $\alpha 2$ Cement + ε. Model 2**:** Minimal cement thickness = $\beta 0 + \beta 1$ Material + $\beta 2$ Cement + ε. ε = error term with random statistical noise.
Box plots for cement thickness and peripheral cement thickness were made using ggplot2 package in R statistical computing (CRAN.org, Vienna, Austria).
Microsoft Excel spreadsheet (Version 16.16.25, Microsoft office 2018) was used for calculating the correlation between tensile bond strength and cement thickness, and skewness for each cement/ceramic combination.

3.6 Paper III

3.6.1 Part one

3.6.1.1 Shear bond strength

Test specimens were mounted in a universal mechanical testing machine and shear force with a crosshead speed of 1.00 mm/min was applied to the dentin-cement-ceramic interfaces using a chisel. Force at break (N) and shear bond strength (MPa) were registered.

3.6.1.2 Fracture morphology

After shear bond strength testing, both dentin and ceramic rods were studied with a light microscope to determine fracture morphology. Fractures were classified in the same manner as for tensile bond strength testing.

3.6.2 Part two

3.6.2.1 Dentin surface

Dentin samples (n=40) were randomized to grinding with either P80 or P1200 SiC paper.

3.6.2.2 Surface topography evaluation

Selected dentin samples ground with P80 or P1200 SiC papers were studied in SEM (Tabletop Microscope, HITACHI, TM4000Plus, Hitachi High- Technologies Corporation, Tokyo, Japan) and 3D surface topography was assessed using Hitachi map 3D Standard 7.4 (Hitachi High-Technologies Corporation, Tokyo, Japan). Mean Sa-value was calculated for each of the two roughness groups.

3.6.2.3 Test specimens

Variolink Esthetic DC and RelyX Unicem were selected for cementing Zir E (Dental Direkt Bio ZW iso, Germany, n=40) to the additional 40 dentin samples. Ten test specimens were created with each cement for dentin ground with P80 and P1200.

3.6.2.4 Shear bond strength

Test specimens were subjected to shear bond strength testing identical to point 3.6.1.1

3.6.2.5 Cement-dentin relation

Liquid nitrogen was used for fracturing selected samples of bovine dentin vertically to the cemented area after bond strength testing. The samples were studied and photographed in SEM to see the relation between cement and dentin.

3.6.3 Statistical analysis

Statistical analysis was performed using the STATA SE (version 16.1, StataCorp, Texas, USA) and R (version 4.0.3, R Project, Vienna, Austria). Comparisons of mean bond strength for cements and ceramics were performed using Student´s t-test with significance level <0.05. Pictures were made using *ggplot2*-package in R.

4 Results

4.1 Paper I

The surfaces of the three ceramic groups are illustrated in Figure 4, showing great differences in morphology after surface treatment.

The highest and lowest mean Sa-values were calculated for Zir A and Zir E respectively.

Figure 4. Representative SEM images of Zir A (a), Zir E (b), and LDS (c).

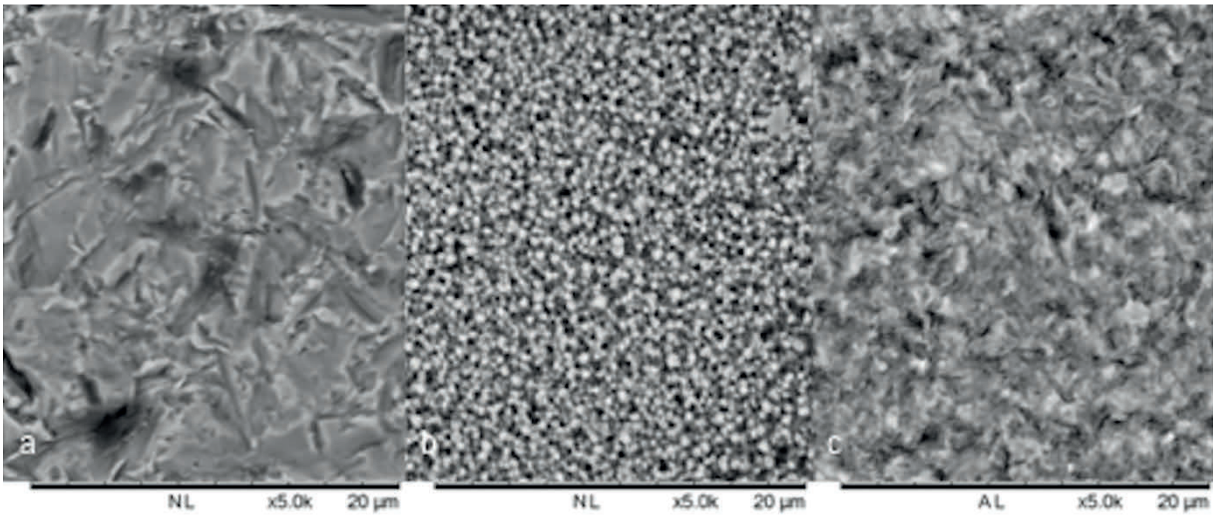


Table 2. Sa (mean surface roughness) in µm for each ceramic material.

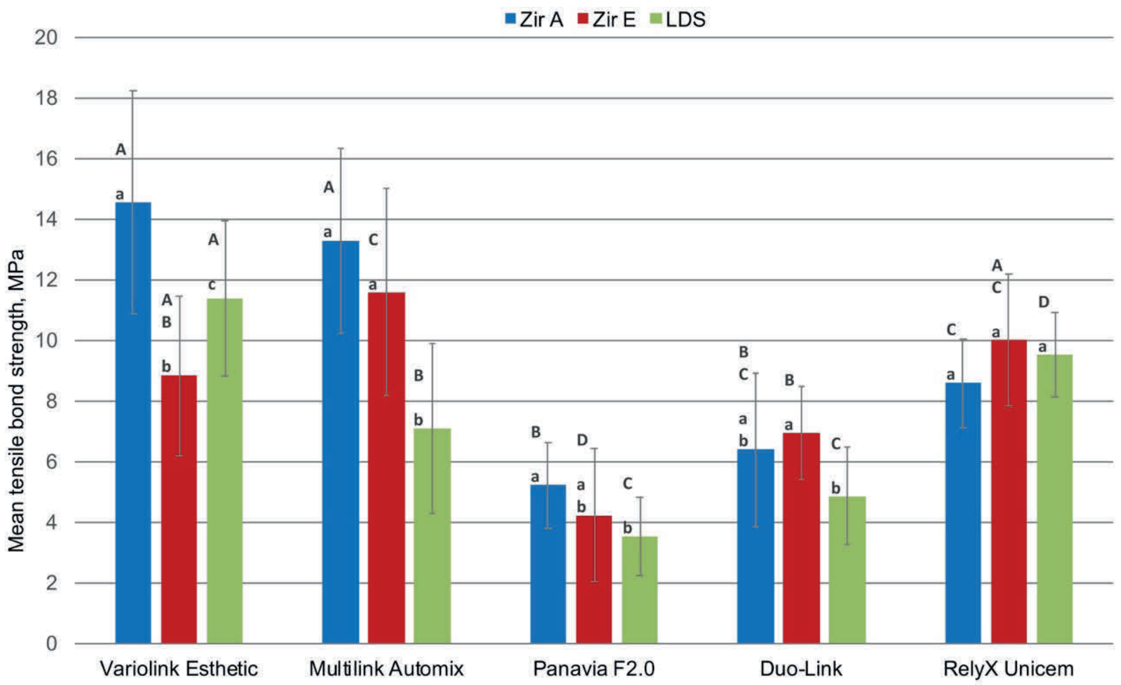
Sa-values in µm		
Zir A	**Zir E**	**LDS**
0.53-0.59	0.12-0.13	0.18-0.25

Zir A: airborne particle abraded zirconia, Zir E: KHF_2 etched zirconia, LDS: hydrofluoric acid etched lithium disilicate.

Mean tensile force at break and standard deviation were calculated for each ceramic-cement combination and are presented in Figure 5. The highest mean tensile bond strength was observed when cementing Zir A with Variolink, whereas the lowest bond strength was obtained for LDS cemented with Panavia.

Results of ANOVA and Tukey´s HSD, calculated for difference between cements for each ceramic rod, and difference between ceramic rods for each cement, are also given in Figure 5. There were no differences in bond strength with different surface treatments of zirconia rods, except for Variolink, where Zir A showed significantly higher bond strength compared to Zir E. Compared to both zirconia groups, LDS had lower or similar mean tensile bond strength with all cements except Variolink.

Figure 5. Mean tensile bond strength and standard deviation for ceramic rods cemented to dentin. Different uppercase letters illustrate significant differences ($p<0.05$) between cements for each rod material. Different lowercase letters illustrate significant difference ($p<0.05$) between Zir A, Zir E, and LDS for each cement.



Zir A: airborne particle abraded zirconia, Zir E: KHF_2 etched zirconia, LDS: hydrofluoric acid etched lithium disilicate.

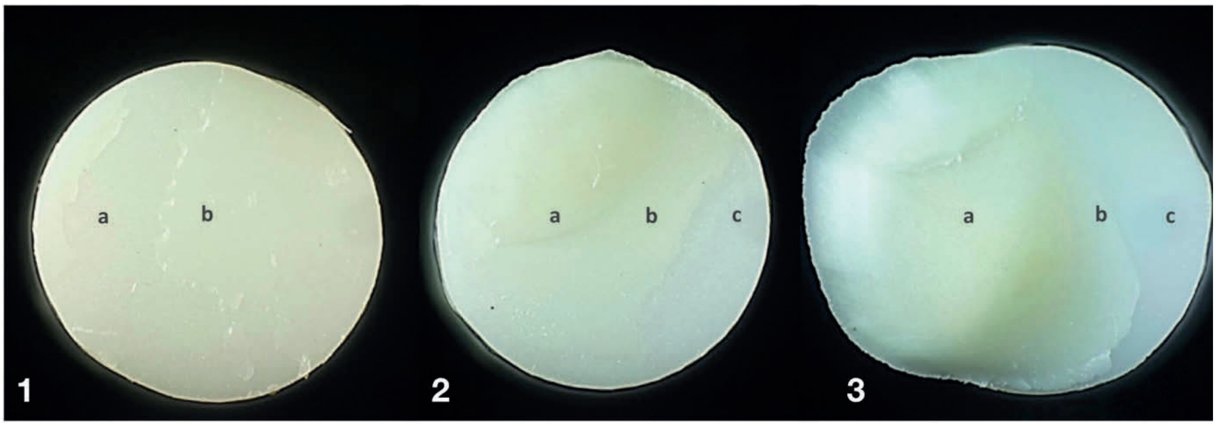
Table 3 gives an overview of the distribution of fracture morphology for each ceramic-cement combination. Cohesive fractures in cement and combined adhesive and cohesive fractures dominated. Figure 6 illustrates examples of fracture morphology observed.

Table 3. Fracture morphology after tensile bond strength testing.

	Adhesive						Cohesive								
Fracture type	**Dentin-cement**			**Ceramic-cement**			**Dentin**			**Cement**			**Combination**		
Cement/ ceramic	Zir A	Zir E	LDS	Zir A	Zir E	LDS	Zir A	Zir E	LDS	Zir A	Zir E	LDS	Zir A	Zir E	LDS
Variolink	1		4				3			1	10	2	5		4
Multilink			1			2	2	2		1		2	7	8	5
Panavia				2						6	9	10	2	1	
Duo-Link										10	10	10			
RelyX	5	1						1			2	8	5	6	2

Zir A: airborne particle abraded zirconia, Zir E: KHF_2 etched zirconia, LDS: hydrofluoric acid etched lithium disilicate.

Figure 6. Examples of fracture morphology observed in light microscope (diameter 5 mm). 1: combination of adhesive fracture between cement-zirconia (a) and cohesive fracture in cement (b); and 2: combination of cohesive fracture in dentin (a) and adhesive fracture between cement-dentin (b) and cement-zirconia (c); 3: combination of cohesive fracture in dentin (a) and cement (b), and adhesive fracture cement-zirconia (c).



4.3 Paper II

The cement thicknesses are given in Figures 7 and 8. The mean cement thickness ranged from 20 to 40 µm. There was a tendency for thinner cement layers with Zir E and when using Multilink cement. A similar tendency was observed for peripheral cement thickness measurements.

Figure 7. Cement thickness defined as the thickness of the central point of the cement layer of each ceramic-cement combination. The box-plots show mean value (horizontal line), 25% and 75% percentile. Vertical lines represent 90% confidence interval.

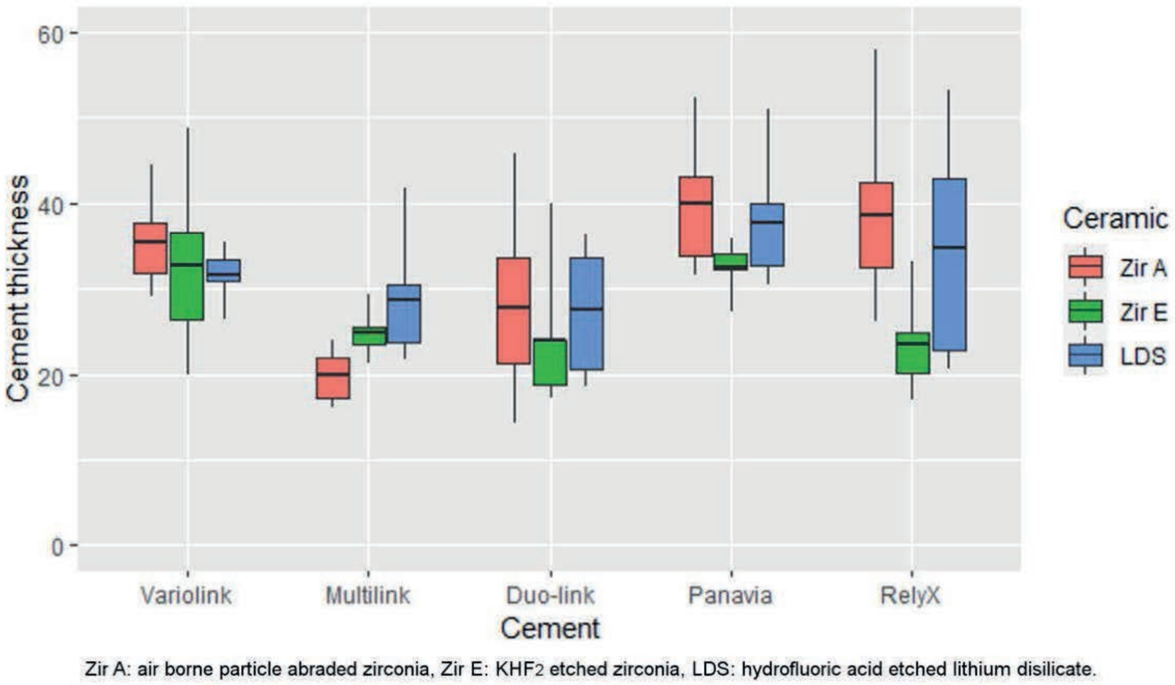
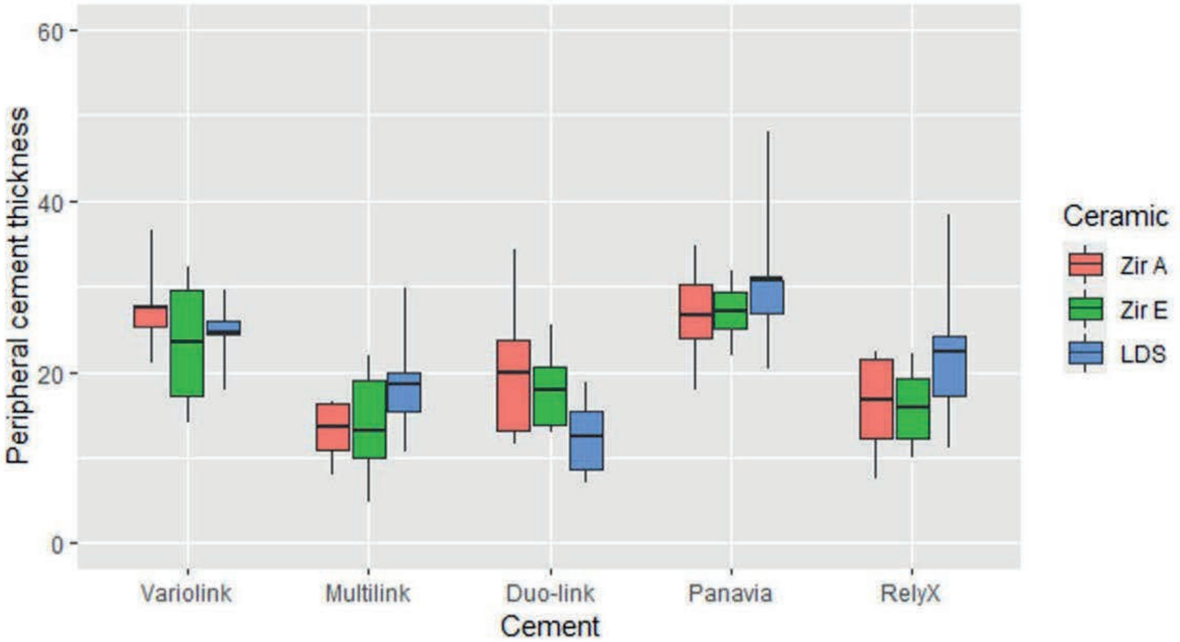


Figure 8. The peripheral thickness of the cement layer (called peripheral cement thickness) was derived from the regression parameters and located at the circumference of the rods. This is regarded as the thinnest cement layer. The box-plots show mean value (horizontal line), 25% and 75% percentile. Vertical lines represent 90% confidence interval.



Zir A: air borne particle abraded zirconia, Zir E: KHF_2 etched zirconia, LDS: hydrofluoric acid etched lithium disilicate.

Results of multiple linear regression analysis for cement thickness and peripheral cement thickness are given in Table 4. Combining all three ceramic types, a significantly thicker cement layer was found for Panavia than for the other types, except Variolink. The same was also observed for both cement thickness and peripheral cement thickness. The thinnest cement layers were observed for Multilink and Duo-Link, and for the Zir E specimens for most cements.

Table 4. Results of regression analysis of the effect of type of ceramic and type of cement on cement thickness and peripheral cement thickness, respectively. Zir A and Panavia are used as reference materials.

	Cement thickness **Value (Confidence interval)**	**Peripheral cement thickness** **Value (Confidence interval)**
Ceramic		
Zir A	0	0
Zir E	-4.83 (-8.15, -1.50)*	-1.36 (-3.76, 1.04)
LDS	-0.30 (-3.81, 3.21)	0.95 (-1.93, 3.83)
Cement		
Panavia	0	0
Variolink	-3.46 (-7.01, 0.09)	-2.91 (-6.24, 0.41)
Multilink	-12.32 (-15.70, -8.93)*	-13.05 (-16.40, -9.70)*
Duo-Link	-10.34 (-14.46, -6.21)*	-11.47 (-15.18, -7.76)*
RelyX	-4.50 (-9.25, -0.26)*	-9.78 (-13.43, -6.14)*
Constant		
	38.50 (35.21, 41.80)*	28.33 (25.42, 31.24)*
R-squared	0.27	0.38
Number of observations	150	

* Significantly different

Zir A: airborne particle abraded zirconia, Zir E: KHF_2 etched zirconia, LDS: hydrofluoric acid etched lithium disilicate.

While most specimens showed some variation in cement thickness across the section, a difference of up to 90 µm from one side to the other was found in several specimens. This corresponds to an inclination angle of 1.1 degree between the axis of the ceramic rod and the right angle to the dentin surface. However, most observations of the inclination angle ranged between −0.3 and +0.3 degree (Figure 9).

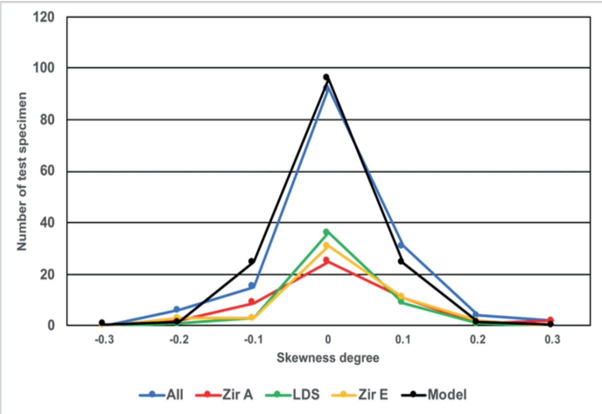


Figure 9. Distribution of the observed variation in cement thickness. The y-axis shows number of test specimens and the x-axis shows the degree of inclination for all ceramics combined (All) compared to a best-fitting normal distribution (Model) with standard deviation 0.28°. Zir A: airborne particle abraded zirconia, Zir E: KHF_2 etched zirconia, LDS: hydrofluoric acid etched lithium disilicate.

Measured cement thickness was plotted against tensile force at break, registered in Paper I. Test specimens with cement thickness of 20-35 µm—especially Multilink and Variolink—appeared to have the highest tensile bond strength, although this observation was not statistically significant (Figure 10). For test specimens with cohesive fractures in cement, or combined fractures after tensile testing, a negative correlation (−0.5) with cement thickness was observed (data not shown).

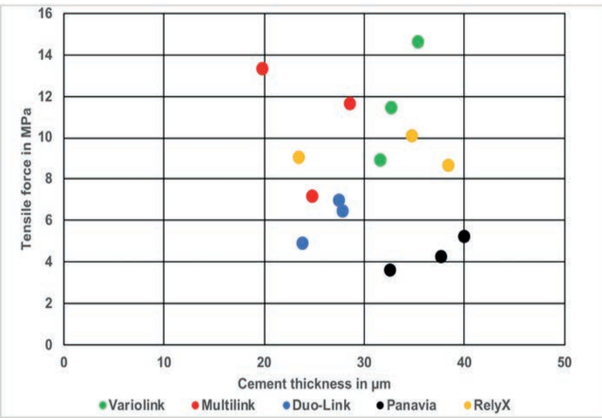
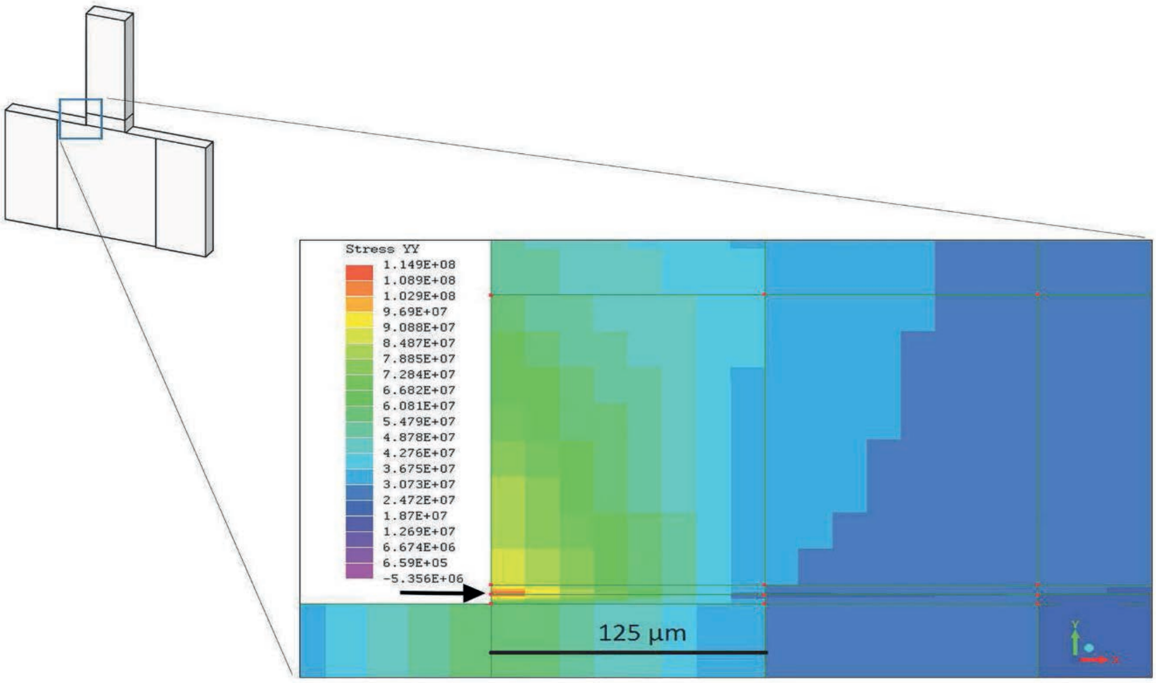


Figure 10. Cement thickness plotted against tensile force at break. Points with same colour represent results from ceramic rods with different surface treatment.

The finite element analyses indicated that the tensile stress in the cement was concentrated at the periphery of the cement layer (Figure 11).

Figure 11. Presentation of finite element analysis of stress concentration. Detail of cement (two thin layers of elements extending across the image, black arrow), ceramic rod (above the cement layer) and dentin (below the cement) showing the location of the maximum stress. Colour scale for stress (Pa) in the vertical Y- direction.



In specimens with varying cement thickness, the maximum computed tensile stress was at the thinnest edge of the cement layer. The analysis showed that the radial and lateral (hoop) stress components were large and tensile, regardless of the elastic modulus and Poisson ratio chosen for the cement. It was noted that the values obtained for the coarse 1292-element and the refined 21,168-element models agreed to within 10%.

All cements fulfilled the ISO requirement for cement film thickness—however, the largest cement thickness was observed for Variolink (Table 5).

Table 5. ISO cement film thickness in µm measured according to ISO 4049:2019 (88).

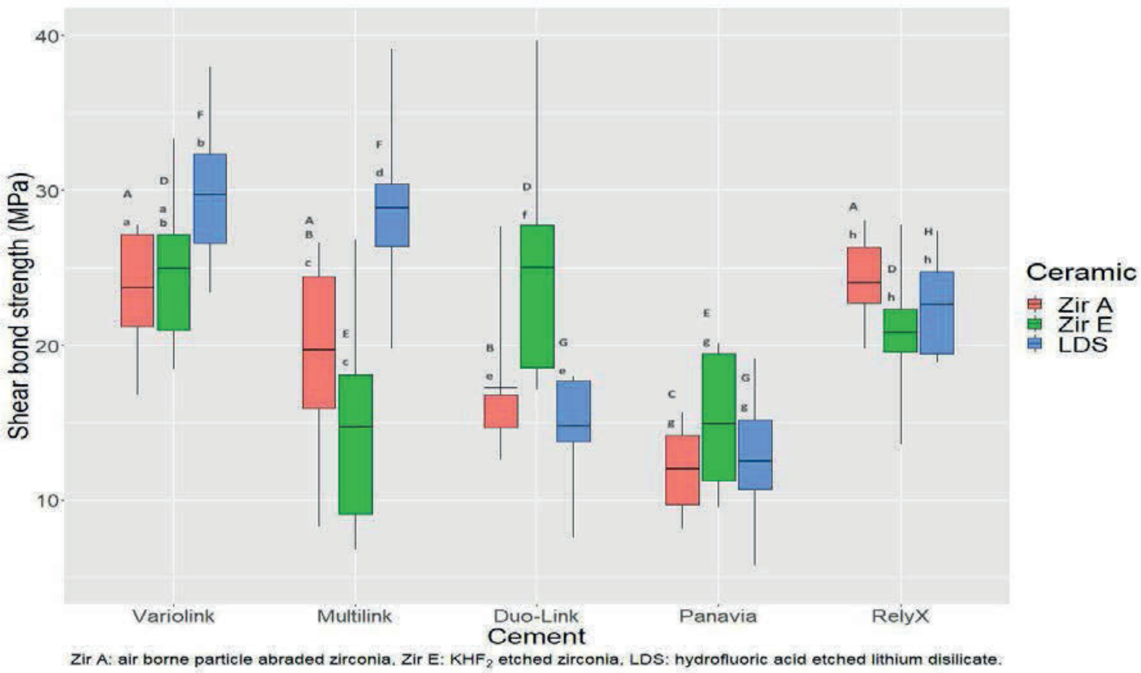
Measurement	Variolink	Multilink	Duo-Link	Panavia	RelyX
1	12	8	12	6	7
2	11	3	4	11	5
3	21	6	1	5	19
4	21	7	4	4	5
5	22	7	5	10	6
Median	21	7	4	6	6

4.2 Paper III

4.2.1 Part one

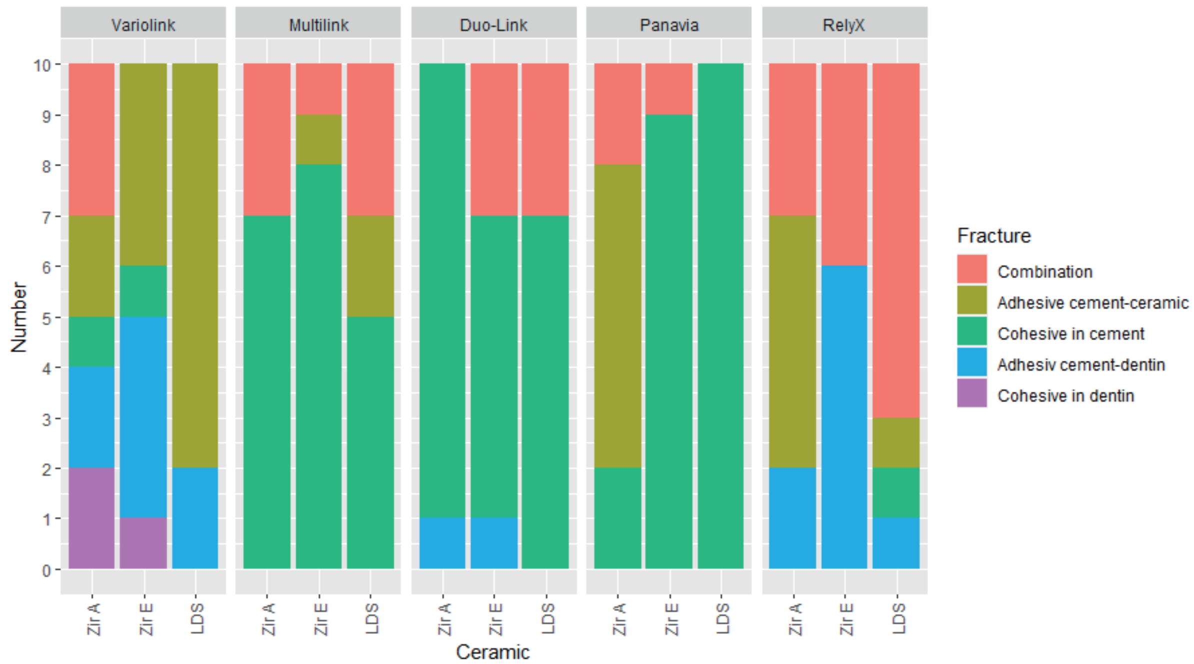
The highest mean shear bond strength was observed for LDS cemented to dentin using Variolink and the lowest for Zir A cemented to dentin using Panavia (Figure 12). There was no difference in bond strength between Zir A and Zir E for all cements used, except for Duo-Link. The bond strength for the reference group (LDS) was higher, similar, or lower than the zirconia groups (Figure 12).

Figure 12. Shear bond strength for ceramic rods cemented to dentin (P500). The horizontal line represents the mean value, the lower part of the box represents the 25% quartile, and the upper part of the box represents the 75% quartile. The vertical lines represent a 90% confidence interval. Different uppercase letters illustrate significant differences ($p<0.05$) between cements for each ceramic rod. Different lowercase letters illustrate significant difference ($p<0.05$) between Zir A, Zir E and LDS for each cement.



Cohesive fracture in cement and combined adhesive and cohesive fractures were dominating when Multilink, Duo-Link and Panavia were used for cementing ceramic rods to dentin ground with P500 SiC paper. The highest frequency of adhesive fracture between cement and dentin was observed when Variolink and RelyX were used for cementing Zir E to dentin (Figure 13).

Figure 13. Fracture morphology after shear bond strength testing for ceramic rods cemented to dentin ground with 500 grit SiC paper.

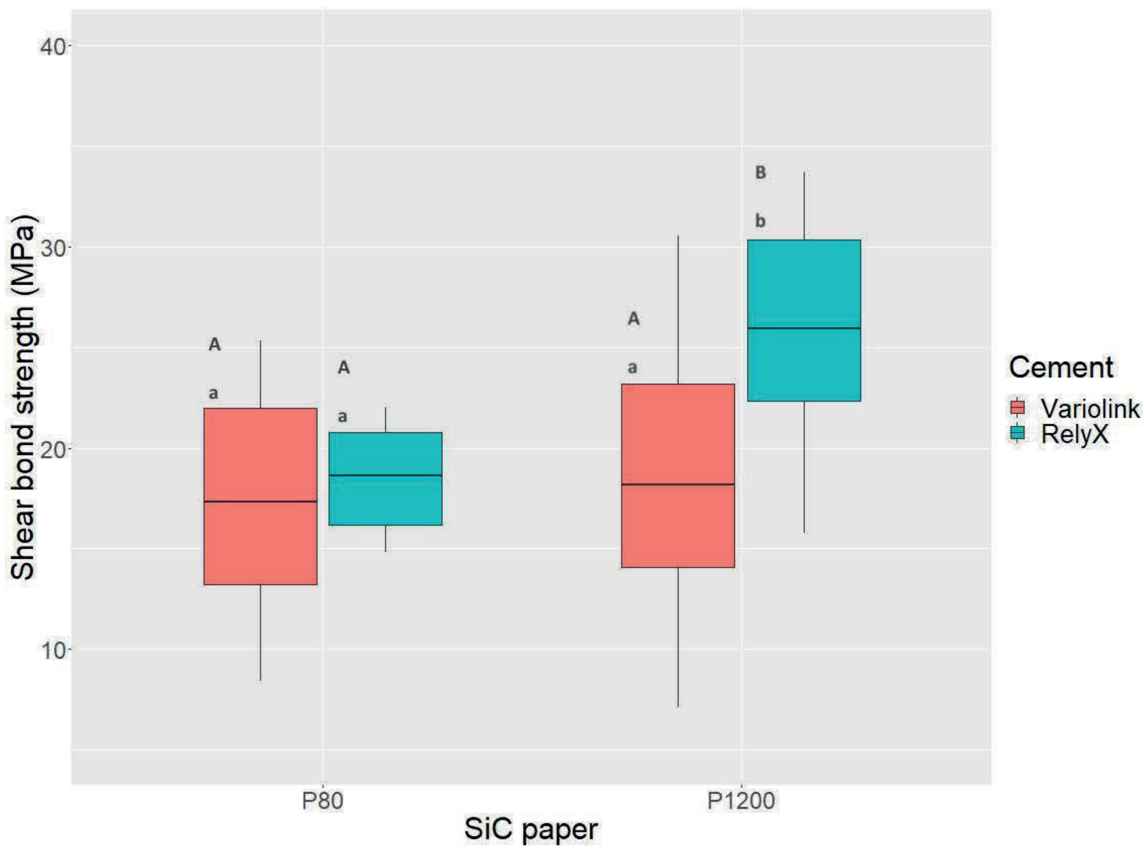


Zir A: air borne particle abraded zirconia; Zir E: KHF_2 etched zirconia; LDS: hydrofluorid acid etched lithium disilicate.

4.2.2 Part two

For dentin ground with P80 (Sa 5.40 µm) and P1200 (Sa 0.50 µm) SiC papers, the highest mean shear bond strength was observed when RelyX was used for cementing to P1200 dentin (Figure 14). P1200 resulted in significantly higher bond strength compared to P80 for RelyX but, for Variolink, no difference was observed between the surfaces. Between the cements, RelyX resulted in significantly higher bond strength compared to Variolink, when dentin was ground with P1200—but for P80 no difference was observed (Figure 14).

Figure 14. Shear bond strength for Zir E cemented to dentin ground with P80 and P1200 grit SiC paper. The horizontal line represents the mean value, the lower part of the box represents the 25% quartile, and the upper part of the box represents the 75% quartile. The vertical lines represent a 90% confidence interval. Different uppercase letters illustrate significant differences ($p<0.05$) between P80 and P1200 for each cement. Different lowercase letters illustrate significant differences ($p<0.05$) between cements for each surface roughness.



Regardless of SiC paper grit used to grind dentin before cementing with RelyX, fracture morphology after shear bond strength testing was mainly adhesive between cement and dentin and combined fractures. When dentin was ground with P1200, some adhesive fractures between cement and ceramic were observed. The most frequent fracture morphology for Variolink was also adhesive between cement and dentin, but a greater variation in fracture morphology was observed compared to RelyX (Figure 15). SEM revealed different relation between dentin and the two cements (Figure 16).

Figure 15. Fracture morphology after shear bond strength testing of Zir E cemented to dentin ground with different SiC paper grit.

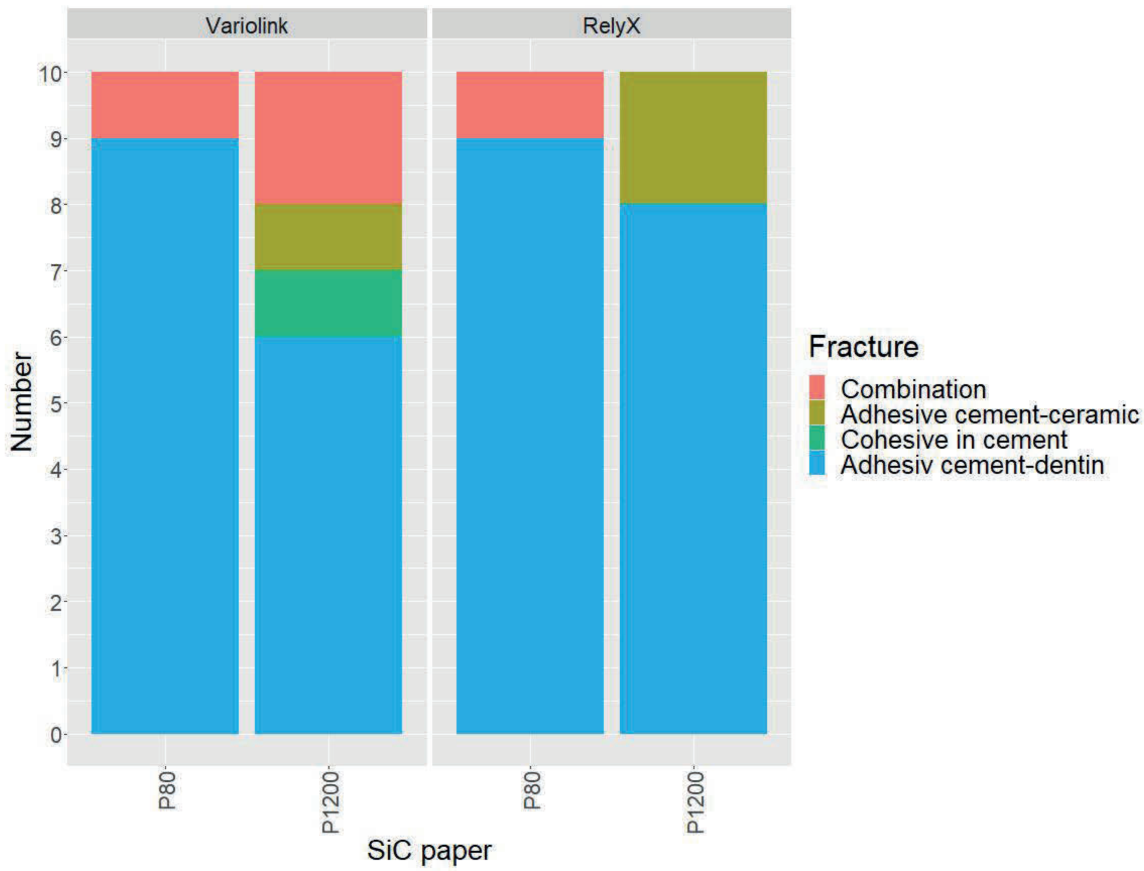
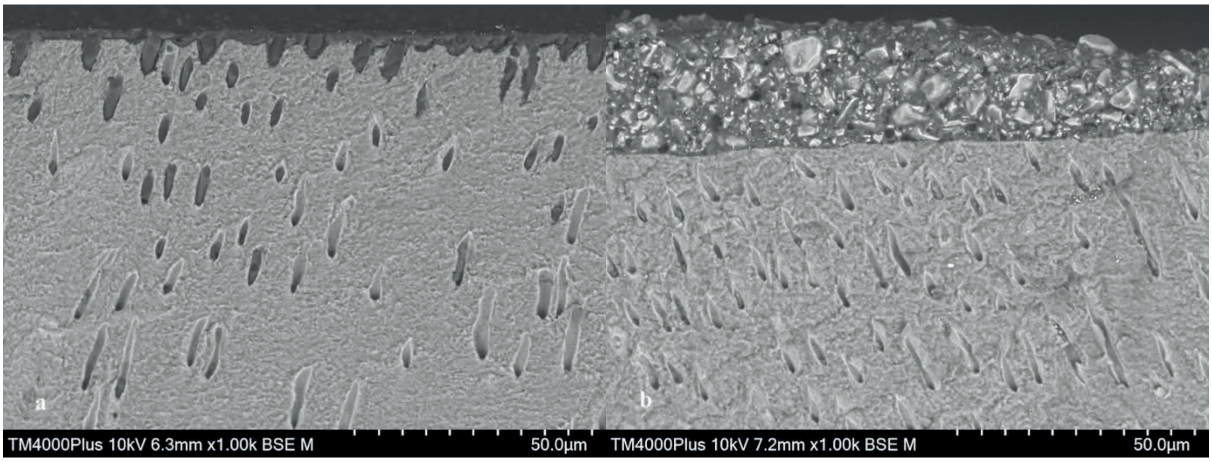


Figure 16. Scanning electron microscope photo of dentin and remnants of cement after shear bond strength testing of Zir E cemented with a) Variolink, b) RelyX to dentin ground with P1200 SiC paper.



5 Discussion

5.1 Methodological considerations

5.1.1 Test groups and sample size

Three different ceramic surfaces were selected for studying adhesion between ceramics and resin cement. Hot etching of the zirconia surface using KHF_2 was regarded as a promising method for establishing chemical and micro mechanical adhesion to resin cement (76). Surface treatment of zirconia with Al_2O_3 airborne particle abrasion was selected for comparison, since this is a widely used method with most evidence in the literature (73). In addition, LDS was selected as a control material to zirconia since both chemical and micro mechanical adhesion are established with resin cement after HF-etching and silane application to this ceramic (18).

The five dual-cure resin cements selected for cementing ceramic rods to dentin are extensively used in in vitro testing and in clinics (56). They have different adhesive methods to tooth substance and represent a variation in available products for the bonding of ceramic restorations.

Ten test specimens were created of each ceramic-cement combination. The selected sample size was based on a number of previously published comparable studies (56, 69, 89). To determine the correct sample size, a power calculation should be conducted prior to execution of a study.

In the present project, statistical power was calculated post hoc. The study power is the ability to detect differences that exist between groups and should be high, preferably >80%. If the power is low, the risk of committing a type 2 error becomes high, meaning that the null hypothesis might be accepted even though it is wrong (90).

When comparing groups with large differences in mean bond strength and small standard deviation, the power reached >90%. For groups with small differences in mean bond strength and large standard deviation—e.g., Multilink and RelyX in with a difference of 1.57 MPa and standard deviation of 3.42 MPa and 2.18 MPa respectively in tensile testing—the power was calculated to be around 21%. To obtain a power of 80% for the latter groups, sample size had to be increased to 55.

5.1.2 Bovine dentin

In all three papers, bovine dentin was used as the substrate to which ceramic rods were cemented. An advantage in using bovine compared to human dentin is the availability of sound tooth substance in extensive amounts, which is necessary when performing in vitro testing with a certain sample size or repetitions. According to ISO standard 11405:2015 (91), the buccal surface of mandibular incisors of bovine cadavers can be used for testing of adhesion to tooth substance. Still, there are differences between bovine and human dentin that might have affected the results of bond strength testing. Even though the teeth are histochemical and anatomical similar, only superficial layers of bovine dentin can be used as comparison to human dentin because of the density and diameter of tubules (92). Teeth embedded in epoxy resin were ground with SiC papers and due to the different size of the teeth, the dept of the exposed dentin varied to obtain a minimum 5 x 5 mm bonding area. Therefore, ceramic rods might have been cemented to different layers of dentin. Also, the smear layer produced during grinding is different regarding thickness and quality for different value SiC papers (93), which can affect adhesive strength between resin cement and dentin (85). Bovine teeth were reported by Nortura to be from cadavers between 3 and 6 years of age. Age-related differences between teeth may have affected bond strength, since it is known that deposition of intratubular dentin is a continuous process leading to reduction, or even complete closure, of tubular diameter and a limit on the formation of retentive resin tags (15). Equally, the increase in mineral content might result in increased bond strength due to chemical interaction with adhesives (58). After extraction, teeth were stored for a maximum of 6 months in distilled water before usage in in vitro testing. Some differences in bond strength have been observed between freshly extracted and older teeth (92), but it is not likely that different storage times had any large impact on bond strength since the time variation was small.

5.1.3 Artificial ageing

After cementation, test specimens were stored in 37 °C distilled water for 24 h. Thereafter, they were subjected to artificial ageing by 5000 cycles in 5 °C and 55 °C water baths, as recommended in ISO standard 10477:2020 (94). A dwell time of 20 s in each water bath was used.

Thermocycling simulates temperature changes the restoration, cement and tooth substance are exposed to in the oral cavity and is used to evaluate mechanical and structural characteristics after ageing (95). Different thermal expansion coefficients of the three materials results in hoop stress leading to deterioration of the bond strength—and even degradation of the resin cement, due to different thermal expansion coefficients of filler particles and matrix (96). In the study by Ruyter et al. (76), thermocycling was not performed, which might explain higher bond strength compared to Paper III in this thesis.

Eliasson and Dahl (97) investigated temperature changes in specimens of various size and dwell time during thermocycling. Specimens with large dimensions (10x10 mm) never reached the temperature of the water baths using a dwell time shorter than 60 s. Even after 60 s the temperatures in large, epoxy embedded specimens remained 8 °C different compared with the water baths. The use of 20 s dwell time in this project might have been too short for the specimen to reach the temperature of the water baths. This would lead to a different degree of ageing of the specimen and potentially affect bond strength (97).

5.1.4 Bond strength test methods

The tensile and shear bond strength of ceramics cemented to dentin were tested in Paper I and III, respectively, and showed a similar relation between the cements, even though the MPa values were different. Tensile bond strength was tested by applying axial force to the ceramic rod until breakpoint using a centred wire. For a true estimate of tensile force needed to break the bond, the rod must be bonded perpendicular to the dentin surface and lined up in the test machine (98). In Paper II it was detected that some of the ceramic rods were bonded with a slight inclination to the dentin surface. Since the same protocol for producing test specimens was used in all three papers, it is expected that some inclination also might have been present in Paper I, which again might have affected tensile bond strength. In shear bond strength testing the force was applied horizontally to the dentin-cement-ceramic interfaces until break, using a chisel. Measurement of shear bond strength will introduce nonhomogeneous stress in the interfaces, starting at brittle points leading to debonding (98, 99), and the term shear bond strength testing applies more to the test method used than the applied forces (100). Considering possible inclination of the ceramic rod to the dentin surface in Paper II, inclination might also have affected the shear bond strengths registered.

Macro test methods, meaning bonding area >3 mm^2, were selected due to easier fabrication and handling of specimens than for micro test methods (84, 100). Opponents to macro test methods argue that there is lower probability of critical flaws in the cement when using smaller test specimens and that the occurrence of cohesive fractures in tooth substance or cement is higher with larger bonding area and therefore precludes evaluation of interfacial bond strength (85, 100, 101). Still, there is no standardized protocol for adhesive bond strength testing, which makes comparison of bond strength results in different in vitro studies difficult.

5.1.5 Light microscope for fracture morphology characterization

To characterize fracture morphology after bond strength testing, a light microscope was used to study both dentin and ceramic rods. The colour similarity of the materials, especially cements and ceramics, made the characterizing challenging. Some of the fracture types could more easily be characterized than others. For adhesive fractures, the cement was retained either on dentin or the ceramic rod. The light-yellow colour in dentin was helpful in determining on which surface the cement was retained. Cohesive fractures in cement were frequently observed. For this fracture type, the cement was retained on both dentin and the ceramic rod. Because of the colour similarity of these materials, differentiating between cohesive fractures and combined fractures was challenging. SEM is used in many publications regarding fracture characterization after bond strength testing and is the only tool that can properly distinguish the different fracture types (87). This method provides a detailed and specific characterization, but is extremely time and resource consuming compared to using a light microscope (102). As a solution, characterization performed in a light microscope could have been validated in SEM for some of the samples to report the distribution of fractures with more confidence.

5.1.6 Finite element analysis

The finite element method (FEM) is a way of analysing mechanical tension in a body exposed to forces in one or several directions by using a computerized model. The body consists of simply shaped elements representing materials with different properties—for example, elastic modulus. Forces, such as tensile or shear, are applied to the exterior of the body to imitate mechanical testing (103).

To obtain correct mechanical tension in thin components of the model, like the cement in the test specimens used in this project, division into many small elements is preferred (103). In the present project, the cement was divided into small elements to observe how the tension changes with small differences in composition of the model.

Finite element analysis (FEA) was performed for all cement thickness measurements included in Paper II to evaluate the effect of difference in cement thickness across the specimens due to angulation of the ceramic rod. FEA showed that the stress was highest at the periphery. For test specimens where the ceramic rod appeared to have a slight angulation, the stress was highest where the cement was at its thinnest.

5.1.7 Test specimens for correlation between tensile bond strength and cement thickness

After tensile bond strength testing, fracture morphology revealed that the cement was the weakest link in test specimens because of a high number of cohesive fractures. Therefore, similar test specimens were produced in Paper II to measure the cement thickness in search of an explanation for the observations in Paper I. Tensile bond strength from Paper I and cement thickness measurements from Paper II were combined in a dot-plot (Figure 10) to explore the interrelationship between bond strength and cement thickness. Even though test specimens were produced with the same protocol and under similar condition in the two papers, a direct comparison of the results must be viewed with caution. A more reliable comparison would be between cement thickness and tensile bond strength measured in the same test specimens. However, this was impossible to perform in the present project since cutting and scanning in an electron microscope would damage the specimens. Another solution could be cutting macro specimens into smaller micro specimens and further randomizing them to two groups: 1) bond strength testing or 2) cement thickness measurement. However, the inclination of the ceramic rod to the dentin surface observed in some specimens could still lead to incomparable groups with this method.

5.2 Discussion of results

The primary aim of this thesis was to investigate the different aspects of ceramics resin cemented to dentin in order to improve bond strength.

The secondary aims of the thesis were to investigate:

- Bond strength in relation to different surface treatment of ceramics.
- Cement layer thickness of dual-cure resin cements in relation to bond strength.
- Dentin surface roughness in relation to bond strength of zirconia cemented to dentin.

5.2.1 Bond strength in relation to different surface treatment of ceramics

To evaluate roughness after surface treatment, the mean Sa value was calculated for each ceramic type. The parameter expresses difference in height compared to an arithmetical mean of the surface, but due to its shortcoming in differentiating symmetry and spacing of grooves and peaks, the parameter should only be used as an indicator of differences in roughness (104). Mean Sa-values for Zir A, Zir E and LDS were significantly different from each other. The highest values were found for Zir A, indicating that airborne particle abrasion using 50 µm Al_20_3 with a pressure of 2.5 bar at a 10 mm distance perpendicular to the rod results in a rougher surface than hot etching of zirconia with KHF_2 and HF etching of LDS. SEM images (Figure 4) illustrate differences in morphology after surface treatment. The surface of Zir A appears more irregular than the other two surfaces, with deeper and wider grooves, which explains the high Sa-value for Zir A. The surface of Zir E appears nodular and regular, which was reflected by the low Sa-value. LDS has a morphology and Sa-value in between the two zirconia surfaces.

Surface roughness of ceramics is important for adhesion between cement and ceramic but, despite the many studies performed on the subject, the ideal roughness remains unclear (105). In general, increased roughness increases the bonding surface area, which further enhances wettability and increases bond strength. However, the roughest surfaces have also demonstrated lower bond strengths than smoother surfaces after artificial ageing, probably due to stress and defects in the material from the roughening process which leads to failure of the adhesion, as well as unfavourable morphology of the treated surface (47, 106).

The different surface treatments used were expected to affect adhesion between cement and ceramic and be reflected in fracture morphology. In tensile bond strength testing, only a few adhesive fractures between cement and ceramic were observed, indicating that the observed differences in surface roughness and morphology were of negligible importance. After shear bond strength testing, a higher number of adhesive fractures between cement and ceramic were observed for some combinations indicating that, when shear forces are applied, surface roughness of the ceramic might be of greater importance.

In tensile testing, a significantly higher bond strength for Zir A compared to Zir E was observed for one of the cements—Variolink. For the other cements, tensile bond strength was similar for the two zirconia groups (Figure 5). The distribution of bond strength in shear testing was similar to the results from tensile testing. Limited differences were found for the surface treatment groups of zirconia, with Duo-Link being the only cement with significantly higher bond strength for Zir E compared to Zir A (Figure 12). The results are in concurrence with the published study by Ruyter et al. (76), who detected no significant difference in bond strength between particle abrasion and hot etching of zirconia. Another recently published study on KHF_2 etched zirconia (81) also concluded that the bond strength provided was similar to more established surface treatment methods.

The difference in roughness for the two zirconia surfaces (Table 2) indicates that the adhesion to Zir A should be stronger than to Zir E due to the increased bonding surface area. However, as proposed by Ruyter et al. (76), after KHF_2 etching the zirconia surface has active hydroxyl sites which promote chemical adhesion to primers. With this in mind, Zir E was expected to result in higher bond strengths than Zir A. In addition, adhesion between ceramic and primer/cement is dependent on good wetting of the surface. Akazawa et al. (82) reported significantly higher surface free energy and lower contact angle for three liquids tested when comparing KHF_2 etching to particle abrasion of zirconia, showing that the wettability is different and favouring adhesion to the etched surface. The effect of surface modification of zirconia must be put in relation to the fracture morphology observed. A high frequency of cohesive fractures in cement and combined adhesive-cohesive fractures shows that the cement layer was weaker than adhesive strength to ceramic, and an overall conclusion about the difference in effect of the surface treatment of zirconia cannot be drawn.

Surprisingly, bond strength of LDS only exceeded that of zirconia for some cements in shear testing. Even though this was in accordance with results published by Kwon et al. (107), the bond strength for LDS was expected to be higher because of the well-known establishment of both chemical and mechanical adhesion to resin cement (18). However, the observed high frequency of cohesive fractures in cement and combined adhesive-cohesive fractures, indicated that the cement was the weakest link in the test specimens, and no conclusion about difference in adhesive strength of primer/cement to LDS and zirconia could be drawn.

Combining roughening and primer application to the ceramic prior to cementation has shown increased bond strength compared to using these surface treatments separately (47), and today most resin cement systems include application of primer prior to cementation. So-called "universal" primers, containing both silane and functional monomers with adhesive properties to zirconia, are available for application on all types of ceramic surfaces and reduce the need for separate systems (108).

Prior to cementation with Multilink and Variolink, Monobond Plus was applied to both LDS and zirconia surfaces. The content of different functional monomers (silane methacrylate, phosphoric methacrylate and sulphide methacrylate) makes this primer universal and suitable for use on both glass ceramics and zirconia (109). When cementing with Panavia, a MDP and silane containing primer, Clearfil Ceramic Primer Plus was applied to all ceramic surfaces (108). With Duo-Link, Z-Prime Plus, containing both MDP and bisphenyl dimethacrylate monomer (BPDM), was applied to zirconia. Two-component silane was mixed and applied to LDS. The manufacturer of Z-Prime Plus state that combining MDP and silane requires a low pH, which will cause instability of the silane, promoting it to hydrolyse and lose bond strength to glass ceramics. Therefore, priming of different types of ceramics should be with separate primers (110). The composition and content of adhesive monomers in primers are reported by some manufacturers (111, 112) but not all (108). This might be due to monomer concentration below threshold for reporting.

Differences in primer composition and monomer content might be reflected in bond strength, as a higher content of MDP has been shown to increase the bond strength of zirconia (113). Also, the use of universal primers instead of separate silane primers might result in lower bond strength for LDS.

Again, the effect of different primers must be evaluated in light of fracture morphology after testing. The only cement with no adhesive fractures when applied to zirconia, considering both tensile and shear bond strength, was Duo-Link. This might be explained by the selective zirconia primer and its adhesive monomers promoting high bond strength to zirconia.

For Panavia, adhesive fractures were observed for Zir A but not for Zir E. This could be related to primer composition and the different surface energy of the two zirconia rods (82). RelyX Unicem was the only self-adhesive cement used in the present project. This cement requires no chemical pre-treatment of zirconia before cementation due to phosphoric acid methacrylate monomers that adhere to the surface (114). A higher frequency of adhesive fractures between ceramic and cement was observed for Zir A compared to Zir E, indicating that the surface free energy might affect the wettability and the adhesive capacity of RelyX. For LDS primed with Monobond Plus and cemented with Variolink (and Multilink) before application of shear forces, a relatively high frequency of adhesive fractures was observed. This indicates that the adhesion to LDS promoted by Monobond Plus is the weakest link in these test specimens.

With a low frequency of adhesive fractures between cement and ceramic in total, no conclusion about the effect of primers can be drawn. Also, with a combination of surface roughness and primer application, the effect of the two interventions cannot be separated.

5.2.2 Cement layer thickness and composition of dual-cure resin cements in relation to bond strength

Cohesive fractures in cement and combined adhesive and cohesive fractures dominated in both tensile and shear bond strength testing, indicating that the cement was the weakest link. A review of bond strength methods and fracture morphology by Scherrer et al. (87) showed that, in macro tensile and shear bond strength testing, the frequency of cohesive and mixed failures were high but dependent on the cement type. This is also reflected in the results of Paper I and III, with some cements showing exclusively cohesive cement and combined fractures.

Differences in cement composition—such as filler particle type, amount, shape and size—are reflected in bond strength and fracture morphology (25). The filler content of Panavia is 76% (115), which is the highest among the tested cements (Table 1). The high filler content is not likely to explain the inferior bond strength results as high inorganic filler contents (>75wt%) have been associated with favourable mechanical properties of resin-based composite material (25). Still, an explanation might be related to size and surface of the particles. Lack of adhesion between resin matrix and the particles, incomplete wetting of the surface of the particles, or unevenly distributed particles in the matrix, may reduce the strength. The base and catalyst of Panavia are deposited on a mixing pad and mixed by hand for 20 s. Compared to auto-mixed cements, Panavia might have a greater risk of becoming an inhomogeneous mixture and this affects both laboratory testing and clinical performance (116). Also, a higher filler content increases viscosity of the cement and results in a thicker cement layer (24). Further, a thick cement layer might result in lower bond strength than thinner cement layers, as shown in Figure 10.

Duo-Link was also in the lower range of bond strength and had a high number of cohesive fractures in cement. This might also be related to the filler characteristics or filler content, which was in the lower range (60%) of cements tested.

Due to the content of MDP in both Clearfil Ceramic Primer Plus and Panavia, a higher bond strength was expected for this cement compared to the others tested. However, this was not the case. In previously published studies by Go et al. (99) and de Souza et al. (117), a combination of MDP containing primer and cement did not result in enhanced bond strength compared to that of MDP containing primer and non-MDP containing cement. The reason for this might be hydrolytic degradation of resin cements and the ceramic-resin bond when exposed to artificial ageing (118, 119). In a study by Shibuya et al. (120) a higher water sorption was observed for the cement with the greatest MDP concentration. This was attributed to increased molecular polarity due to the phosphate group in MDP and further water uptake. Cohesive fractures and combined fractures for Panavia in the present project might be related to degradation of the cement.

Another monomer often added to dental adhesives and cements is HEMA, due to its hydrophilicity which promotes wetting of dentin and increases adhesion. One disadvantage with the hydrophilicity of HEMA is water absorption which may lead to swelling after polymerization (20). In the present project, Multilink was the only cement containing HEMA, and water uptake might have contributed to the cohesive fractures in cement.

One of the cements used has a different composition compared to the other four cements. The powder-liquid system of RelyX contains silanated and alkaline glass fillers and acidic methacrylate monomers which are activated and mixed prior to cementation. The monomers contribute to self-adhesive properties and a high degree of crosslinking in the cement building cohesive strength (66, 68). The bond strength for RelyX was intermediate in both tensile and shear testing, indicating that the cement properties are comparable to traditional resin cements.

The five cement systems used have different adhesive methods to tooth substance (Table 1). Duo-Link has a three-step etch-and-rinse method, which is considered the gold standard for durability of adhesion (13). For Variolink an etch-and-rinse adhesive method is also used, but with only two steps. Two of the cements, Multilink and Panavia, are so-called self-etching with a two-component acidic primer mixed and applied to the tooth substance before cementation. RelyX was the only self-adhesive cement tested.
Adhesive fractures between cement and dentin were mainly observed for Variolink and RelyX in both tensile and shear testing, indicating that adhesion to dentin was a weak link in these specimens.

RelyX requires no pre-treatment of tooth substance due to phosphoric acid groups of the methacrylate monomer that can establish a bond with calcium in hydroxyapatite (18). Immediately after mixing, the cement has a low pH, which is reported by the manufacturer to be crucial for the self-adhesive mechanism (66). A study by Gerth et al. (68) on the interaction of RelyX with dentin revealed a low demineralization effect despite the low initial pH. This could further affect the mechanical adhesion of RelyX to dentin. Observation of a relatively high frequency of adhesive fractures in the present project indicates that adhesion of RelyX to dentin is a weak link.

Prior to cementation with Variolink, dentin was etched with phosphoric acid and applied Adhese Universal. Universal adhesives are compatible with different etching protocols (self-etch, selective enamel-etch and total-etch) due to their pH which leads to demineralizing of dentin in various degrees. However, combining total-etch with a universal adhesive has shown divergent bond strength, as the risk of "over etching" is present (121). With the fracture morphology after bond strength testing in mind, adhesive fractures between Variolink and dentin might be related to the etching strategy chosen.

Tensile bond strength measured in Paper I was compared with cement thickness measured in Paper II to evaluate the effect of cement thickness on bond strength. The observation of the highest bond strength for cement thickness in the range 20 to 35 μm, and the lowest for thicker cement layers, indicated that cement thickness does have effect on bond strength. A study on fracture resistance of cemented glass ceramic published by Rojpaibool et al. (122) showed that a thin cement layer was favourable.

Cement thickness is affected by conditions like filler particle content and size, viscosity, roughness of bonded surfaces, pre-treatment of tooth substance and ceramic, and seating load

(18, 24, 123). As previously mentioned, the filler content of Panavia was the highest among the cements used, indicating that this cement also had the highest viscosity (24). Even though all specimens were loaded with the same weight during cementation, the viscosity of the cements could lead to differences in thickness of the layer. In addition, Panavia was the only cement with a hand-mixing protocol, which was likely to affect the homogeneity of the cement to some degree. The observed significant thicker cement layer for Panavia compared to three of the other cements might be explained by these conditions.

Cement thickness measurements showed significantly thinner layers in test specimens composed of Zir E compared to Zir A (Table 4). The surface roughness measured in Paper I demonstrated significantly different Sa-values between the three rods, with the smoothest surface registered for Zir E. Roughness of the bonded substrates might affect thickness of the cement layer, with deeper grooves and higher peaks contributing to a thicker layer (24). Also, the surface tension of the ceramic affects wettability of the applied adhesive (105). Surface tension of airborne particle abraded and KHF_2 etched zirconia was studied by Akazawa et al. (82), who showed that both the wettability and surface energy were higher after KHF_2 etching. These parameters were not evaluated in the present thesis but could contribute to explaining the difference in cement layer thickness observed for Zir A and Zir E.

The ISO recommends that the thickness for resin cements should not exceed 50 μm (88). In the second paper, both cement thickness in test specimens and cement film thickness using the ISO method were measured. By performing these two measurements, the ISO method was evaluated for its ability to reflect cement thickness when cementing ceramics to dentin. Most measurements of cement thickness in the test specimens were far below 50 μm, showing that the ISO requirements seemed reasonable. However, the measurements of the peripheral cement thickness in cemented test specimens, which reflected the thinnest cement layer achieved, did not concur with the results of the ISO cement film thickness measurements. This indicates that the ISO test method does not directly reflect a clinically relevant cementation procedure. Reasons for the discrepancy could be related to differences in applied loading during polymerization, the use of bonding agents and primers when cementing ceramic restorations to dentin, and the difference in surface roughness between ceramics and glass plates used in the ISO test method. The ISO film thickness measurement method is best suited for comparing differences between cements.

5.2.3 Dentin surface roughness in relation to bond strength of zirconia cemented to dentin

In Paper III, dentin was prepared using P80 and P1200 SiC papers to evaluate the effect of surface roughness on shear bond strength. The SiC papers correspond to very coarse and extra-fine diamond bur grits, respectively, and were selected due to the wide range in surface roughness created. The difference in roughness was confirmed by Sa-measurements performed in SEM. An increase in surface roughness increases the surface area available for physical interaction with the cement, and this has been shown to enhance bond strength of resin bonded ceramics (61)—but this was not the case in Paper III. Dentin roughness seemed to be of importance for RelyX, showing significantly higher bond strength to dentin with a smooth surface. This finding was in agreement with results published by Ren et al. (124), who also demonstrated higher bond strength for a smoother surface when using self-adhesive resin cement, whereas the surface roughness had no significance for the cement with an etch-and-rinse method.

Reducing roughness of dentin using P1200 SiC paper changed the fracture morphology for Variolink. Even though there was no change in bond strength, a decrease in adhesive fractures between dentin and cement was observed. This might indicate that when roughness in dentin decreases, the adhesion between dentin and cement increases.

The two cements have different adhesive methods to tooth substance—with etch-and-rinse protocol for Variolink, whereas RelyX is self-adhesive (Table 1). In the light of the bond strengths registered for the two cements, it is likely that adhesive method and cement properties can explain the differences.

The use of phosphoric acid etching removes smear layer, exposes dentin tubules and assures penetration of bonding and cement. Figure 16a shows resin tags in dentin tubules after shear bond strength testing of ceramic rods cemented to dentin using Variolink. The resin tags ensure a mechanical adhesion of the cement (125), which seems to be of more importance than the surface roughness. Another factor could be the viscosity of the cement, which is mainly affected by filler content (24). The low initial pH of RelyX demineralizes the dentin surface, but the demineralization appears to be shallow compared to etching with phosphoric acid (126, 127). As illustrated in Figure 16b, mechanical adhesion in dentin tubules is lacking for RelyX. The higher bond strength for RelyX to dentin ground with P1200 compared to P80 might be explained by the viscosity of the cement and the limited penetration time to the surface before polymerization. Also, the load (8.7 N) applied on the ceramic rods during cementation might have been too low for the cement to adapt properly to the dentin surface and reduce porosities (61, 126).

6 Conclusions

Airborne particle abrasion and KHF_2 etching of the zirconia surface had no impact on tensile and shear bond strength. The results were comparable to those obtained for HF etched lithium disilicate glass ceramic.

The highest tensile bond strength was registered for cement layer thicknesses between 20 and 35 μm. Thicker cement layers were associated with lower tensile bond strength.

Surface roughness in dentin was of more importance for the self-adhesive resin cement than the cement with an etch-and-rinse adhesive method. For the self-adhesive resin cement, higher shear bond strength was observed for a smoother dentin surface.

To improve the bond strength of ceramics resin cemented to dentin, a cement layer thickness of 20-35 μm—and for self-adhesive resin cement, a smooth dentin surface—are favourable.

7 Clinical implications

Lithium disilicate glass ceramics and resin-based cements are often chosen in cases where a union between the restoration and tooth substance is important. The present thesis has shown that zirconia can replace LDS in such cases and might be the first choice when material strength is also of importance.

The cement layer thickness for ceramic restorations cemented to dentin should be in the range 20-35 μm for increased bond strength. This must be borne in mind when ceramic restorations are fabricated.

The bur grit used for preparing tooth substance for ceramic restorations should be adjusted for the adhesive method of the selected cement. When self-adhesive cement is selected for cementation, an extra-fine bur grit should be used for preparation. For cement with a two-step etch and rinse method, the bur grit is of less importance.

8 Future perspectives

In the present thesis, ceramic rods were cemented to dentin, giving two different interfaces to study—ceramic-cement and dentin-cement—in addition to the cement itself. The bond strength registered only reflects the weakest link in the test specimens and not the adhesive strength in the separate interfaces. To overcome this, test specimens with just one interface—for example, ceramic-cement—should be used in future studies.

Today, translucent and multi-layered zirconia is widely used for restoring both posterior and anterior teeth. Altered crystal structure and increased amounts of stabilizing compounds compared to traditional zirconia give the material a more natural-looking aesthetic, and the mechanical properties are still above those of glass ceramics. In addition, less tooth substance removal is required since the restorations are made monolithically. The effect of different surface treatment methods on translucent and multi-layered zirconia is a popular research field but, to the best of my knowledge, only a limited number of studies evaluating the effect of KHF_2 etching for adhesion to different of resin cements have been published.

The cements used have different adhesive methods to tooth substance. Shear bond strength testing with different surface roughness in dentin revealed that adhesive method affected which roughness was optimal. Further studies on dentin surface roughness and adhesive methods are necessary to find the optimal combinations for the highest bond strengths.

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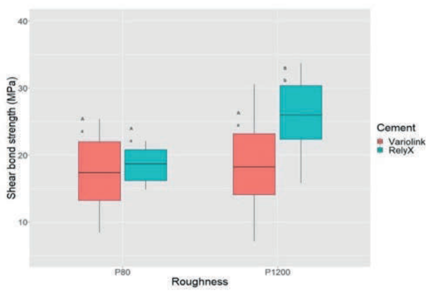
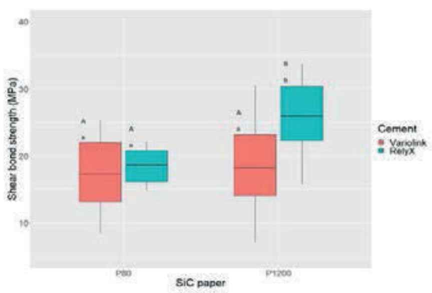
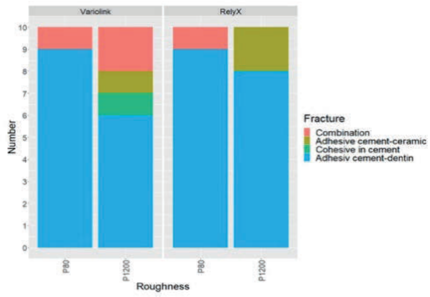
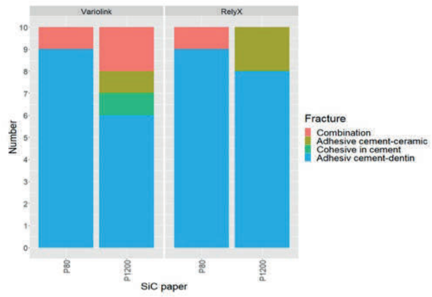
Errata

Page	Line	Original text	Corrected text
14	14	Dentin consists of 50-70 % apatite, 20-30 % organic material..	Dentin consists of 50-70% apatite, 20-30% organic material..
15	23	Filler content is 30-70 % of the cement..	Filler content is 30-70% of the cement..
17	28	The atomic structure is up to 99 % crystalline,..	The atomic structure is up to 99% crystalline,..
25	5	3.1 Test samples	3.1 Test specimens
26	8	2) Hot etching with potassium hydrogen difluoride…280° for 10 min	2) Hot etching with potassium hydrogen difluoride…280 °C for 10 min
26	11	LDS rods were etched with 4.5 % hydrofluoric..	LDS rods were etched with 4.5% hydrofluoric..
31	13	**3.5.4 ISO-cement film thickness**	**3.5.4 ISO cement film thickness**
41	7	Test groups with cement thickness..	Test specimens with cement thickness..
43	Legend	Table 5. ISO Cement film thickness..	Table 5. ISO cement film thickness..
46	x-axis		

47	x-axis		
49	25	..the power reached >90 %.	..the power reached >90%.
49	28	Calculated to be around 21 %. To obtain a power of 80 %..	Calculated to be around 21%. To obtain a power of 80%..
55	9	..observed in one of the cements- Variolink	..observed for one of the cements- Variolink
57	25	The filler content of Panavia is 76 %..	The filler content of Panavia is 76%..
57	27	..high inorganic filler contents (>75wt %)..	..high inorganic filler contents (>75wt%)..
58	9	..lower rang (60 %)..	..lower rang (60%)..
60	9	..composed of Zir E compared to Zir A (Table 5).	..composed of Zir E compared to Zir A (Table 4).
71	5	with different yttria content. Dent Mater. 2021	..with different yttria content. Dent Mater. 2021;37(9):1425-36.
74	1	94. ISO. Dentistry – Polymer-based crown..	94. ISO. 10477:2020 Dentistry – Polymer-based crown..

Papers I-III

I

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Debonding mechanism of zirconia and lithium disilicate resin cemented to dentin

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Debonding mechanism of zirconia and lithium disilicate resin cemented to dentin

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ABSTRACT
To evaluate debonding mechanism of zirconia and lithium disilicate cemented to dentin mimicking what could occur in a clinical setting. A null hypothesis of no difference in tensile bond strength between groups of zirconia and lithium disilicate cemented with resin cements was also tested. Zirconia rods ($n = 100$) were randomly assigned to two different surface treatment groups; air borne particle abrasion and hot etching by potassium hydrogen difluoride (KHF_2). Lithium disilicate rods ($n = 50$) were surface etched by hydrofluoric acid (HF). Five different dual cure resin cements were used for cementing rods to bovine dentin. Ten rods of each test group were cemented with each cement. Test specimens were thermocycled before tensile bond strength testing. Fracture morphology was visualized by light microscope. Mean surface roughness (Sa value) was calculated for randomly selected rods. Cohesive fracture in cement was the most frequent observed fracture morphology. Combination of adhesive and cohesive fractures were second most common. Fracture characterized as an adhesive between rod and cement was not observed for KHF_2 etched zirconia. Highest mean tensile bond strength was observed when cementing air borne particle abraded zirconia with Variolink Esthetic (Ivoclar Vivadent). All surface treatments resulted in Sa values that were significant different from each other. The number of cohesive cement fractures observed suggested that the cement was the weakest link in bonding of zirconia and lithium disilicate.

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KEYWORDS
Zirconia; ceramics; resin cement

Introduction

Zirconia has become one of the most used ceramic in prosthetic dentistry the last decades [1].

The material has a high flexural strength [2] due to its crystal content and transformation toughening from crystal transformation [3]. These characteristics make it appropriate for use as both core material in bi-layered restorations or as monolithical restorations with smaller dimension [4].

Despite excellent mechanical properties of zirconia there are complications related to clinical use. Loss of retention of tooth supported crowns is reported as one of the most frequent technical complication. Many approaches have been studied with the aim to increase bond strength between resin cement and zirconia [5]. Tribochemical silica coating, plasma spraying, selective infiltration technique, hot etching and different lasers have been investigated [6]. The results varied when it came to both tensile and share bond strength in laboratory tests, and storage in water or thermocycling showed low predictability of a stable bond [7].

Air borne particle abrasion using particles of aluminum oxide, diamond or boron nitride is the most used surface treatment [6]. This technique is often combined with 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) containing primer to create a chemical bond [2]. Air borne particle abrasion of zirconia surface has shown phase transformation from tetragonal to cubic and monoclinic crystal structure due to temperature changes [8]. This might reduce flexural strength and potentially lead to fracture [9]. Recommendations from different producers regarding air borne particle abrasion vary, both in particle size and pressure, even if it should be performed as a surface treatment because of potential risks.

High crystallinity of zirconia and lack of glass phase makes the material resistant to etching by hydrofluoric acid (HF). This is in contrast to lithium disilicate, where etching by HF establish micromechanical and chemical bond to silanoles and resin cement [6].

An alternative method of surface etching of zirconia was studied by Ruyter et al. [8]. High share bond

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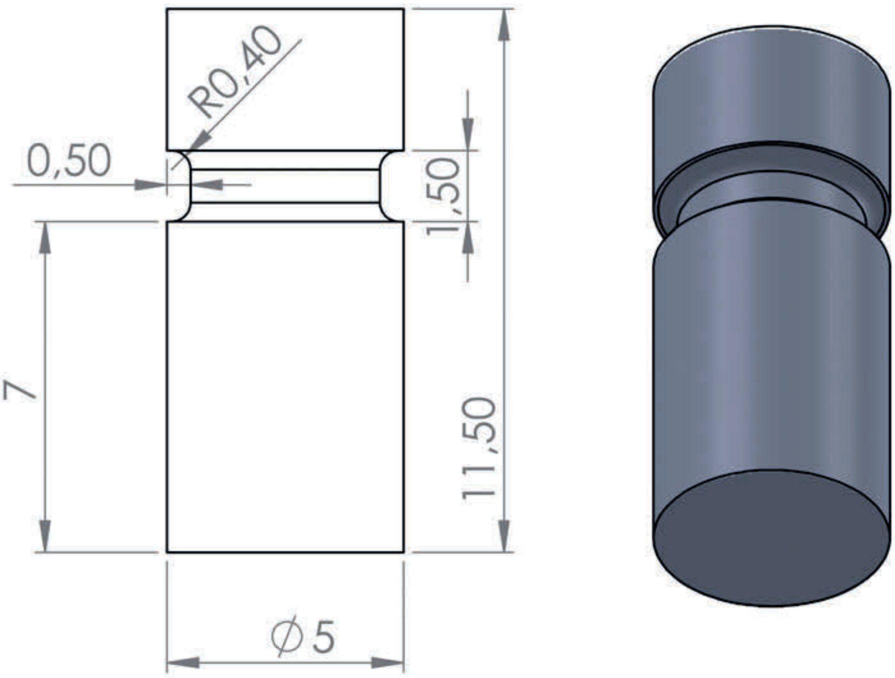

Figure 1. Design of ceramic rod. The illustration shows the dimensions in mm and a copy of the computer aided design (CAD).

strength was observed when fluoride compounds were used for hot etching, and quantitative analysis detected low volume fracture of monoclinic crystals in the surface. SEM images of the surface after testing showed cement partly remaining on zirconia, indicating a strong bond between the etched surface and cement [8].

When cementing ceramic restorations using resin cement, the tooth substance is often pretreated by acidic etch and adhesive components, either as multiple or single step. The pretreatment creates mechanical interlocking and chemical bond between tooth substance and adhesive [10].

In a clinical setting, loosening of the restoration may occur in the weakest part, which represents the bond strength. This could be the cement – restoration bond, in the cement – tooth structure bond or in the cement itself.

The aim of the present study was to evaluate debonding mechanism of zirconia and lithium disilicate cemented to dentin mimicking what could occur in a clinical setting, and to test the null hypothesis that no difference in tensile bond strength between groups of zirconia and lithium disilicate cemented with resin cements would be found.

Materials and methods

Preparation of specimen

Bovine mandibular incisors ($n = 150$) were extracted (from bovine cadaver, 4–6 years old, Nortura), cut 2 cm length and embedded in epoxy resin (EpoFix, Struers) with buccal surface exposed. Embedded teeth were ground at DP-U2 with rotating 500-grit silicon carbide paper (Struers, Denmark) under water until 5×5 mm dentin surface was obtained and further stored in distilled water.

Circular zirconia ($n = 100$, Starceram Z, H.C. Starck Ceramics GmbH, Germany) and lithium disilicate ($n = 50$, IPS e.max CAD, Ivoclar Vivadent, Lichtensein) rods with diameter of 5 mm and length of 11.5 mm were produced by CAD/CAM technique. Rods were produced with a notch in the circumference (Figure 1) facilitating the grip during tensile testing.

One end of the rods was ground with 500 grit silicon carbide sandpaper under water to reflect use of a fine bur in a clinical situation [11,12], and to obtain uniform surface roughness. There after cleaned with a dental steam cleaner (Steamer X3, Amann Girrbach, Austria) and thoroughly air-dried.

Surface treatment of zirconia and lithium disilicate rods

Zirconia rods were randomly assigned to two different surface treatment groups ($n = 50$ each group), lithium disilicate rods ($n = 50$) formed one group. The groups were:

- Zir-A: zirconia, air borne particle abraded, 50 µm aluminum oxide (Al_2O_3, Korox)

Table 1. Materials used for cementing.

Cement	Manufacturer	Adhesive	Manufacturer	Ceramic primer	Manufacturer
Variolink Esthetic	Ivoclar Vivadent	Adhese	Ivoclar Vivadent	Monobond Plus	Ivoclar Vivadent
Multilink Automix	Ivoclar Vivadent	Multilink primer A & B	Ivoclar Vivadent	Monobond Plus	Ivoclar Vivadent
Panavia F2.0	Kuraray Noritake Dental	ED primer 2 A & B	Kuraray Noritake Dental	Clearfil Ceramic Primer Plus, Clearfil SE Bond Primer, Porcelain Bond Activator	Kuraray Noritake Dental
Duo-Link	Bisco	All-Bond 2 primer A & B, Pre-Bond Resin, D/E Resin	Bisco	Z-prime Plus, Bis-silane	Bisco
RelyX Unicem	3M			Bis-silane	Bisco

- Zir-E: zirconia, etched by potassium hydrogen difluoride (KHF_2)
- LDS: lithium disilicate etched by 4.5% hydrofluoric acid (HF)

Five resin based cements were used for cementation of the rods ($n = 10$ of each group with each cement).

Hot etching procedure

KHF_2 was ground to fine powder using a mortar and inflicted equally on the bonding surface of zirconia rods. Thereafter rods were heated in a precalibrated furnace (Jelenko, acc-therm II 2000, NY-USA) for 10 min at 280 °C for the KHF_2 to melt. After cooling, rods were thoroughly steam cleaned and ultrasonically cleaned in distilled water for 15 min. Finally, they were air-dried.

Air borne particle abrasion

Zirconia rods were air borne particle abraded at 2.5 bar for 10 s. The nozzle was kept perpendicular to the zirconia surface at 10 mm distance. Rods were air steamed and ultrasonically cleaned in distilled water for 15 min before thoroughly air-dried.

Hydrofluoric acid

The bonding surface of lithium disilicate glass ceramic rods were etched with hydrofluoric acid (HF 4.5%, IPS Ceramic Etching Gel, Ivoclar Vivadent) for 20 s, cleaned by running water >20 s and thoroughly air-dried.

Surface evaluation

The surface on randomly selected rods was studies in scanning electron microscope (Hitachi Analytical TableTop Microscope/Benchtop SEM TM3030), with energy dispersive spectroscopy, EDS. Surface roughness was measured using a confocal microscope (Sensofar S neox). Mean surface roughness (Sa value) was calculated for randomly selected rods [13].

Cementation

Five different dual cure resin cements were used for cementing rods to bovine dentin; Multilink Automix (Ivoclar Vivadent), Variolink Esthetic (Ivoclar Vivadent), Panavia F2.0 (Kuraray Noritake Dental), Duo-Link (Bisco) RelyX Unicem (3 M ESPE) (Table 1).

Cementation was performed according to producers' manual and primer was applied when recommended (Table 1).

Ten rods from each of the three groups; KHF_2 etched zirconia, air borne particle abraded zirconia and HF etched lithium disilicate, were cemented by each cement.

Dentine was cleaned using pumice powder dispensed in water prior to cementation.

After placing the rods onto dentin, a standardized 882 g seating load was applied by a cementation jig. Excess cement was removed using quick stick microbrush before light curing 20 s each from 4 directions.

All specimens were kept dry at room temperature for 15 min following cementation and thereafter immersed in 37°C distilled water for 24 h.

Specimens were sandblasted using Al_2O_3 to remove cement remnant outside the rods and evaluated by light microscopy. Test units were thermocycled 5000 cycles in 5 °C and 55 °C water baths.

Tensile bond strength testing

Specimens were mounted in a universal mechanical test machine (Lloyd LRX, Lloyd Instruments Ltd, Leicester, UK). Tensile force was applied until break using a centered wire with a cross head speed of 1 mm/min. Figure 2 illustrates the experimental design of tensile bond strength test. Tension force (N) at break was recorded and tensile bond strength

(MPa) calculated in Nexygen DF Force Measuring Software.

Fracture characterization

Rods and dentin were studied in light microscope (American Optical Stereo Star/Zoom, model 570, American Optical Corporation, Buffalo NY, USA. Magnification 10X–63X) for visualizing fracture morphology.

Fractures were classified in to 5 different types: (1) adhesive failure between cement and rod, (2) adhesive failure between cement and dentin, (3) cohesive in cement (4) cohesive failure in dentin, (5) combination of adhesive and cohesive failure.

Statistical analysis

Microsoft Excel (version 14.2.3) was used for calculating mean tensile bond strength and standard deviation. Komogorov–Smirnov was used for calculation of normality and differences among groups were evaluated using ANOVA tests followed by Tukey's HSD test. Evaluations were done (1) among rod materials for each cement and (2) among cements for each rod material. $p < .05$ was regarded as statistical significant different.

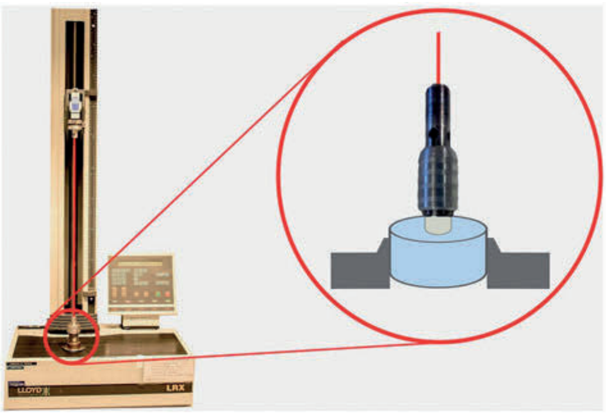

Figure 2. Experimental design of tensile bond strength test. A metallic jig enclosed the ceramic rod at the notch in the circumference for adequate grip. The rod was cemented onto the dentin surface of bovine tooth embedded in epoxy resin.

Results

Fracture morphology

Cohesive fracture in cement was the most common fracture morphology visualized by light microscope, as presented in Table 2. Duo-Link cement showed exclusively cohesive fractures in cement, regardless of test group. Combination of adhesive and cohesive fractures were second most common. Figure 3 show examples of fracture morphology observed in light microscope. Fracture characterized as adhesive between rod and cement was not observed for KHF_2 etched zirconia.

Adhesive fracture between cement and dentin and cohesive fracture in dentin was observed for Multilink Automix, Variolink Esthetic and RelyX Unicem. This was also the cements with the highest tensile bond strength, as shown in Figure 4.

Tensile bond strength

Mean tensile bond strength and standard deviation for the three test groups cemented with different dual cure resin cements are illustrated in Figure 4. Results of ANOVA and Tukey's HSD tests calculated for differences between test rods for each cement and between cements for each rod material are also given in Figure 4. Highest mean tensile bond strength was observed when cementing air borne particle abraded zirconia with Variolink Esthetic and Multilink Automix cement. The lowest bond strengths were obtained with Panavia F2.0 and Duo-Link. There were no differences regarding the effects of the different surface treatments of the zirconia rods for all cements, except for Variolink Esthetic where air

Table 2. Fracture characterization.

	Adhesive						Cohesive								
Fracture type	Dentin-cement			Rod-cement			Dentin			Cement			Combination		
Cement/material	Zir A	Zir E	LDS	Zir A	Zir E	LDS	Zir A	Zir E	LDS	Zir A	Zir E	LDS	Zir A	Zir E	LDS
Multilink Automix			1			2	2	2		1		2	7	8	5
Variolink Esthetic	1		4				3			1	10	2	5		4
Panavia F2.0				2						6	9	10	2	1	
Duo-Link										10	10	10			
RelyX Unicem	5	1						1			2	8	5	6	2

The table show number of adhesive, cohesive, and combined fractures for each material and cement. Rods and dentin were studied in light microscope for visualizing fracture morphology. Fractures were classified into 5 different types based on the type for 2/3 of the surface. Fracture was classified as combined if less than 2/3 was of one specific type.
Zir A: air borne particle abraded zirconia; Zir E: KHF2 etched zirconia; LDS: hydrofluorid acid etched lithium disilicate.

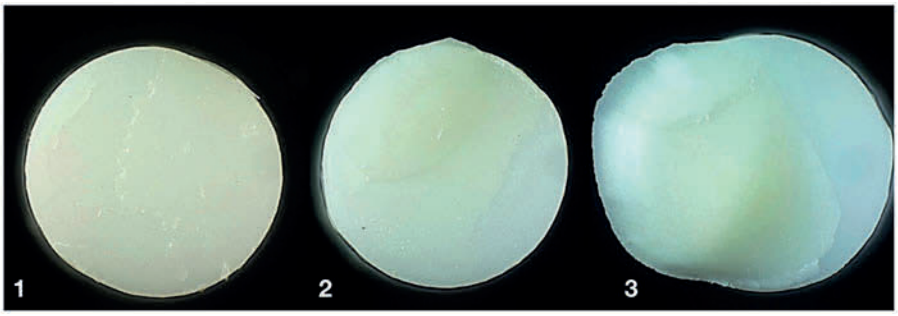

Figure 3. Examples of fracture morphology observed in light microscope (diameter 5 mm). 1: combination of cohesive fracture in cement and adhesive fracture between cement-zirconia; 2: combination of cohesive fracture in dentin and adhesive fracture between cement-dentin and cement-zirconia; 3: combination of cohesive fracture in dentin and cement, and adhesive fracture cement-zirconia.

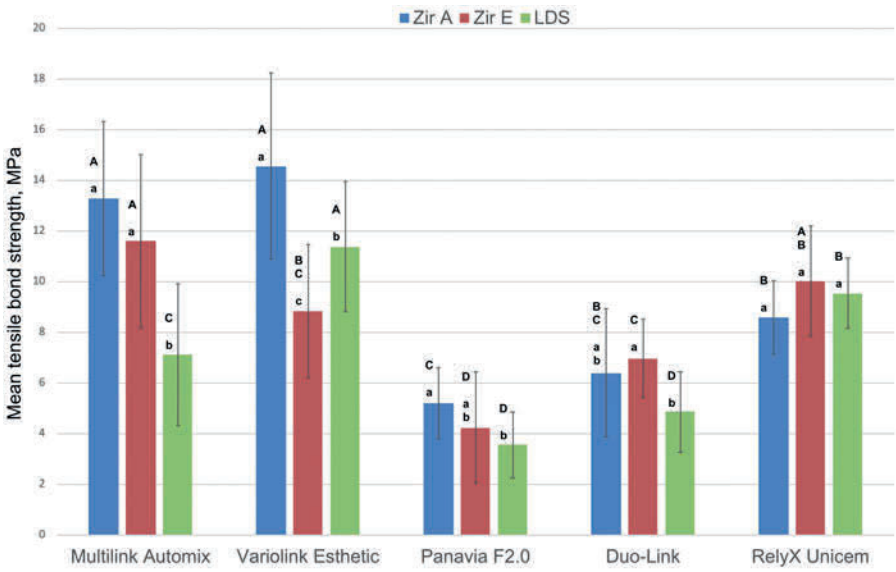

Figure 4. Mean tensile bond strength and standard deviation. Zir A: air borne particle abraded zirconia; Zir E: KHF_2 etched zirconia; LDS: hydrofluorid acid etched lithium disilicate. Different lowercase letters illustrate significant difference ($p < .05$) between Zir A, Zir E, and LDS for each cement. Different uppercase letters illustrate significant differences ($p < .05$) between cements for each rod material.

Table 3. Mean surface roughness (Sa) measured in nanometer and statistical comparison between the groups.

Parameter	Zir A	Zir E	LDS	Zir A/ Zir E	Zir E/LDS	Zir A/LDS
Sa	534–592	127–131	184–255	$p < .01$	$p < .01$	$p < .01$

Zir A: air borne particle abraded zirconia, Zir E: KHF_2 etched zirconia, LDS: hydrofluoric acid etched lithium disilicate.

borne particle abraded zirconia showed higher bond strength. Compared to both zirconia rod types, lithium disilicate rods had lower or similar mean tensile bond strength to all cements except Variolink Esthetic.

Surface evaluation

Sa value after surface treatment of randomly selected rods were measured using a confocal microscope. As presented in Table 3, air borne particle abraded zirconia had the highest Sa value. All surface treatments resulted in Sa values that were significant different from each other. The marked differences in surface morphology of the three test groups are visualized in SEM images (Figure 5).

Discussion

The aim of the present study was to evaluate debonding mechanism of zirconia and lithium disilicate cemented to dentin to mimic what can occur in a clinical setting. A null hypothesis of no difference in tensile bond strength between groups of zirconia and

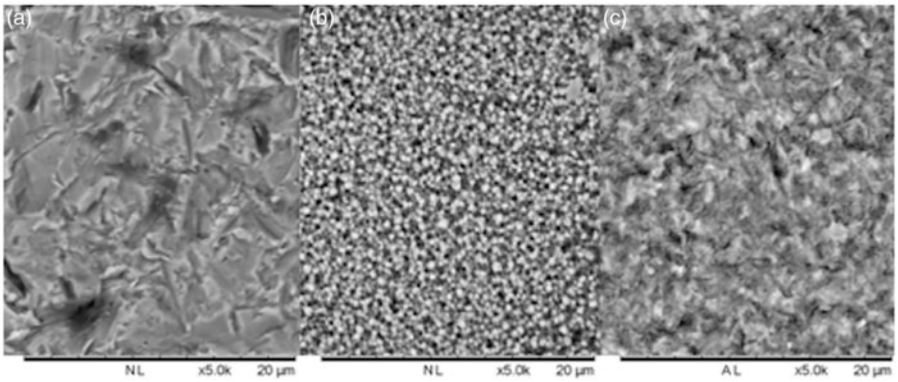

Figure 5. Representative SEM images of air borne particle abraded zirconia (a), KHF_2 etched zirconia (b), and hydrofluoric acid etched lithium disilicate (c). Bar represents 20 μm.

lithium disilicate cemented with resin cements was also tested.

Five dual cure resin cements were used for cementing rods of zirconia (KHF_2 etched and air borne particle abraded) and lithium disilicate (HF etched).

Cohesive fracture in cement was the most frequent fracture morphology visualized by light microscope. Combinations of cohesive and adhesive fractures were second most common.

In a clinical setting loosening of restorations might be because of debonding between cement and dentin, between cement and ceramic or cohesive fracture in cement. Main focus of previous studies on zirconia has been on increasing bond strength between the ceramic and resin cement [6]. Ruyter et al. [8] found increased shear bond strength when zirconia was etched by KHF_2 instead of air borne particle abraded. The authors report adhesive fractures with bonding agent partly remaining on zirconia. In the present study, no exclusive adhesive fractures between cement and KHF_2 etched zirconia were observed, suggesting that other interfaces are important to increase bond strength. Melt etching creates a rough surface of zirconia grains which facilitates the micromechanical retention of coupling agent and luting cement. It is anticipated that by the treatment of zirconia with KHF_2 the surface is fluoridated, which after steam and ultrasonic water treatment is hydrolyzed leaving active hydroxyl groups (OH^-) [8]. Adhesive failure was only detected for two air borne particle abraded zirconia rods and two lithium disilicate rods, cemented with Panavia F2.0 and Multilink Automix respectively. Even though surface treatment of rods in the three different groups were performed as equal as possible, these findings might be explained by variation in micro mechanical and chemical surface properties.

The 10-MDP containing cement, Panavia F2.0, had the lowest tensile bond strength and was the cement with the highest number of cohesive fractures. The low value is in contrast to the previous study on 10-MDP containing cement [14]. 10-MDP is an acid functional monomer with two OH-groups bonded to phosphorous where pka_1 value is 2.2 [15]. Primary chemical bonds to zirconia together with hydrogen bonds can be formed [16].

Multilink Automix was the only cement showing adhesive debonding to lithium disilicate. This was somewhat unexpected finding. Lithium disilicate etched with hydrofluoric acid and primed with silane containing primer is known for establishing micromechanical and chemical bond between resin cement and ceramic [4]. Adhesive fracture between cement and dentin and cohesive fracture in dentin were observed for cements with the highest mean tensile bond strength. This indicates that the bond of zirconia and lithium disilicate to these three resin cements is stronger than the bond between cement and tooth substance.

Combination of adhesive and cohesive fractures in dentin were also common for the three cements with the highest tensile bond strength, this specially applies for zirconia rods regardless of surface treatment. The bonding seemed to be nearly as strong as the inherent strength of the dentin, which must be regarded as the maximum bond strength.

All cements had cohesive fractures to different degree. These observations indicate that cements have different cohesive bond strength that will affect the retention of adhesive cemented restorations.

A thin and uniform cement layer is recommended to reduce shrinkage stresses during polymerization and loading failure of the ceramic [17]. However, there is no standardized procedure for cementing.

In previous studies performed on bond strength of zirconia and lithium disilicate, different loading weight on the cement has been used, 50 N, 750 g, 15 N [8,18,19]. When cementing zirconia and lithium disilicate rods to dentin in this study, a standardized 882 g seating load was applied during light curing. Normally this should result in a uniform cement space for all specimens which will not influence the results.

Cements used today are mainly in the form of automix to ensure equal amount and even mix of components in the cement system. In the present study one cement, Panavia F2.0, was mixed by hand as recommended by the manufacturer. Panavia F2.0 showed the lowest mean tensile bond strength for all three ceramics. The mixing procedure could result in non-homogenous cement and contributed to the weak bond strength.

Micro roughness in the bonding surface of zirconia and lithium disilicate is necessary to establish good bond to resin cement [6]. In this study, surface roughness was created either by air borne particle abrasion or hot/cold etching. All treatments resulted in micro roughness that were significantly different from each other. Increased surface roughness implies a larger surface for bonding and a higher bond strength. Only a few adhesive fractures between cement and ceramic rods were observed. This indicated that other aspects than the surface of zirconia and lithium disilicate was important for the bond strength.

To obtain a clinical perspective in the present study, dentin was chosen as substrate for which zirconia and lithium disilicate were cemented to. Individual differences in dentin are detected in several studies [20] and can affect bonding mechanism and retention of restorations relying on adhesive cementation [21].

Conclusion

The number of cohesive cement fractures observed in the present study suggested that the cement was the weakest link in bonding of ceramics. The null hypothesis of no difference in tensile bond strength between groups of zirconia and lithium disilicate cemented with resin cements was rejected for some combinations.

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Disclosure statement

The authors declare no conflict of interest.

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ORIGINAL ARTICLE

The influence of the resin-based cement layer on ceramic-dentin bond strength

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Abstract

The purpose of the study was to measure the cement thickness obtained when ceramic rods were luted to dentin and to analyze the relation between cement thickness and the previously published tensile bond strength of similar test specimens. In addition, the ISO standard 4049:2019 method was used to determine the film thickness of the used cements. Zirconia (n = 100) and lithium disilicate (n = 50) rods were cemented to bovine dentin using one of five different resin-based cements. The ceramic-dentin test specimens were cut into two slices and the cement thickness was measured using a scanning electron microscope and compared to the bond strength values of similar specimens already published. The mean cement thickness recorded for ceramic rods cemented to dentin was in the range 20–40 µm, which was larger than the cement film thickness found by the ISO method. The cement film thickness determined according to ISO standard methods did not concur with the results obtained when cementing ceramic rods to dentin. For cementing ceramic restorations, a cement thickness in the range 25–35 µm seems to be favorable for the bond strength.

KEYWORDS

finite element analysis, glass ceramics, resin cements, tensile strength, zirconium oxide

INTRODUCTION

Resin-based cements are commonly used for cementing ceramic restorations [1,2]. Their major advantage is greater adhesion between cement and ceramic and between cement and dental tissue compared to water-based cements [3–5]. Resin-based cements are easy to handle, have a fast and regulated setting, and the potential for both mechanical and chemical adhesion [6,7]. Resin cements with different setting modes are available. Exclusively light-activated setting cements are used for cementing thin ceramic veneers, but for cementing restorations with a greater dimension, dual-setting cements are preferable to increase the degree of conversion [8]. Commonly used dual-setting resin cements consist of a resin matrix, activator-initiator systems, silane coupling agent, pigments, and a variable filler content [9–11]. Dual-setting reflects the number and type of initiator: one that is activated by light and the other by a chemical substance [8,12]. RelyX Unicem is a self-adhesive dual-setting resin cement. This cement has a different chemistry compared to other resin cements and is based on glass-ionomer technology reinforced with a light-activated polymerizing resin system [13].

The dimension of the gap allowed for cement between the restoration and preparation is an important factor determining the success and survival of ceramic restorations. May et al. [14] recommended a pre-cement gap around 50–100 µm for resin cements and ceramic crowns. Bonding benefits were lost when cement thickness approached 450–500 µm due to polymerization shrinkage stresses [14,15]. A standard protocol for laboratory cement testing requires that the cement thickness for dual-setting resin cements should not exceed 50 µm [16].

In a previous study [17], the fracture morphologies of test specimens composed of ceramic, resin cement, and bovine dentin were studied in a light microscope to identify crack propagation in tensile testing, a recommended method to test adhesive materials [18]. The results showed a relation between the tensile bond strength and fracture morphology. The test specimens with the lowest tensile bond strength had a higher prevalence of cohesive fractures (that is, crack propagation through the cement alone). This was in accordance with results from bond strength testing by Seitz et al. [19], where the highest frequency of cohesive fractures was observed for the lowest bond strengths.

The aim of the study was to measure cement thickness of dentin-ceramic test specimens and analyze the relation between the thickness and previously published tensile bond strength of similar test specimens [17]. A null hypothesis of no relation between cement thickness and tensile bond strength was tested. In addition, the cement film thickness of the used products was measured according to ISO standard 4049:2019 to investigate if a standardized method could foresee the results obtained when cementing ceramic rods to dentin.

MATERIAL AND METHODS

Preparation of specimen

Specimens were prepared as in the previous study [17]. Cylindrical zirconia (n = 100, Dental Direct Bio ZW; Dental Direkt) and lithium disilicate (n = 50, IPS e.max CAD; Ivoclar Vivadent) rods (diameter = 5 mm) were produced to copy previously used test specimens [17].

Bovine incisors [20,21] were extracted, cut, and embedded in epoxy resin. The buccal surface was ground flat using P500 silicon carbide on Planopol (Struers) rotating grinding machine to create a minimum of 5 x 5 mm exposed dentin surface.

Surface treatment of zirconia and lithium disilicate rods

The surface treatment procedures were performed as in the previous study [17]. The zirconia rods were randomly assigned to one of two surface treatment groups (n = 50 each group): (i) Zir-A: air borne particle abrasion by 50 µm aluminum oxide (Al_2O_3, Korox; Bego), or (ii) Zir-E: hot etching by potassium hydrogen difluoride (KHF_2) [22]. The lithium disilicate rods (LDS, n = 50) were etched with 4.5% hydrofluoric acid (HF, IPS Ceramic Etching Gel; Ivoclar Vivadent) for 20 s.

Cementation

Rods of each ceramic material were cemented to dentin using one of the five dual-setting resin cements being tested (n = 10 rods for each cement) (Table 1). Cementation was performed according to each manufacturer's instructions for use. Specimens were loaded with 8.7 N in a cementation jig during setting.

Cutting of specimens

After 24 h storage in distilled water, the test specimens were embedded in epoxy resin and mounted in a Micracut Precision (Kemet International) cutting machine. Two vertical slices of 2 mm were cut from each specimen with the ceramic rod centered (Figure 1). The slices were kept moist in closed containers. One slice of each specimen was selected for scanning electron microscope study and coated using a combination of platinum (80%) and palladium (20%).

Measurement of cement thickness

A scanning electron microscope (HITACHI SU1510 Variable Pressure SEM; Hitachi High-Tech) was used for studying the cement layer between the ceramic rod and bovine dentin. Each cement layer was imaged in back-scattered

TABLE 1 Cements used in the present study

Cement	Manufacturer	Filler content	Reference
Variolink Esthetic DC	Ivoclar Vivadent	60%–68%	Ivoclar Vivadent [25]
Multilink Automix	Ivoclar Vivadent	61%	Ivoclar Vivadent [25]
Duo-Link	BISCO Dental	62%	Lee et al. [9]
Panavia F2.0	Kuraray Noritake Dental	76%	Hirabayashi et al. [10]
RelyX Unicem	3 M	70%	3 M [26]

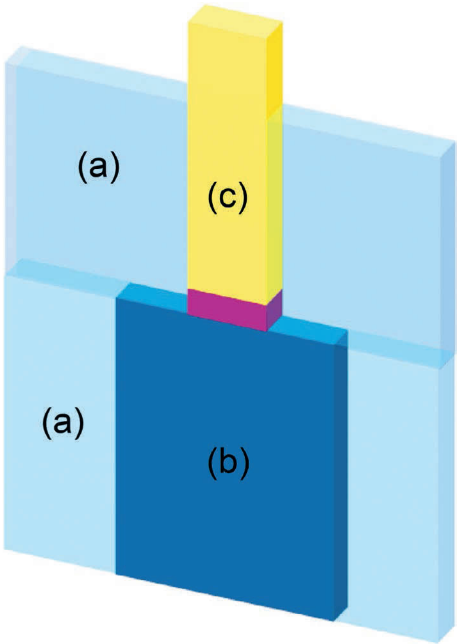

FIGURE 1 Illustration of the specimen after being cut into slices: epoxy resin (A), embedded dentin (B), ceramic rod (C) with resin cement (red)

or secondary electron mode and the thickness measured at five evenly distributed points at 300 x magnification.

ISO cement film thickness

The ISO cement film thickness was measured according to ISO 4049:2019 [16]. Cement was placed between two glass-plates and loaded with 150 N for 2 min before light-activated polymerization. ISO cement film thickness was measured using a micro-meter (Mitutoyo). The procedure was repeated five times for each cement and the median values were calculated.

Geometry of the cement layer

Measurements of the thickness of the cement in the sectioned test specimens were undertaken at five evenly spaced positions across the full width of each section. For some of the test-specimens, it appeared that the ceramic rod surface was oriented with a slight inclination angle to the dentin surface. The thickness at the central point of the cement layer (called cement thickness) and the inclination angle were both estimated for each specimen by linear regression (Thickness = constant + β*distance of measurement) based on the five measuring points. In addition, the thinnest part of the cement layer was derived from the regression parameters and located at the periphery of the rods due to the inclination angle. This was called peripheral cement thickness.

Finite element analysis

Finite element analysis (FEA) was performed to examine the uncertainty in the measured strength introduced by variation in the cement thickness, and to see whether correlation between the average variation for a test specimen and the outcome of the experiments could be explained. A 1292-element and 21168-element models of a rod-cement-dentine-epoxy mounted tensile-test specimens were created in Lisa (version 8.0.0, Lisa-Finite Element Technologies) with refinement of element size down to 1.5 µm for the outer edge of the cement layer in the later model. In a half-section model (Figure 2), the components were divided into sixteen segments of 11° and four segments of 1° about the cylinder axis. To validate the test conditions, the circumference of the epoxy moulding was constrained to zero displacement along the axial direction. The origin along this axis was set at the cement-rod interface.

The three fully bonded components of a specimen (ceramic rod, dentin, and epoxy mould) were assigned the elastic tensile moduli and Poisson ratios given in Table 2. A tensile force summing to the mean force found in tensile tests for each cement was distributed uniformly over the top face of the ceramic rod. Maximum tensile stress was then evaluated for combinations of elastic modulus and Poisson ratio of the cement. The principal stresses in the nodes at and near the edges of the cement section were evaluated for cement layers with uniform thicknesses of 24, 30, 36, and 48 µm and for specimens tilted at the average angle found for that cement. The principal stresses are the three components of stress in a system of coordinates for which shear stresses are zero. They include the largest tensile and compressive stresses acting on the material at the position.

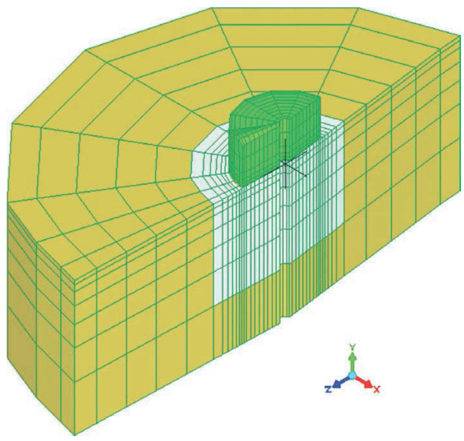

FIGURE 2 Finite element model of the tensile specimens. The components visible are: yellow, epoxy mould; white, dentin; green, ceramic rod. The cement is too thin to resolve. The black cross is at the origin of the axes with direction denoted in the inset. The apparent angle in YZ-plane is an artefact from checking the integrity of the model

The ceramic was then moved in along the axis and rotated in the face of the section to represent the measured cement thickness and its variation. The analysis was repeated for each of the fifteen ceramic-cement combinations with the average cement thickness and inclination found for each combination. Inclination angle was increased by a factor 1.57 over the measured mean value to account for randomness in the direction of the various cement thicknesses relative to the section examined by scanning electron microscopy.

Statistical calculations

The following models were chosen for the regression analysis: (i) Model 1: Cement thickness = $\alpha0 + \alpha1$ Ceramic $+\alpha2$ Cement + ε; And (ii) Model 2: Peripheral cement thickness = $\beta0 + \beta1$ Ceramic $+\beta2$ Cement + ε. ε = error term with random statistical noise. Regression analysis was performed using STATA version 16 (STATACorp). The total sample size in the study was n = 150 (5 cements x 3 ceramics x 10 in each group). A partial R-squared of 0.07 (Model 1) and 0.02 (Model 2) was observed for Ceramic. With a total sample size of 150 and significance level α of 5%, a power of 1-β was 68% (Model 1) and 20% (Model 2) was reached. A partial R-squared of 0.23 (Model 1) and 0.37 (Model 2) was observed for Cement. With a total sample size of 150 and significance level α of 5%, a power of 1- β was 99% (Model 1) and 100% (Model 2) was reached. Power calculation was performed with G*Power version 3.1.9.2. (gpower.hhu.de).

Box plots for cement thickness and peripheral cement thickness were made using ggplot package in R statistical computing (CRAN.org).

Microsoft Excel spreadsheet (Version 16.16.25, Microsoft Office 2018) was used for calculating the correlation between tensile bond strength and cement thickness, and the variation in cement thickness for each cement/ceramic combination.

TABLE 2 Elasticity data employed for each material

Material	Elastic modulus (Isotropic, GPa)	Poisson ratio[a]	Source
Epoxy	3.8	0.4	Tzetzis et al. [27]
Dentine	16	0.29	Palamara et al., [28] Kinney et al. [29]
Cement	6.6–10.4	0.43–0.61	Barbon et al. [24]
Zirconia	>200[*]	0.3	Dental Direkt [30]
Lithium disilicate glass-ceramic	95	0.3	Ivoclar Vivadent [31]

[a]The Poisson ratio is the relative amount by which a body that is stretched longitudinally decreases in a lateral dimension.

*Given the extreme difference between the elastic modulus of the cement and the materials to which it was directly bonded (zirconia and lithium disilicate glass-ceramic), the influence on the stress field in the cement was less than 2%.

RESULTS

The cement thicknesses are given in Figures 3 and 4. The mean cement thickness ranged from 20 to 40 µm. There was a tendency for thinner cement layers with zirconia specimens hot etched by KHF_2 and when using Multilink cement. A similar tendency was observed for peripheral cement thickness measurements.

Results of multiple linear regression analysis for cement thickness and peripheral cement thickness are given in Table 3. Combining all three ceramic types, a significantly thicker cement layer was found for Panavia than for the other types, except Variolink. The same was also observed for both cement thickness and peripheral cement thickness. The thinnest cement layers were observed for Multilink and Duo-Link, and for the Zir E specimens for most cements.

While most specimens showed some variation in cement thickness across the section, a difference of up to 90 µm from

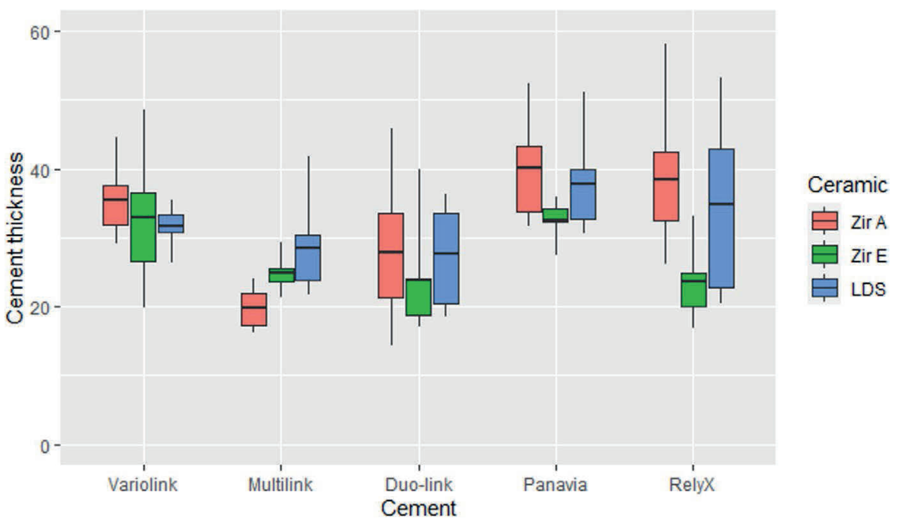

FIGURE 3 Cement thickness is defined as the thickness of the central point of the cement layer of each ceramic-cement combination. LDS, hydrofluoric acid etched lithium disilicate; Zir A, Airborne particle abraded zirconia; Zir E, KHF_2 etched zirconia. The box-plots show mean value (horizontal line), 25% and 75% percentile. Vertical lines represent 90% confidence interval

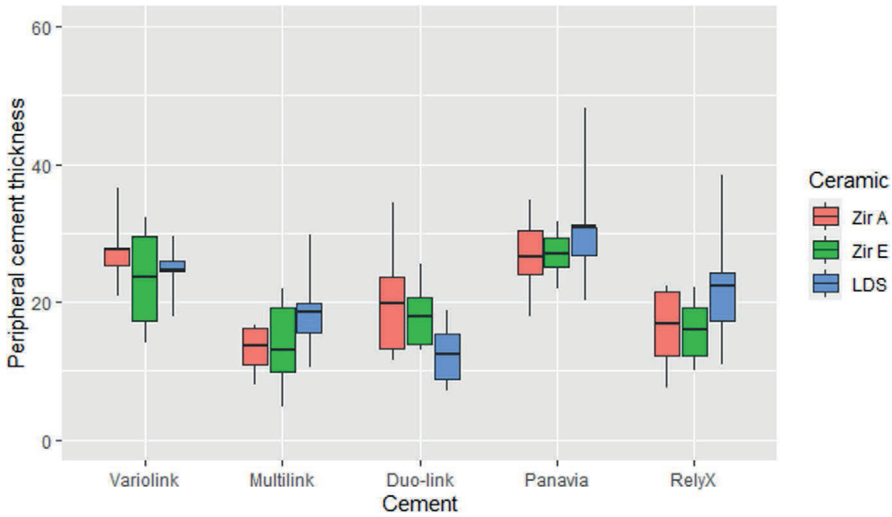

FIGURE 4 The peripheral thickness of the cement layer (called peripheral cement thickness) was derived from the regression parameters and located at the circumference of the rods. This is regarded as the thinnest cement layer. LDS, hydrofluoric acid etched lithium disilicate; Zir A, Airborne particle abraded zirconia; Zir E, KHF_2 etched zirconia. The box-plots show mean value (horizontal line), 25% and 75% percentile. Vertical lines represent 90% confidence interval

TABLE 3 Results of regression analysis of the effect of type of ceramic and type of cement on cement thickness and peripheral cement thickness, respectively. Zir A and Panavia are used as reference materials

	Cement thickness Coefficient, (95% CI)	Peripheral cement thickness Coefficient, (95% CI)
Ceramic		
Zir A (reference)	0	0
Zir E	−4.83 (−8.15, −1.50)	−1.36 (−3.76, 1.04)
LDS	−0.30 (−3.81, 3.21)	0.95 (−1.93, 3.83)
Cement		
Panavia (reference)	0	0
Variolink	−3.46 (−7.01, 0.09)	−2.91 (−6.24, 0.41)
Multilink	−12.32 (−15.70, −8.93)	−13.05 (−16.40, −9.70)
Duo-link	−10.34 (−14.46, −6.21)	−11.47 (−15.18, −7.76)
RelyX	−4.50 (−9.25, −0.26)	−9.78 (−13.43, −6.14)
Constant	38.50 (35.21, 41.80)	28.33 (25.42, 31.24)
R-squared	0.27	0.38
Number of observations	150	150

Abbreviations: Zir A, air borne particle abraded zirconia; Zir E, KHF_2 etched zirconia; LDS, hydrofluoric acid etched lithium disilicate.

one side to the other was found in several specimens. This corresponds to an inclination angle of 1.1 degree between the axis of the ceramic rod and the right angle to the dentin surface. However, the most observations of the inclination angle ranged between −0.3 and +0.3 degree (Figure 5).

The cement thickness measurements were compared to previously published data on tensile bond strength (Table 4) [17] using test specimens with an identical design and are presented in Figure 6.

Test groups with cement thickness of 25–35 µm, especially Multilink and Variolink, appeared to have the highest tensile bond strength, although this observation was not statistically significant. For test specimens with cohesive fractures in cement or combined fractures after tensile testing, a negative correlation (−0.5) with cement thickness was observed (data not shown).

The finite element analyses indicated that the tensile stress in the cement was concentrated at the periphery of the cement layer (Figure 7).

In specimens with varying cement thickness, the maximum computed tensile stress was at the thinnest edge of the cement layer. The analysis showed that the radial and lateral (hoop) stress components were large and tensile, regardless of the elastic modulus and Poisson ratio chosen for the cement. It was noted that the values obtained for the coarse 1292-element and the refined 21,168-element models agreed to within 10%.

All cements fulfilled the ISO requirement for cement film thickness; however, the largest cement thickness was observed for Variolink (Table 5).

DISCUSSION

The aim of the study was to measure cement thickness of dentin-ceramic test specimens and analyze the relation between the thickness and previously published tensile bond strength of similar test specimens [17]. A null hypothesis of no relation between cement thickness and tensile bond strength was tested and accepted. In addition, the cement film thickness of the used products was measured according to ISO standard 4049:2019 to investigate if a standardized

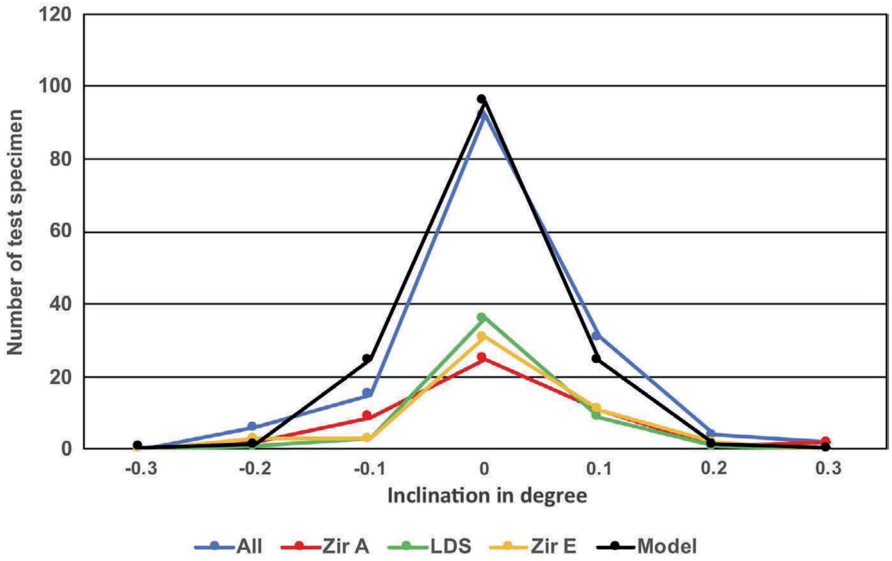

FIGURE 5 Distribution of the observed variation in cement thickness. The y-axis shows number of observations and the x-axis shows the degree of inclination for all ceramics combined (All) compared to a best-fitting normal distribution (Model) with standard deviation 0.28°. LDS, hydrofluoric acid etched lithium disilicate; Zir A, Airborne particle abraded zirconia; Zir E, KHF_2 etched zirconia

TABLE 4 Mean tensile bond strength and standard deviation in MPa for ceramic rods cemented to dentin using five dual-setting resin-based cements, data taken from Sagen et al [17] with permission

Cement	Zir A	Zir E	LDS
Variolink	14.6 (3.7)	8.8 (2.6)	11.4 (2.6)
Multilink	13.3 (3.1)	11.6 (3.4)	7.1 (2.8)
Duo-Link	6.4 (2.5)	7.0 (1.5)	4.9 (1.6)
Panavia	5.2 (1.4)	4.2 (2.2)	3.6 (1.3)
RelyX	8.6 (1.5)	10.0 (2.2)	9.5 (1.4)

Abbreviations: LDS, hydrofluoric acid etched lithium disilicate; Zir A, air borne particle abraded zirconia; Zir E, KHF_2 etched zirconia.

method could foresee the results obtained when cementing ceramic rods to dentin.

Most of the observed failures in tensile testing were cohesive fractures in the cement, suggesting that the cement was the weakest link in the bonding of ceramics [17].

For each test unit, the cement thickness was measured on five evenly distributed points, giving 150 cement layer values for each cement. Linear regression analysis revealed that the cement thickness varied in all groups of test specimens. The inclination angle of the rod with respect to the dentine surface observed in this study raised questions as to whether this would be due to an asymmetry in the apparatus used to load the rod during setting of the cement. If such an asymmetry existed and was greater than any randomly oriented inclination angle generated, for example, by uneven but random application or hardening of the cement, the scatter plot of inclination angle values (Figure 5) would follow a cosine distribution falling to zero for an inclination angle imposed by the apparatus. Otherwise, the scatter plot would follow a normal distribution. Taken over all 150 measurements, the inclination angle was well described by a normal (Gaussian) distribution with a mean of zero and a standard deviation of 0.28°, although values up to 1.1° were observed. Any inclination angle due to a systematic misalignment of the apparatus is no greater than 0.1°.

The thickest cement layer was found for Panavia. The differences were significant when comparing Panavia to the other cements, except for Variolink. Panavia also had the lowest tensile bond strength in the previous published study [17]. The cement thickness was significantly lower when specimens were cemented to Zir E than to Zir A. This could be related to the smoother surface of Zir E, as shown by Sagen et al. [17].

The high number of cohesive fractures reported for Panavia in the former study [17] indicated that the cement was the weakest part of the test unit. Resin-based cements contain filler particles and the filler content of Panavia is 76% [10] which is the highest among the tested cements (Table 1). The high filler content is not likely to explain the inferior results because high inorganic filler contents (>75 wt%) have been associated with the favorable mechanical properties of resin-based composite material [11]. Still, an explanation might be related to the size and surface of particles. Lack of adhesion between the resin matrix and the particles, incomplete wetting of the surface of the particles, or unevenly distributed particles in the matrix may reduce the strength. The base and catalyst of Panavia are deposited on a mixing pad and mixed by hand for 20 s. Compared to auto-mixed cements, Panavia might have a greater risk of an inhomogeneous mixture, which affects both laboratory testing and clinical performance [7].

Properties of the cement, including viscosity, particle size, and applied force during cementation, influence the thickness of the cement. The peripheral cement thickness was on the average 10 μm smaller than the cement thickness (Figures 3 and 4) and represented the thinnest cement layer obtained when cementing ceramic rods to dentin. Determination of the

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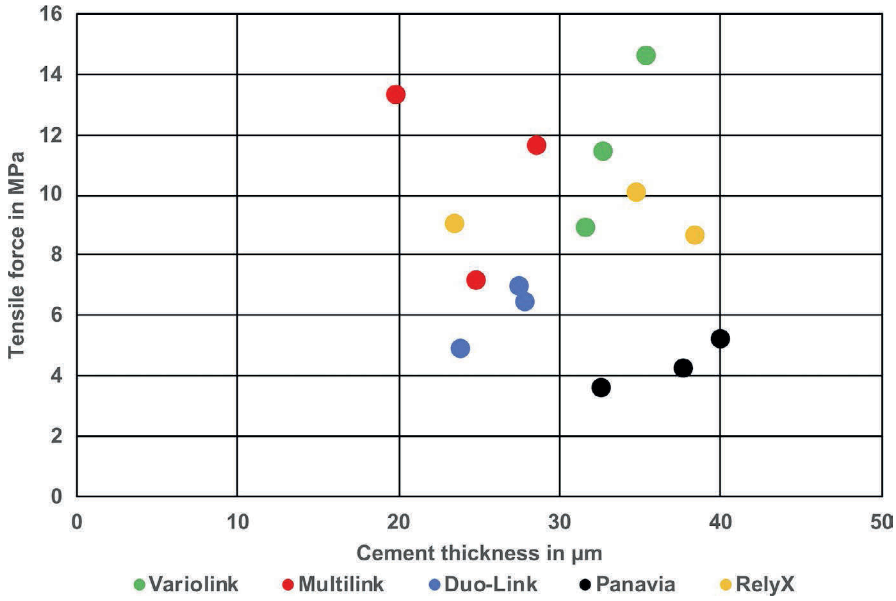

FIGURE 6 Cement thickness measured in the present study plotted against tensile force at break reported by Sagen et al. [17]. Points with same color represent results from ceramic rods with different surface treatment (LDS, hydrofluoric acid etched lithium disilicate; Zir A, Airborne particle abraded zirconia; Zir E, KHF_2 etched zirconia)

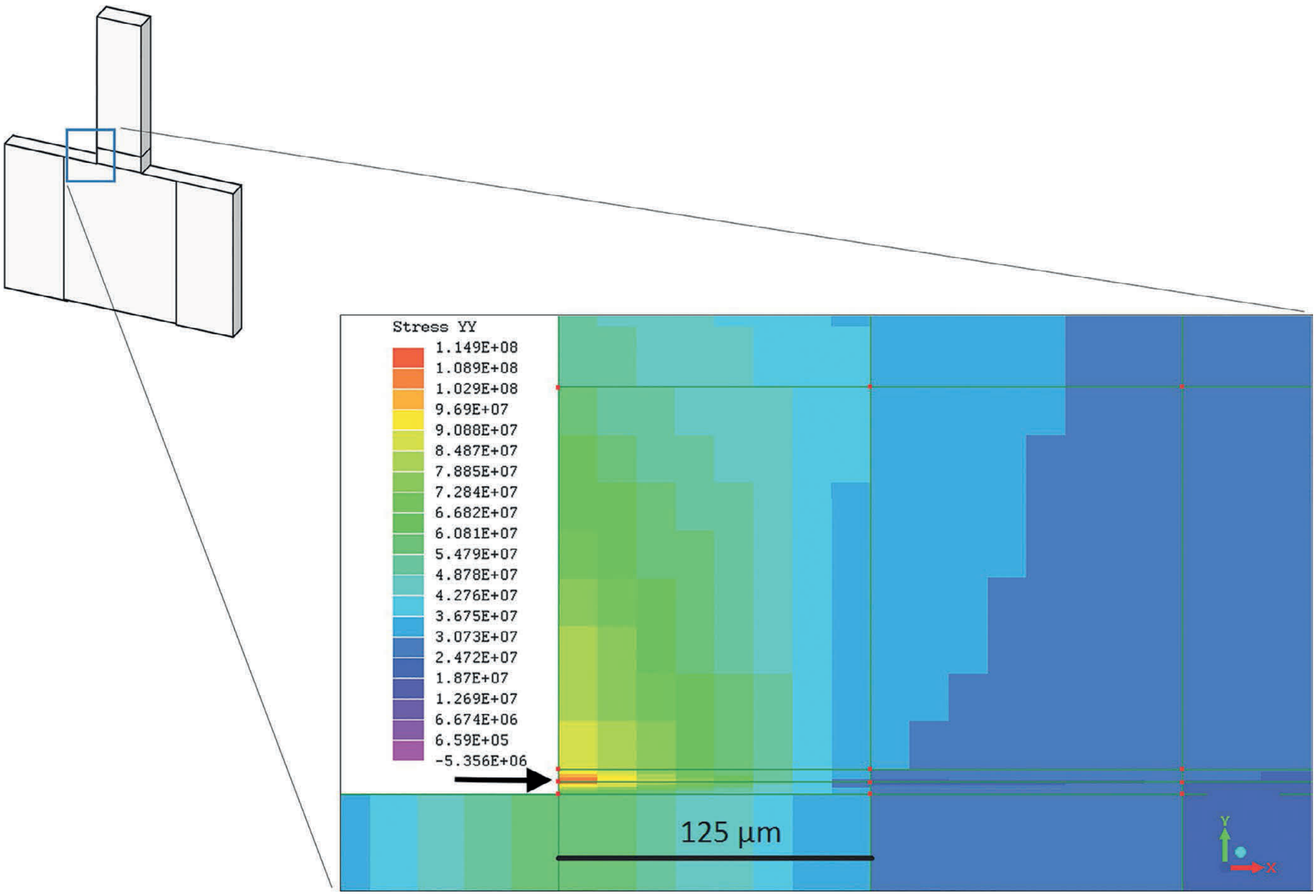

FIGURE 7 Presentation of finite element analysis of stress concentration. Detail of cement (two thin layers of elements extending across the image, black arrow), ceramic rod (above the cement layer), and dentin (below the cement) showing the location of the maximum stress. Color scale for stress (Pa) from violet indicating lowest value to red (highest value) in the vertical Y- direction

ISO cement film thickness should theoretically indicate the minimum expected cement thickness when cementing restorative material to dentin. This was not the case in the present study, except for one of the cements. The reasons for this discrepancy could be differences in the applied load during polymerization and the fact that the cementing situation includes the use of bonding agents and primers that contribute to the thickness of the cement. Another explanation could

TABLE 5 ISO Cement film thickness in µm measured according to ISO 4049:2019 [16]

Measurement	Variolink	Multilink	Duo-Link	Panavia	RelyX
1	12	8	12	6	7
2	11	3	4	11	5
3	21	6	1	5	19
4	21	7	4	4	5
5	22	7	5	10	6
Median	21	7	4	6	6

relate to the roughness of the cemented surfaces. The mean surface roughness of glass is much lower than that of the ceramic rods that were measured by Sagen et al. [17] This may explain the differences observed between ISO cement film thickness and the measured cement thickness.

The mean cement thickness was in the range 20 to 40 µm (Figure 3). When comparing cement thickness with previously published data on tensile bond strength [17], there was a trend for a decrease in tensile bond when the cement thickness exceeded 35 µm (Figure 6). A study on the fracture strength of glass ceramic published by Rojpaibool and Leevailoj [23] showed a significant relation between high fracture load and a thinner cement layer. This indicates that a thin resin-based cement layer is favorable.

The finite element analysis (FEA) indicated that the site of greatest concentration of tensile stress was at the periphery of the cement layer, where it was thinnest because of the inclination of the ceramic rod. In line with this, Barbon et al. [24] observed that fractures started at the border of the specimens in micro tensile bond strength testing. For the ceramic-dentin test specimens, the three principal stresses at the circumference of the cement are all tensile (Figure 7), a situation that is much more likely to initiate fracture in a brittle material (such as hardened resin-composite) even when it exhibits some plasticity. Fractures often initiate in structural flaws in the cement layer. This could be due to the high filler content of Duo-Link and Panavia [9,10], as discussed earlier; the high number of cohesive fractures in the same cements [17] further substantiates that, at least for these cements, the cement was the weakest link in the test unit. Barbon et al. [24] showed than an increase in filler particle content resulted in a more viscous and stiffer resin-based cement and an increase in mixed and cohesive failures were observed. The particle content of RelyX is like that of Duo-Link and Panavia (Table 1). However, these particles are part of the matrix due to the glass ionomer similarity of RelyX [13] and they act differently from the fillers of Duo-Link and Panavia, as they are embedded in the resin matrix. Other possible contributions could be poor polymerization deep within the cement layer [8] and skewed coupling in the test machine so that there is a greater bending moment on the cement [18]. It was found that cement thickness in the range 25–35 µm could be related to the highest tensile bond strength of ceramic rods cemented to dentin. The ISO standard 4049:2019 sets the requirement for cement film thickness at a maximum of 50 µm. Most measurements of the cement thickness of the ceramic-dentin specimens were far below this value, showing that the ISO requirements were reasonable. However, the measurements of the peripheral cement thickness (Figure 4) obtained in cemented test specimens did not concur with the results of the ISO cement film thickness measurements (Table 5). This indicates that the ISO test method does not directly reflect a clinically relevant cementation procedure, especially when it comes to the applied load during setting.

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CONFLICT OF INTERESTS

The authors declare no conflict of interest.

AUTHOR CONTRIBUTIONS

Conceptualization: Mina Aker Sagen, Jon E Dahl, Hans J Rønold; **Methodology**: Mina Aker Sagen, Jon E Dahl, Hans J Rønold; **Investigation**: Mina Aker Sagen; **Software**: John E Tibballs; **Formal analysis**: John E Tibballs; **Resources**: Jukka P Matinlinna; **Data curation**: John E Tibballs; **Project administration**: Mina Aker Sagen; **Supervision**: Jukka P Matinlinna; **Writing- original draft**: Mina A Sagen, Jon E Dahl, Hans J Rønold; **Writing- review & editing**: John E Tibballs, Jukka P Matinlinna; **Visualization**: Mina A Sagen, John E Tibballs.

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