Adhesion and Microleakage of CAD/CAM Crowns
Using Self-adhesive Resin Cements

by

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Graduate Department of Dentistry
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Abstract

Ceramic and composite resin CAD/CAM restorations were introduced as indirect restorative materials that combine high esthetics and superior mechanical properties. However, an important requirement for successful function of these restorations is adequate adhesion between the restoration and tooth substrate. Several factors affect the adhesion of these restorations to the underlying substrate. The aims of this in-vitro project were to analyze some of the factors that can influence adhesion of lithium disilicate ceramic and composite resin CAD/CAM materials to different substrates and to develop more reliable adhesion testing methodologies.

This project consisted of four parts. The first part aimed to develop new microtensile bond strength testing methodology and to evaluate the effect of the resin cement, crown material and bonding substrate on the microtensile bond strength between CAD/CAM crown materials and different substrates. Results indicated that all three variables significantly affected mean microtensile bond strength.

The second part aimed to modify the current microshear testing methodology and to evaluate the effect of the resin cement, crown material and bonding substrate on the
microshear bond strength between CAD/CAM materials and different substrates. Results indicated that the type of resin cement and substrate significantly affected the mean microshear bond strength. Crown materials had no significant effect.

The third part aimed to develop a methodology to measure the retention strength of CAD/CAM crowns to the underlying substrate and to evaluate the effects of the resin cement, crown material and bonding substrate on the retention strength. Results indicated that all three variables significantly affected mean retention strength.

The fourth part aimed to test microleakage at the interface between CAD/CAM crown materials and tooth substrate and evaluate the effects of the resin cement and crown material on the sealability of the bonding interface. Results indicated that only the cement had a significant effect on microleakage.

In conclusion, etch-and-rinse adhesive resin cement provided better bond strength and retention strength and adhesive-interface sealability when used with lithium disilicate and composite resin CAD/CAM crown materials. Furthermore, the bonding substrate had an effect on adhesion; unrestored teeth provided better retention than teeth restored with composite resin and amalgam.
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Chapter 1 Introduction and Literature Review
1. Introduction

The increased demand for esthetic and biocompatible restorations has led to development of new materials and restorative systems. Among these available restorative systems is the CEREC computer–aided-design/ computer-aided-manufacturing (CAD/CAM) system which enabled dentists to provide the patient with an indirect ceramic or composite resin restoration in a single appointment with simpler procedures compared to conventional restorative systems and without the need for dental laboratory support.

Ceramic and Composite resin materials are available for the CAD/CAM restorations in the form of blocks ready to be milled for restoration fabrication. These blocks are characterized by their uniform high mechanical properties that give them advantage over materials of similar composition used for laboratory fabricated restorations following traditional techniques. However, adequate adhesion between these restorations and tooth substrate is a prerequisite for proper function and longevity of the restorations.

Resin cements are utilized to gain adhesion between indirect esthetic restorations and the tooth substrate. Self-adhesive resin cements simplify the clinical procedures and are less technique sensitive compared to adhesive-based resin cements.

The inner surface of the ceramic CAD/CAM restorations is roughened by acid etching and/or grit-etching to achieve micro-mechanical interlocking. Also, silane-coupling agents can be utilized to attain chemical bonding between ceramic restorations and resin cements. For composite resin CAD/CAM restorations, although CAD/CAM milled surfaces may be sufficiently rough for routine bonding, they are further roughened by grit-etching for better micro-mechanical interlocking. Also, silane-coupling agents can be used to enhance the chemical bond between these restorations and resin cements. Hydroflouric acid etching of
composite restorations can damage the resin matrix and decrease both the mechanical properties and the bonding performance of the restoration.\textsuperscript{9-12}

Several factors influence the bond strength between the CAD/CAM restoration and the underlying bonding substrate, such as the type of the substrate whether it is an intact tooth structure or a tooth with composite resin or amalgam restorations, the type of surface treatment in the indirect restoration, the type of the resin cement, and the method of adhesion testing.\textsuperscript{4,13,14} Several tests can be used to evaluate adhesion including bond strength tests, retention tests and microleakage tests. To evaluate bond strength, the most common in-vitro adhesion tests are shear, tensile, microshear and microtensile bond strength tests.\textsuperscript{15} The microtensile bond strength test has several advantages as opposed to the shear and tensile bond strength tests such as cutting multiple specimens from one large specimen and stress concentration at the adhesive interface during loading.\textsuperscript{16} This results in higher bond strength values with less cohesive fractures.\textsuperscript{17-19}

Also, the microshear bond strength test was used in some studies aiming to generate multiple specimens from a single tooth.\textsuperscript{20,21} In some studies, microshear bond strength test was more accurate in bond strength evaluation than the microtensile bond strength test, as the latter showed high standard deviations and failed to differentiate between different adhesives regarding the bonding performance.\textsuperscript{21,22} Also, the microshear bond strength test appeared to be more capable of concentrating the stresses at the interface as it was mainly associated with adhesive failures at the interface compared to microtensile bond strength which showed a high percentage of cohesive failure of the bonded substrate.\textsuperscript{21}

Retention tests, as opposed to bond strength tests, were developed to consider the complex geometry of the abutment preparation where the bonding interface consists of several
surfaces. The configuration factor (C-factor) is the ratio of the bonded to the unbonded surfaces, \(^{23}\) is much higher in cemented crowns compared to cemented cylinders and can affect adhesion. \(^{15,23,24}\)

Microleakage and adhesive performance are strongly connected. Microleakage could lead to failure of adhesion, and weak adhesion could lead to microleakage. Microleakage tests the ability of cement to seal the adhesive interface and could lead to secondary caries and even pulpal pathology if left untreated. \(^{25,26}\)

2. CAD/CAM Technology

2.1 Brief history

Fabrication of conventional metallic restorations starts by taking an impression of the prepared tooth, then pouring the impression with a gypsum material for model fabrication, then waxing up on the created die, and finally casting using the lost wax technique. In contrast, CAD/CAM technology uses digital impressions of the prepared teeth that are directly recorded intraorally instead of taking conventional impressions. The created virtual impression is manipulated using computer aided design software to design the restorations on a computer monitor as a digital wax-up. Finally, a computer-aided processing machine mills the restoration from a prefabricated ceramic, composite or metal block. This computer-aided manufacturing technology has been applied in different industrial fields since the 1970s, however, major developments of dental CAD/CAM technology occurred in the 1980s. \(^{27}\)

Three pioneers have contributed to the development of the dental CAD/CAM systems. The first was Dr. Francisco Duret of France. In 1971, he started to fabricate crowns with functionally shaped occlusal surfaces using a series of systems that started with scanning the prepared tooth intraorally using an intraoral camera. \(^{28}\) The created virtual impression was then
reconstructed on a monitor as a three-dimensional graphic and the optimal shape of the crown was ‘virtually designed’ on the monitor. The crown was then fabricated by milling a block using a numerically controlled milling machine. Dr. Duret developed the commercial Sopha® System, which had an impact on the later development of dental CAD/CAM systems in the world. However, this system was not widely used due to some technical limitations such as the lack of digitizing accuracy, limitations of computer technology and materials not fully-developed to apply this system in dentistry.

The second pioneer was Dr. Werner Moermann of Switzerland, the developer of the CEREC® system. CEREC is an abbreviation for (computer-assisted CERamic REConstruction). Early in the 1980s, posterior composite restorations had inherent problems such as polymerization shrinkage, marginal gap formation, and inferior mechanical properties. Therefore, Dr. Moermann decided to apply new technology in the dental office to fabricate indirect esthetic restorations and overcome the drawbacks of conventional composite restorations. The basic concept hardware was first produced by Moermann and his colleagues in 1980, and was developed later to the CEREC 1 system in 1985, which was capable of capturing digital images of powdered teeth with a compact intraoral camera, designing and milling the inlay from a ceramic block using a chair side machine.

The development of this system allowed the fabrication of same-day ceramic restorations. However, the application was limited to inlays, since the occlusal morphology was not initially accurately reproduced. When this system was introduced, the technical term of CAD/CAM became popular to the dental profession.

In 1988, Moermann and his colleagues improved the CEREC 1 system to be capable of fabricating chair-side inlays, onlays and veneers. In 1994, Siemens developed the CEREC 2
system, which was capable of fabricating inlays, onlays, veneers, partial and full coverage crowns. In 2000, Sirona developed the CEREC 3 & inlab system, which was capable of fabricating three-unit bridge frames in addition to other restorations fabricated by CEREC 2. All these CEREC systems were using two-dimensional software.

In 2003, Sirona developed the CEREC 3D system capable of producing four-unit bridge frames with three-dimensional software. In 2005, Sirona improved the CEREC 3D which was capable of automatic virtual occlusal adjustment.

In 2009, Sirona developed the CEREC AC (acquisition center). This system enables the fabrication of restorations both at chairside and at the laboratory by sending digital impressions to the laboratory, it is characterized by a very high degree of accuracy and unprecedented speed. The intraoral digital impression is taken as multiple digital images of powdered prepared teeth using Bluecam. The technique is very fast and captures multiple images. This is convenient as it removes the necessity of a pedal or button. More recently, the Omnicam was introduced which allows powder-free scanning and precise 3D images.

Two companies developed CAD/CAM systems that are currently available for in-office chair-side use. Sirona Dental Systems developed the CEREC® 3D and CEREC® AC, while E4D Technologies developed the E4D Dentist. They are capable of capturing digital impressions and chair-side milling of restorations.

The E4D Dentist (D4D Technologies, Richardson, Texas) was introduced in 2008 as powder-free chair-side CAD/CAM system capable of capturing digital impression, designing and fabricating non-metallic inlays, onlays, crowns and veneers.
CEREC AC and E4D systems allow capturing the digital impressions and data transmitted to the laboratory for the design (CAD) and milling process using CEREC Connect and E4D LabWorks respectively.\(^{36-38}\)

Other CAD/CAM systems that are used for crown and fixed partial denture (FPD) restorations require production of a stone model for the abutment teeth using conventional impression methods, which acts as the starting point. A digital impression of the stone model is recorded to design restorations using computer software and then various processing machines can be used to fabricate the restorations. These systems can be used as a laboratory tool by dental technicians.\(^ {27}\) Also, there are systems that require completing the wax up, as in the conventional method, and then digital images are captured for wax patterns followed by restoration milling.\(^ {27}\)

The third pioneer is Dr. Matts Andersson of Sweden, who developed the Procera® system. In the early 1980s, nickel-chromium alloys substituted gold alloys in dental applications due to the very high expense of gold. However, nickel allergies appeared as a problem and non-allergic biocompatible titanium was proposed as a substitute. However, casting of the titanium was difficult and technique sensitive. So, Dr. Andersson used spark erosion to produce titanium copings using CAD/CAM technology and this system was known as the Procera system.\(^ {39,40}\)

Later on, a processing center was introduced and connected to satellite digitizers around the world through the Internet to fabricate all ceramic frameworks. Single or multiple unit ceramic frameworks were made from high strength industrial sintered polycrystalline alumina or zirconia ceramic materials that were not used in conventional dental laboratories. A digital impression of the prepared tooth die was recorded at the satellite laboratory office and the data
was transferred to processing centers based in Sweden and the USA,\textsuperscript{41,42} where copings were fabricated and delivered back to the laboratory office for layering porcelain. CAD/CAM technology played a part within the total restoration-fabrication process.\textsuperscript{39,41} Also, customized titanium and ceramic implant abutments could be manufactured using the CAD/CAM software program for this system.\textsuperscript{39,41}

### 2.2 Advantages of CAD/CAM technology

The advantages of using CAD/CAM technology in fabricating non-metallic restorations can be summarized as:

- CAD/CAM technology allows the use of high-strength newly-developed materials in the fabrication of dental restorations, which cannot be processed using conventional laboratory methods. This in turn allows the dentist to avoid the potential for errors that occur during laboratory processing.\textsuperscript{27,29}

- The application of CAD/CAM technology saves time and effort by eliminating the need for lengthy conventional clinical and laboratory procedures.\textsuperscript{27,42}

- The cost is reduced by eliminating the need for some materials that were used in conventional clinical and laboratory procedures. Also, the cost is reduced due to mass-production of the prefabricated ceramic or composite resin blocks.\textsuperscript{42,43}

- The CAD/CAM technology decreases the number of visits to the dental office and allows the fabrication of chair-side indirect all-ceramic restoration.\textsuperscript{42,44}

- The use of CAD/CAM technology enables quality control of dental non-metallic restorations through reproducible processing. The quality of the ceramic and composite resin blocks, from which restorations are milled, is confirmed by the manufacturer to be
free from internal defects, whereas internal porosity is usually present in restorations processed by conventional lab procedures.\textsuperscript{27,42}

### 2.3 Limitations of the CAD/CAM technology

- Although several dentists may share a centrally located CAD/CAM machine, the equipment is very expensive\textsuperscript{35,45}
- It is difficult to digitize subgingival margins in severely broken teeth. Conventional gingival retraction procedures are required in these cases.\textsuperscript{34,45}
- The use of monochromatic blocks makes the color of the restoration unnatural looking, however, some polychromatic blocks are now available and are specially layered to simulate the natural appearance of the enamel and dentine.\textsuperscript{45,46}
- More time is needed to adjust and polish the CEREC restorations compared to that required for conventional laboratory fabricated restorations. However, recent software developments and experience allows dentists to adjust and polish the restorations faster and more efficiently.\textsuperscript{45,47}

### 2.4 CAD/CAM materials

The reliability of the CAD/CAM materials is related to the reproducibility of the manufacturing process. The CAD/CAM blocks are manufactured in an identical manner to produce dense and void-free material.\textsuperscript{42} All CAD/CAM material blocks are characterized by fine-particle microstructure that results in reduced machining damage, improved polishability, decreased abrasion coefficient and improved mechanical properties.\textsuperscript{46}

Several materials can be used with chair-side CEREC system and dental laboratory-based CEREC inlab system. These materials include:
• IPS e.max CAD (Ivoclar Vivadent, Schaan, Liechtenstein): a lithium disilicate glass-ceramic processed while still in the partially crystallized (soft state). In the soft state, IPS e.max CAD shows its characteristic bluish color. Manual adjustments and sprue cut back can be easily performed and the fitting accuracy of the restoration can be checked. Crystallization process (approx. 20 min) is then performed in a ceramic furnace to increase the flexural strength of the material to 360 MPa and to achieve the desired esthetic tooth shade and translucency. This material can be used for the fabrication of veneers, inlays, onlays, partial coverage and full coverage crowns as well as frameworks.46,48,49

• Paradigm MZ100 (3M ESPE, Minnesota, United states): a highly filled (85-90 percent by weight) resin-based composite with micrometer and sub-micrometer zirconia-silica fillers. It has higher mechanical properties compared to conventional Z100 Restorative direct resin-based composite and other direct resin-based composites. The material was developed as an alternative to porcelain, and it has the advantages of being dense, uniform, free of polymerization shrinkage, with a low abrasion coefficient which gives it enamel-like wear characteristics, easy to finish and polish and can be easily repaired intraorally. The flexural strength of this material is 150 MPa and can be used for the fabrication of veneers, inlays, onlays, partial coverage and full coverage crowns.46,48

• Lava Ultimate (3M ESPE, Minnesota, United states): a highly filled (80 percent by weight) resin-based composite with silica and zirconia nanomer and zirconia-silica nanocluster fillers. Similar to paradigm MZ 100, it is dense, uniform, and free of polymerization shrinkage, which makes it superior to other direct resin-based composites. The flexural strength of this material is 200 MPa. The material has a low
abrasion coefficient and can be easily repaired intraorally. This material can be used for the fabrication of veneers, inlays, onlays, partial coverage and full coverage crowns.\textsuperscript{50}

- **Paradigm C (3M ESPE, Minnesota, United States):** a leucite-reinforced glass ceramic material that contains 30 percent by weight leucite. Paradigm C blocks are the newest addition to the Paradigm line of blocks named Paradigm C where “C” identifies it as a ceramic. It is characterized by its well-balanced translucency and fluorescence, by providing a chameleon effect for good shade matching and by its ease to polish. This material can be used for the fabrication of veneers, inlays, onlays, partial coverage and full coverage crowns.\textsuperscript{48,51}

- **Vitablocs Mark II (Vita Zahnfabrik, Bad Sackingen, Germany):** fabricated using fine-grained feldspathic porcelain powder particles that produce a nearly pore-free ceramic with fine crystals.\textsuperscript{46,52} This material is characterized by improved polishability, increased strength and a relatively low abrasion coefficient which results in decreased wear of opposing enamel.\textsuperscript{52,53} The flexural strength of this material is 130 MPa and can increase to 160 MPa when glazed, which is twice the strength of conventional feldspathic porcelains.\textsuperscript{46,54} This material can be used for the fabrication of veneers, inlays, onlays, partial coverage and full coverage crowns.\textsuperscript{46,48,52,55}

- **CEREC Blocs (Sirona Dental Systems, Chiswick, United Kingdom):** a feldspathic porcelain, similar to Vitablocs Mark II but with a different shading nomenclature. This material is characterized by its ease of polishability and high degree of translucency and provides a chameleon effect to blend perfectly with the adjacent teeth. The polychromatic version of the CEREC Blocs is specially layered in order to resemble the
enamel, dentine and neck of a natural tooth. This material can be used for the fabrication of veneers, inlays, onlays, partial coverage and full coverage crowns.\textsuperscript{48,56}

- CELTRA Duo (Dentsply, York, U.S.A): zirconia reinforced lithium silicate glass ceramic. This material is characterized by its high flexural strength (370 MPa) similar to that of the IPS e.max CAD, however, it doesn’t require a crystallization step. This material can be used for the fabrication of veneers, inlays, onlays, partial coverage and full coverage crowns.\textsuperscript{57}

- VITA ENAMIC (Vita Zahnfabrik, bad Sackingen, Germany): a hybrid ceramic with a polymeric network that interpenetrates the ceramic network providing reinforcement and adding resilience. This material is characterized by a flexural strength of 160 MPa. This material can be used for the fabrication of veneers, inlays, onlays, partial coverage and full coverage crowns.\textsuperscript{58}

### 3. Bonding of Non-metallic CAD/CAM materials

Proper function and serviceability of CAD/CAM restorations depends on their good adhesion to the underlying substrate\textsuperscript{3,59}. Proper bonding to tooth structure can be achieved when an appropriate cement is selected, proper surface treatment of the internal surface of the restoration is performed and the recommendations of the manufacturer are followed.\textsuperscript{60,61}

Laboratory bond strength testing can be performed to predict the clinical performance of these materials.\textsuperscript{20}

Resin cements are used clinically to bond non-metallic restorations to the tooth structure due to their esthetics, good mechanical properties and high bonding performance.\textsuperscript{62,63} Resin cements can bond to tooth structure either through one of the adhesive systems currently available (adhesive-based resin cements) or by direct application to the tooth structure without
the need of an adhesive system (self-adhesive resin cements). They can also bond to the non-metallic restoration following proper surface treatment of these restorations. 60,61,64,65

3.1 Adhesive systems

Throughout the last two decades, new adhesive systems have been introduced for bonding to tooth structure. Adhesive materials can interact with tooth structure either mechanically, chemically, or both. 66

The adhesive systems currently used for bonding to tooth tissue can be summarized as follows:

3.1.1 Total Etch Systems (Etch-and-Rinse Adhesives)

Total Etch is a multi-step approach that includes a separate etch-and-rinse phase. An acid (usually 30-40% phosphoric acid) is applied for 15 seconds on dentin and then rinsed off. This demineralizes dentin up to a depth of a few micrometers resulting in the exposure of a hydroxyapatite-deprived collagen mesh. Etching is followed either by priming then the application of the adhesive resin (three-step procedure), or by applying a combined primer and adhesive resin (two-step procedure). 66,67

The total etch technique is the most effective approach to achieve bonding to enamel. Etching results in dissolution of the hydroxyapatite crystals creating micro-porosities that are infiltrated by fluid resin, which polymerizes and forms resin tags. Two types of resin tags are formed: macro-tags that fill the space surrounding the enamel prisms, and micro-tags that fill the micro-porosities within the etched enamel prisms and contribute greatly to the retention in enamel. 20,66
Etching dentin dissolves and removes the smear layer leaving a rinsed collagen layer, which, when resin is applied, produces a resin–collagen hybrid layer, that is susceptible to degradation upon water sorption and enzymatic degradation process.\textsuperscript{68-70}

Chemical bonding between resin and the organic component of dentin that remains after the acid etch procedure contributes to a better bonding performance. However, this chemical bonding is lacking due to hydrophilicity of the resins and hydrolytic reactions taking place at the adhesive interface. Chemical bonding can be regarded as the major shortcoming of today’s etch-and-rinse adhesives.\textsuperscript{71,72}

The priming step is very critical in the total etch approach. The highly technique-sensitive “wet-bonding” technique should be used when using an acetone-based adhesive.\textsuperscript{73} The less technique-sensitive “dry-bonding” technique, by applying a gentle stream of air-drying following the rinsing step of the acid, should be employed when a water/ethanol-based adhesive is used.\textsuperscript{74,75}

### 3.1.2 Self-etch Systems (Etch-and-Dry Adhesives)

These adhesives use monomers that were grafted with one or more carboxylic or phosphate acid groups, to be able to simultaneously condition and prime dentin.\textsuperscript{76} This approach eliminates the rinsing step, resulting in shortening of the clinical application time and reducing the technique-sensitivity of the adhesive system application.\textsuperscript{77} Also, the infiltration of monomers occur simultaneously with the self-etch process eliminating the possibility of discrepancies between both processes and reducing the presence of unprotected collagen fibrils, which in turn reduces nanoleakage.\textsuperscript{66,78}
All self-etch adhesives incorporate water as a solvent in their composition in order to allow the ionization of the incorporated acidic monomers to be capable of etching the tooth structure. Self-etching adhesives, only dissolve the smear layer without removal of the dissolved calcium phosphates since the rinsing step is eliminated. They can be classified according to their acidity and etching power into: strong (pH ≤ 1), intermediate strong (pH≈1.5) and mild (pH≥2).

Strong self-etch adhesives dissolve more hydroxyapatite crystals that become embedded within the interfacial zone. At the enamel level, the etching pattern is similar to that of phosphoric acid treatment used in the total etch approach. At the dentin level, the collagen network is exposed and nearly all the hydroxyapatite crystals are dissolved to a depth of 3 μm similar to what happens in the total etch approach, and the transition of the exposed collagen network to the underlying unaffected dentin is quite abrupt.

Strong self-etch adhesives are associated with low laboratory and clinical dentinal bonding performance, especially at the dentin substrate. This may be due to the soluble calcium phosphates that are embedded in the interfacial zone. Also, the high concentrations of acidic resin monomers make these adhesives behave like hydrophilic permeable membranes and allow water movement from dentin to the restoration-adhesive interface. In addition, the residual solvent that remains within the adhesive interface can weaken the bond.

Mild self-etch adhesives (pH≈2) dissolve fewer calcium phosphates. At the enamel level, the etching pattern is very weak and results in weak micromechanical bonding. At the dentin level, the surface is only partially demineralized to a depth of 1 μm, creating microporosities sufficient for micro-mechanical interlocking, with a substantial amount of hydroxyapatite crystals remaining undissolved and protecting the collagen fibrils and adhesive
interface from early hydrolytic reactions. These hydroxyapatite crystals also serve as receptors for additional chemical bonding.

Functional monomers with specific phosphate groups, such as phenyl-P (2-methacryloyloxyethyl phenyl hydrogen phosphate), and 10-MDP (10-methacryloxydecyl dihydrogen phosphate) or carboxylic groups such as 4-META (4-methacryloxyethyl trimellitic acid), are capable of chemical bonding with calcium of the residual hydroxyapatite crystals through primary ionic binding. \(^ {88,89}\) 10-MDP results in more effective and hydrolytically stable chemical bonds as opposed to other functional monomers such as 4-META and phenyl-P (2-methacryloyloxyethyl phenyl phosphoric acid). \(^ {88}\)

Intermediately strong adhesives have a pH value about 1.5 and cannot be classified as mild or strong self-etching adhesives. This results in better micromechanical interlocking than mild self-etching adhesives, at enamel and dentin levels. The resulting demineralized layer is 2.5 \(\mu\)m in thickness that has its surface totally demineralized whereas the base contains residual hydroxyapatite crystals that allow for chemical interactions, and thus the transition of the exposed collagen network to the underlying unaffected dentin is more gradual. \(^ {66}\)

The interaction between self-etch adhesives and collagen is better than that occurring between etch-and-rinse adhesives and collagen, which increases the chances for chemical bonding between residual hydroxyapatite crystals and monomeric groups to enhance bonding. \(^ {80}\)

The resultant two-fold micro-mechanical and chemical bonding mechanism is believed to be advantageous in terms of restoration durability. The micro-mechanical bonding component provides resistance to abrupt de-bonding stress while the chemical bonding provides resistance to hydrolytic breakdown. \(^ {67,90}\)
Two-step self-etch adhesive systems involve the use of hydrophilic self-etch primer followed by the application of a more hydrophobic adhesive resin. This results in a more hydrophobic interface and allows better bond durability.\textsuperscript{91} One-step self-etch adhesives are simpler and fast to use but they show lower bonding efficiency compared to two-step self-etch adhesives.\textsuperscript{92} This may be related to their inferior mechanical properties, lower degree of conversion and increased water sorption by osmosis from dentin.\textsuperscript{82,92}

3.2 Resin Cements

Clinically, resin cements are becoming very popular due to their ability to strongly bond to the tooth structure and the indirect restorations. They have high mechanical properties and the lowest solubility compared to the other available cements.\textsuperscript{64,93}

Resin cements were initially based on the chemistry of acrylic resins that improved with time due to further developments in composite resins and adhesive systems. There are two main categories of resin cements: those requiring the use of an adhesive system (adhesive-based resin cements), and those that don’t require the use of any adhesive system (self-adhesive resin cements).\textsuperscript{63,94}

3.2.1 Adhesive-based resin cements

These are composite resin cements that require the application of an adhesive system prior to the application of the cement. They can be classified according to the method used to activate polymerization: light-cured, self-cured and dual cured. They can also be classified according to the adhesive system used: total etch and self-etch resin cement systems.\textsuperscript{63,64}

Since the 1970s, adhesive-based resin cements have been available as a two-paste system. Once the adhesive system has been applied to tooth structure, the resin cement is mixed according to the manufacturer instructions and applied to the intaglio surface of the treated
indirect restoration.\textsuperscript{61,64,65} Their composition is usually a mixture of dimethacrylate oligomers, inorganic fillers and polymerization initiator. These components are adjusted to maintain low film thickness and appropriate working and setting time.\textsuperscript{61}

Self-cured adhesive-based resin cements are mainly indicated for cementation of metallic restorations, metal-ceramic restorations and posts.\textsuperscript{95} Their use in dentistry is very limited as they have several disadvantages such as their limited working time, their color instability as their aromatic amines accelerator oxidizes with time and changes the cement color to a more yellow shade, and their difficulty in mixing uniformly resulting in non uniform curing of the cement and thus lower mechanical properties.\textsuperscript{96}

Light-cured adhesive-based resin cements provide extended working time but their use is limited to cementation of laminate veneers or shallow inlays, where curing light can pass through the restoration and initiate polymerization of the cement.\textsuperscript{97,98}

Dual-cured adhesive-based resin cements can be used in situations where the restoration might block the curing light from reaching deeply.\textsuperscript{99} Usually supplied as two-paste systems, where one of the pastes contains the photo-initiator and the chemical activator (reducing amine), while the other paste contains the chemical initiator, which is usually benzoyl peroxide.\textsuperscript{61,100}

An interesting feature of these dual-cured cements is that polymerization is accelerated after the placement of the restoration when the surrounding environment is deprived from the ambient oxygen supply. This ambient oxygen feature provides extended working and setting times before placement of the restoration.\textsuperscript{101} Immediate light curing of the dual cure cements may negatively limit the self-cure mechanism which may adversely affect the mechanical properties of the cement, which is why it is recommended in these cements to allow time for the
self-curing mechanism to occur, followed by the light cure mechanism that augments the whole polymerization process to reach the best mechanical properties. The ideal time frame between mixing and the light-activation has not yet been determined, but some studies have shown that light-curing 5 to 10 minutes after mixing does not seem to interfere with final properties, at least for most of the cements evaluated.

Without the light curing step, the dual cure cements will act similar to exclusively self-cure cements and will require more time to cure, which might allow for the transudation of water from dentin to occur and adverse hydrolytic reactions to take place.

3.2.2 Self-adhesive Resin Cements

Self-adhesive resin cements were introduced to the dental market in 2002 with aims to provide an alternative to adhesive-based resin cements. These cements are composite resins that can adhere to tooth structure without the need for adhesive or etching process. They combine features of restorative composites, self-etching adhesives and dental cements. They were introduced to dentistry as a subgroup of resin cements and have gained popularity.

Rely X Unicem from 3M ESPE represents the first of this new class of materials. Now several self-adhesive resin cements are available in the dental market. They differ in terms of composition, working/setting time, number of shades available and the delivery system.

All the current self-adhesive resin cements consist of two pastes that require hand-mixing, auto-mixing or capsule trituration. Once the cement is mixed, it can be applied in a single clinical step.

The major benefit of these materials is their simplicity of application. According to the manufacturer’s information no post-operative sensitivity is expected as the smear layer is not removed. These cements are claimed to be moisture tolerant and some are capable of releasing
fluoride ions in a manner comparable to glass ionomer cement.\textsuperscript{63,104} Also, they offer good esthetics, adequate mechanical properties and dimensional stability similar to other categories of resin cements.\textsuperscript{63,104}

Available self-adhesive resin cements are dual-curing radiopaque materials that are indicated for adhesive cementation of any indirect restoration whether it is ceramic, composite or metal.\textsuperscript{103,105} Clinicians generally prefer adhesive-based light-curing materials as opposed to self-adhesive for luting veneers, due to the need for the longer working time offered by the light-curing procedure, which allows them to position and adjust several veneers simultaneously, prior to initiation of the cement polymerization.\textsuperscript{103,105}

Self-adhesive resin cements utilize monomers with functional acidic groups to demineralize the tooth structure. These monomers are mainly methacrylate monomers with either carboxylic acid groups, as with PMGDM (pyromellitic glycerol dimethacrylate) and 4-META, or phosphoric acid groups, as with 10-MDP (10-methacryloxydecyl dihydrogen phosphate), Phenyl-P (2-methacryloxyethyl phenyl hydrogen phosphate), Penta-P (dipentaerythritol pentaacrylate monophosphate) and BMP (Bis 2-methacryloxyethyl acid phosphate).\textsuperscript{103,106}

The concentration of these acidic monomers in self-adhesive resin cements is balanced to be low enough to avoid excessive hydrophilicity in the resulting polymer and high enough to have a proper degree of self-etching property.\textsuperscript{106} Once the self-adhesive resin cement is mixed, it shows high initial hydrophilicity, which facilitates their wetting and adaptation to the tooth structure. The acidic groups react with the calcium of the tooth structure and the metal oxides released from the ion-leachable fillers.\textsuperscript{106} The material becomes more hydrophobic as the acidic groups are consumed throughout the reaction. The adhesion obtained is due to micromechanical
interlocking with tooth structure and chemical bonding between the acidic monomers and hydroxyapatite. 106

3.2.3 Self-adhesive resin cements versus adhesive-based resin cements

Several studies have been performed to determine the bonding efficiency of self-adhesive resin cements and to determine if they are a possible replacement for current conventional adhesive-based resin cement systems. 63 The current adhesive-based resin cement systems can be classified into two categories: those utilizing etch-and-rinse adhesives and those utilizing self-etch adhesives.

In some bond strength studies that were conducted using shear, tensile and microtensile bond strength tests, self-adhesive resin cements performed comparably to conventional adhesive-based resin cement systems when bonding to coronal dentin. However, in other studies, self-adhesive resin cements show significantly lower microtensile bond strength compared to adhesive-based resin cement systems. 108-113

Another study evaluated the tensile bond strength of self-adhesive resin cements to indirect composites. The indirect composite resin restorations luted with the self-adhesive resin cements showed better results in comparison with those luted with etch-and-rinse and self-etch adhesive-based resin cements. 114 In another study, the self-etch adhesive resin cement systems and self-adhesive resin cements showed higher bond strength results compared to conventional etch-and-rinse adhesive-based resin cements, and the luting agents had a stronger influence on bond strength between restorative materials and dentin than the type of the restorative material. 115 It was also shown that when correct application procedures are followed, the etch-and-rinse and self-etch adhesive-based resin cements as well as self-adhesive cements are equally effective in bonding to enamel and dentin 112. Other studies have shown that self-adhesive resin
cements are satisfactory and comparable to other multi-step adhesive-based resin cements regarding bonding to dentin, while adhesion to enamel appears to be weak. Assessment of long-term clinical performance of self-adhesive resin cements is needed for better evaluation of these materials.\textsuperscript{105}

The effect of cementation of feldspathic ceramic CAD/CAM molar crowns with etch-and-rinse adhesive-based resin cement or self-adhesive resin cement on their fracture resistance has also been investigated. Crowns cemented with self-adhesive resin cement showed higher fracture resistance when compared to those cemented with multistep etch-and-rinse adhesive-based resin cement.\textsuperscript{59} However in another study, the fracture resistance of ceramic crowns cemented to dentin using self-adhesive resin cement was not different from those cemented with conventional etch-and-rinse adhesive-based resin cement.\textsuperscript{116}

Comparing the nanoleakage of CAD/CAM ceramic blocks bonded to dentin with self-adhesive and those bonded using etch-and-rinse adhesive-based resin cement, one self-adhesive resin cement (Rely X Unicem) demonstrated similar sealing ability when compared to conventional etch-and-rinse adhesive-based resin cement system with and without thermocycling (500 cycles)\textsuperscript{117}.

Self-adhesive resin cement also shows better marginal sealing compared to self-etching adhesive-based resin cements, when used to cement feldspathic ceramic CAD/CAM molar partial crowns.\textsuperscript{118} However, in another study, self-etching adhesive-based resin cements resulted in significantly lower microleakage scores (better marginal sealing) compared to self-adhesive resin cements, irrespective of crown material being composite or feldspathic porcelain.\textsuperscript{119}
3.3 Surface treatment and adhesion

The clinical success of indirect esthetic restorations depends on the quality of bonding achieved with the underlying tooth structure. \(^{120,121}\) Surface treatment of the ceramic or indirect composite resin restorations has an impact on the bonding performance to the tooth structure. Surface treatment depends on the composition of the restorative material and can be classified into mechanical or chemical. \(^{122,123}\)

3.3.1 Mechanical surface treatment

Resin cements are characterized by their low solubility and good adhesion to the tooth structure. \(^{124,125}\) These cements acts as the main link between the indirect esthetic restoration and the tooth structure. To achieve good bonding to the resin cement, micromechanical means of retention should be created on the intaglio surface of indirect restorations. \(^{120,122,126,127}\)

Hydrofluoric acid etching and grit-etching are well-known procedures for mechanical surface treatment. \(^{128}\)

Hydrofluoric acid etching is a procedure that creates micro-roughness on the intaglio surface of ceramic restorations and improves cement bond. \(^{128,129}\) The chemical etching of feldspathic ceramics with hydrofluoric acid results in honeycomb-like topography of the ceramic surface, which is ideal for micromechanical adhesion. \(^{129,130}\) This surface topography is a result of the chemical reaction between hydrofluoric acid and the silica phase of feldspathic ceramics forming a hexafluorosilicate salt that is easily removed by water rinsing. \(^{130-132}\)

A study performed on the effect of etching time on the bonding procedures evaluating five different etching times (5, 30, 60, 120 and 180 s) showed that resin cement did not bond to unetched ceramic while bonding was successfully achieved with the etched ceramic, and the 120 s etch resulted in the highest bond strength. \(^{131}\) This was further confirmed by Bona et al.
who stated that the bond strength of resin cements increases with increasing ceramic surface roughness induced by acid etching.\textsuperscript{133}

According to Bona et al. lithium disilicate ceramics have shown higher flexure resistance and bond strength values when etched with 9.5\% hydrofluoric acid as opposed to 4\% acidulated phosphate fluoride.\textsuperscript{134} Borges and colleagues reported that etching dental ceramics with 9.5\% hydrofluoric acid for 20 seconds creates an adhesion-favorable surface.\textsuperscript{122}

However, hydrofluoric acid etching results in shallow surface roughness that is unfavorable for micromechanical interlocking in alumina-reinforced ceramic restorations like In-Ceram alumina system, and zirconia-reinforced restorations like In-Ceram zirconia system (Vita Zahnfabrik, Seefeld, Germany) due to their low silica content.\textsuperscript{120,122,135,136} Sen et al. reported that hydrofluoric acid etching was not enough to achieve strong bonding between resin cements and zirconia ceramics compared to grit-etching which creates a micromechanical adhesion-favorable surface and can be considered a good alternative to hydrofluoric acid etching.\textsuperscript{137} This study also defined several important parameters to be followed to maximize the results of surface grit-etching such as particle type, size, shape, incidence angle, and wet versus dry particles.

Grit-etching alone provides insufficient bond strengths for felspathic ceramics and may induce chipping and loss of ceramic material and is therefore not recommended for treatment of silica-based all-ceramic restorations.\textsuperscript{138-140} Kato et al. compared grit-etching to different acid-etching agents on feldspathic porcelain and found that hydrofluoric acid and sulphuric acid provided the highest bond strengths.\textsuperscript{130}

Lithium disilicate ceramics are capable of achieving good bonding with resin cements when sandblasted or acid-etched by hydrofluoric acid. It has been shown that lithium disilicate
ceramics attained similarly high bond strength values with aluminum oxide grit-etching for 10 seconds and also with hydrofluoric acid etching for 20 seconds.\textsuperscript{128}

Etching indirect composite resin restorations with hydrofluoric acid results in microstructural alteration of the composite due to the dissolution of the inorganic particles present in composite resins, and thus the resin organic matrix becomes the dominant component of the surface which makes the restoration-cement adhesive interface less resistant to debonding.\textsuperscript{123} However, grit-etching indirect composite restorations with aluminum oxide particles is the best alternative to increase the restoration surface energy.\textsuperscript{10,123} Grit-etching promotes non-selective degradation of the composite and results in better adhesion to the resin cement.\textsuperscript{10,123,141,142}

### 3.3.2 Chemical treatment

Silane coupling can be used to enhance the bond between the indirect restoration and the resin cement. Although it cannot substitute the mechanical surface treatment, it is used as an additional step to strengthen the bonding by chemical means.\textsuperscript{128}

A silane molecule has ethoxy groups to bond to the inorganic particles of ceramics and an organofunctional group, typically a methacrylate, to react with the organic matrix of composite resins forming covalent bonds.\textsuperscript{143,144} The chemical structure of the silane is $R' - Si(OR)3$, where $R'$ is the organofunctional group and $(OR)$ is the ethoxy group where $R$ refers to the alkyl group. The ethoxy group is hydrolyzed to result in a silanol ($SiOH$) that reacts with the silicon inorganic particles in the ceramic creating a siloxane covalent bond ($Si-O-Si$).\textsuperscript{145-148}

A silane-coupling agent is applied to the ceramic surface to chemically bond the inorganic phase in the ceramic and the organic phase of the composite cement.\textsuperscript{143,149} Bona et al.
have demonstrated that silane application improves the bonding to ceramics reinforced with feldspar, leucite, or lithium disilicate, but can’t be a substitute for mechanical surface treatment.\textsuperscript{134}

Inorganic filler particles that exist on the surface of indirect composite resin allow the development of chemical adhesion between these filler particles and the organic matrix of the resin cement resulting in higher bond strength values.\textsuperscript{123} Soares et al. have reported that grit-etching of indirect composite resins together with the use of silane, results in high bond strength values as opposed to grit-etching without silane application.\textsuperscript{136} All indirect composite resins present similar composition and their surface treatment tend to be the same.\textsuperscript{123}

4. Adhesion testing

Variations and advances in adhesive materials have made in-vitro adhesion testing of great importance. The most common laboratory tests to evaluate adhesion of different adhesives to the tooth substrate are tensile and shear bond strength tests.\textsuperscript{150} However, studies have shown that the variability in specimen geometry, loading conditions and material properties have significant effect on the tensile and shear testing results.\textsuperscript{151} Also, the crown pull-off test has been used as a more clinically relevant in-vitro adhesion test.\textsuperscript{15}

4.1 Microtensile test

Sano et al. developed the micro-tensile bond-strength (\(\mu\)TBS) test in 1994 to overcome some of the limitation of macro-tests.\textsuperscript{152} The tested bonding area is about \(1\text{mm}^2\) or less, which gave the \(\mu\)TBS test several advantages including: better economic use of teeth since multiple specimens can be obtained from one tooth, better stress distribution at the bonding interface with more adhesive failures thereby avoiding cohesive failure in tooth substrate or restoration,
and the ability to compare a variety of substrates and areas within the same tooth (e.g. peripheral versus central dentin). \textsuperscript{152-155}

However, several protocols are being used in specimen preparation for microtensile testing, and are classified into trimming and non-trimming protocols. The trimming protocol is more technique-sensitive than the non-trimming one. Each protocol has its own advantages and drawbacks. \textsuperscript{156,157}

Non-trimmed microspecimens are easier and faster in preparation, where the bonded specimens are sectioned into rectangular bars 0.5–1.5 mm in thickness. Microspecimens trimmed to a dumbbell or hourglass shape show better stress concentration at the adhesive interface. Moreover, trimming has to be carefully performed, otherwise interfacial defects may occur and act as stress concentrators facilitating crack-propagation when the specimen is loaded in tension, causing premature failure at the interface with a lower recorded bond strength.\textsuperscript{158}

Trimming the interfaces free-hand using a dental handpiece is a very difficult and technique-sensitive procedure that is dependent on the operator skill.\textsuperscript{16,156,157} Semi-automatic trimming of micro-specimens can be achieved using the so-called microspecimens former (University of Iowa, Iowa City, IA, USA) to trim rectangular specimens in a standardized well-controlled manner, into dumbbell shaped specimens or specimens with a circular cross-section.\textsuperscript{159,160}

Also, more factors need to be standardized within the test settings such as attachment of specimen to the jig, alignment of specimens and loading speed.\textsuperscript{16} Otherwise, these factors will influence the final outcome of the testing procedure by changing the direction and the severity of the loading applied.\textsuperscript{16,161,162}
The number of recorded pre-testing failures is a major point of debate in the current literature regarding the $\mu$TBS testing.\textsuperscript{163} Three main approaches have been applied to manage the pre-testing failures: one is to ignore pre-testing failures from statistical analysis, which results in overestimated bond strength results. Another assigns a zero MPa bond strength value to every pre-testing failure record. However, this is non-realistic as there is a certain initial bond strength, which in turn penalizes the tested product severely. The third approach is a modification of the previous one by assigning a certain pre-determined value to every pre-testing failure. This value can be the lowest $\mu$TBS testing value measured within the respective group.\textsuperscript{164-166} These approaches have obvious effects on the mean $\mu$TBS value and the subsequent statistical analysis.

Certainly, micro-specimen processing should be performed as carefully as possible and special measures should be utilized to avoid pre-testing failures. The measures include the use of materials such as alginate or gypsum to fill up the spaces between the slabs, which result in better support for the slabs during the second cutting stage.\textsuperscript{20} Several studies have shown that $\mu$TBS testing appears to be able to discriminate between adhesives regarding their bonding performance in a better way than the traditional shear and tensile bond strength tests. This is likely why up to 60% of current scientific papers reporting on bond strengths have employed the $\mu$TBS approach.\textsuperscript{20}

4.2 Microshear test

In 2002, the microshear bond strength test ($\mu$SBS) was introduced as an alternative to the microtensile test.\textsuperscript{167-169} In the microshear test, load is applied to the adhesive interface using a blade attached to a universal testing machine to induce shearing forces.\textsuperscript{169,170} Shimida et al. modified the microshear test settings by using a looped orthodontic wire instead of using a
blade. In a study by Foong et al. on bond strength to enamel, using the orthodontic wire to apply shearing forces was easier and more reliable in comparison to using a blade.

In the microshear test, it is possible to test several specimens per tooth and the bonding area can be controlled using microbore (tygon) tubes. However, when testing microshear bond strength of composite resins to tooth structure, considerable bending and non-uniform stress distribution within specimens can occur as a result of the relatively thick adhesive layer compared to the composite resin cylinder 0.7-1.0 mm in diameter. This non-uniform stress distribution is more pronounced than conventional shear test. Furthermore, it is impossible to confine the adhesive resin to the bonding area. As a consequence of these shortcomings, the µSBS test has been used in only 7% of the recent bond strength studies.

In a recent study comparing microshear and microtensile tests, the microshear bond strength values were about 1/3 of the micro-tensile values, and there was no difference in the mode of failures. Another study has shown that microshear test is more accurate in evaluation of the bond strength compared to the microtensile test since the former was associated with more adhesive failures.

4.3 Retention Testing

Retention tests were developed in the 1970's to simulate the clinical condition in a better way than bond strength tests. In retention tests, whole crowns are made and cemented to the underlying tooth substrate and then subjected to pull-away forces to detach from the underlying substrate.

The main advantage of the retention tests over the bond strength tests is that it takes into account the complex configuration of the substrate to which the crown is cemented, not like the bond strength tests that consider the substrate as a one-surface flat area. Studies that compare
both the retention of the crowns and the bond strength results of dental cements are lacking in the dental literature.

The configuration factor (C-factor) is the ratio between bonded surfaces and non-bonded surfaces. The higher the value of the configuration factor, the greater are the stresses induced at the bonded interfaces as a result of polymerization shrinkage. \(^{23}\) This configuration factor is much higher in crowns cemented to their underlying tooth abutments as in the retention tests assembly, than of composite or ceramic cylinders cemented onto flat substrate surfaces as in conventional bond strength tests assembly. \(^{15}\) Several studies have shown the significant effect of the configuration factor on adhesion, which might explain the low values obtained from retention testing in the range of 1–10 MPa compared to those obtained from bond strength testing in the range of 20–40 MPa. \(^{24}\)

Since the development of retention tests, most of the tested crowns were metal crowns modified with a ring or hook through which the crown could be pulled away from the underlying tooth substrate. \(^{175-179}\) Recently with the developments in the CAD/CAM technology and with further developments in ceramic industry, all-ceramic crowns became popular and several trials were undertaken in attempts to pull them away from the underlying substrate. \(^{15,180-182}\) However, pulling away the ceramic crowns from the underlying substrate without causing fracture of the crown remained problematic.

Some studies have increased the thickness of the occlusal portion and formed a hole in the occlusal surface through which a hook could be attached to pull-off the crown. However, failure rates up to 65 % of the specimens were recorded due to specimens fracturing before being detached from the underlying substrate. \(^{183}\)
Other studies have designed CEREC crowns with the pull-off loop as an integral part of the crown structure. However, failures related to fractures of the loop during the pull-off were reported. Other studies have attached macro-retention features to the crown design, by modifying the shape of the crown to have bars projecting from it, either from the occlusal surface or from the sides of the crowns, or by making the crown conical in shape. Failures were recorded in those crowns with bars on the occlusal surface, however, no failure was mentioned with the conical shaped crowns or with crowns having bars projecting from their sides.

Another main issue is the effect of the surface area on the retention test results. Some studies did not measure the surface area and just distributed the teeth to groups based on their type, so that the groups had either molars or premolars. Another study measured the size of teeth and divided them into three groups: small, medium, and large. The teeth were assigned into the equal testing groups, so that every group had the same size distribution. These studies only measured the force required to dislodge the crowns from their underlying substrate.

Other studies tried to measure the surface area using a correlation method, where the weight of a tin foil wrapped around the prepared surfaces was correlated to the weight of a standardized tin foil of 1cm² cross-sectional area. Other studies attempted to calculate surface area based on the approximate size of the occlusal surface and compared it with standardized circles of known surface area and perimeter. All these studies gave an approximate estimate of the surface area. Retention tests are time-consuming and require large number of extracted teeth. Several factors can affect the outcome of these tests, such as the surface area of the preparation, the method used to pull the crowns, standardized preparations and the aging method.
5. Microleakage

Microleakage is the movement of fluids and bacteria at the interface between the restoration and tooth tissue. Microleakage leads to staining of the margins of the restoration, hydrolytic breakdown of the adhesive interfaces, and recurrent caries at the tooth-restoration interface, which if left untreated can result in pulpal pathology.

Polymerization shrinkage is considered one of the main factors that can result in microgaps at the tooth restoration interface. These microgaps facilitate fluids and bacterial ingress at the tooth-restoration interface. Also, clinically, if the adhesive interface or the adhesion process is compromised, this results in microleakage, which in turn could result in further adhesive failure. Masticatory chewing cycles and thermal fluctuations in the oral cavity can induce crack propagation at the adhesive interface, which in turn can cause microleakage.

Several methods have been used to evaluate microleakage either in-vivo or in-vitro. In-vitro microleakage testing is commonly accepted to evaluate the sealability at the adhesive interface and can help in understanding the mechanisms of microleakage and developing measures to minimize or even eliminate microleakage. Most of the in-vitro microleakage tests use tracers like dyes or chemical tracers to evaluate the extent of microleakage. Some studies use radioactive isotopes or bacteria as tracers. Other studies use air pressure, neutron activation analysis, electrical conductivity, or scanning electron microscopy to evaluate microleakage.

In-vivo microleakage tests typically involve the use of human teeth that have previously been selected for orthodontic extraction. These teeth are restored and later extracted.
after a certain period of time, and microleakage is tested using dye tracers. The teeth are sectioned and examined under stereomicroscope to assess the degree of dye penetration. 195, 196
Chapter 2 Rationale and objectives
1. **Statement of the problem**

The use of CAD/CAM restorations is steadily increasing. However, proper function and longevity of these restorations depends on their good adhesion to the underlying substrate.\(^3\)

Self-adhesive resin cements simplify the clinical procedures and are less technique sensitive compared to adhesive-based resin cements. The use of self-adhesive resin cement with lithium disilicate CAD/CAM material has not as yet been explored. Also, little information exists on the use of self-adhesive resin cements with composite resin CAD/CAM material.

To evaluate the interaction between self-adhesive cements and CAD/CAM materials, microtensile and microshear bond strength tests offer several advantages compared to the conventional shear and tensile bond strength tests, such as more uniform stress distribution during loading and the capacity to obtain multiple specimens from one tooth. However, the cutting procedures induce stresses that lead to several pre-test failures and affect the measured bond strength results.\(^{17-20}\) The bonding of the specimens after their cutting to avoid these stresses and then performing the tests have not as yet been explored. Also, little information exists regarding comparing microtensile and microshear bond strength tests.

Retention testing is another alternative method to evaluate bonding performance of resin cements. It takes into consideration the complex configuration of the abutment tooth. Retention of lithium disilicate glass ceramic and composite resin crowns has not as yet been explored. Also, the retention test methodology needs modification in order to be applicable to ceramic and composite crowns and the abutment surface area needs to be measured to express the results of the retention tests in terms of stress values, which is more accurate than just evaluating the retention force.
Microleakage testing indicates the sealability of the adhesive interface between the restoration and the underlying tooth. Microleakage is an important factor that can affect adhesion as well as it can be affected by adhesion. Microleakage of lithium disilicate glass ceramic and composite resin crowns has not as yet been investigated.

Therefore, this study was conducted to evaluate the adhesion and microleakage of lithium disilicate ceramic CAD/CAM material and composite CAD/CAM material to the underlying substrate using self-adhesive resin cements. Microtensile and microshear bond strength tests were applied to evaluate the bond strengths. Retention testing was performed to measure the retention strength. Dye penetration was used to evaluate microleakage.

2. Objectives

- To evaluate the effect of resin cements, crown materials and bonding substrates on microtensile bond strength between CAD/CAM materials and different substrates using two self-adhesive resin cements and two adhesive-based resin cements, employing a modified technique for specimen preparation.
- To evaluate the effect of resin cements, crown materials and bonding substrates on microshear bond strength between CAD/CAM materials and different substrates using two self-adhesive resin cements and two adhesive-based resin cements, employing a modified technique for specimen preparation.
- To evaluate the effect of resin cements, crown materials and bonding substrates on retention strength between CAD/CAM materials and different substrates using a self-adhesive resin cement and an adhesive-based resin cement, employing a modified technique for specimen preparation.
- To compare a self-adhesive resin cement and an adhesive-based resin cement with respect to microleakage between CAD/CAM materials and natural tooth substrate.
Chapter 3 Microtensile bond strength of self-adhesive resin cements to non-metallic crown CAD/CAM materials, composite and dentin
1. Abstract

Objectives: To evaluate microtensile bond strength (μTBS) between two CAD/CAM crown materials and dentin or composite-resin substrate using two self-adhesive, an etch-and-rinse and a self-etch resin system; and to compare the effect of two surface treatments of the ceramic crown material on μTBS. Methods: Lithium disilicate ceramic (IPS e.max CAD, Ivoclar-Vivadent/EMX), composite-resin (Paradigm MZ-100, 3M-ESPE/PZ) blocks, and custom-made composite-resin (Z100 restorative, 3M-ESPE) blocks were cross-sectioned into microbars approximately 1×1×5 mm³. Similar dentin specimens were obtained from the coronal portion of freshly extracted molars. EMX microbars were either sandblasted (50μm Al₂O₃) or etched with HF acid and silanated. PZ microbars were only sandblasted and silanated. EMX and PZ specimens were then bonded to either dentin or composite resin microbars using two self-adhesive cements (RelyX Unicem, 3M-ESPE; Clearfil SA, Kuraray) and two adhesive-based cements (Multilink Automix, Ivoclar-Vivadent; RelyX-ARC, 3M-ESPE) using a custom-made device (n=10). A static load of 146 g was maintained until initial setting of the cement. Specimens were stored in water (37°C) for 24 h, thermocycled (500 cycles, 5-55°C), then subjected to μTBS test. Means and standard deviations were calculated and data statistically analyzed by factorial ANOVA test (α=0.05). Results: ANOVA revealed significant differences among the groups (P<0.05). For composite substrate, highest mean μTBS (32.7±2 MPa) was obtained when RelyX-ARC was used with PZ, while lowest mean μTBS (11.6±1.3 MPa) when Rely-X Unicem was used with EMX. For dentin, highest mean μTBS (28.8±2.3 MPa) was obtained when RelyX-ARC was used with PZ, while lowest mean μTBS (8.61±1.3 MPa) was obtained when Clearfil-SA was used with EMX. Conclusions: Overall adhesive-based cements resulted in higher mean μTBS irrespective of the substrate material. Whereas, self-adhesive
resin cements resulted in lower mean μTBS irrespective of the substrate. For EMX, HF-treatment resulted in higher mean μTBS as compared to grit-etched surface.

2. Introduction

Computer-aided-design/computer-aided-manufacture (CAD/CAM) systems are becoming more popular since this technique is less time-consuming for the dentist and the patient by providing esthetic restorations in a single appointment without the need for dental laboratory support. These restorations are made of high quality ceramic or composite resin materials. However, adequate adhesion between these restorations and tooth substrate is a prerequisite for proper function and longevity of these restorations.

Tooth structure needs special treatment with certain adhesive systems in order to achieve adequate micromechanical bonding with the overlying esthetic restoration. However, self-adhesive resin cements simplify the clinical procedures and can be applied directly to the tooth structure without the application of any adhesive system.

Esthetic restorations also require special treatment to achieve adequate bonding with the underlying substrate. Ceramic CAD/CAM restorations are roughened by acid etching and/or grit-etching to achieve micro-mechanical interlocking. Some studies showed that hydrofluoric acid etching and grit-etching of ceramic restorations result in similar bonding performance between the restorations and resin cements, while other studies have shown that hydrofluoric acid etching is more advantageous than grit-etching with regard to the bonding performance. On the other hand, composite CAD/CAM restorations can be roughened by grit-etching only for micro-mechanical interlocking, since hydrofluoric acid etching of composite resins causes total dissolution of the inorganic fillers and partial degradation of the resin matrix which negatively affects the adhesion to these restorations. After mechanical surface treatment, it
is better to establish a chemical link between the restoration and the resin cement. Silane coupling agents are used to coat the treated ceramic and composite restorations in order to achieve the desired chemical bonding with the resin cement.\textsuperscript{4,6-8} Whereas, hydrofluoric acid etching of composite restorations can damage the resin matrix and decrease both the mechanical properties and the bonding performance of the restoration.\textsuperscript{9-12}

Several factors influence the bonding of CAD/CAM restoration to the underlying substrate, such as the type of the resin cement, the type of the restoration and even the type of underlying substrate.\textsuperscript{4,13,14} These factors are commonly investigated using in-vitro adhesion tests such as tensile, shear, microtensile and microshear bond strength tests.\textsuperscript{15} The microtensile bond strength test offers several advantages compared to the conventional shear and tensile bond strength tests since multiple specimens can be cut from one large sample and the stress distribution produced during loading is more uniform.\textsuperscript{16,19} This leads to higher bond strengths with fewer cohesive fractures.\textsuperscript{17-19}

In the conventional microtensile testing technique: the dentin surface is exposed within the tooth (bonding substrate) and the crown material is cemented to the dentin substrate, which is followed by cutting the whole specimens in two perpendicular directions to obtain microbars.\textsuperscript{16,172} However, large amount of specimens have shown pre-test failure and even those microbars that survived the cutting procedures have been exposed to the cutting stresses that may make the results doubtful.\textsuperscript{199} Also, the pre-test failures create problems in statistical analysis, where three approaches exist to deal with these pre-test failures, either by excluding specimens that show pre-test failure so that they will not contribute to the result, or by assigning them half of the lowest value or by assigning them zero value.\textsuperscript{16,199} Every statistical approach results in a different outcome even within the same study.\textsuperscript{16,199}
Thermocycling is considered to be a clinically relevant aging technique. In our study, thermocycling was used to stress the adhesive interface by temperature fluctuations for 500 cycles between 5 °C and 55 °C, causing volumetric expansions and contractions in aqueous conditions. The stresses induced during these cycles are due to differences in the coefficient of thermal expansion and contraction in the materials constituting the adhesive interface. Versluis et al. (1996) stated that temperature fluctuations could cause fatigue of the adhesive joint.

Also, Hashimoto et al., 2000 mentioned that a hot water bath (55 °C) may accelerate the hydrolytic reactions at the adhesive interface facilitating leaching out of residual oligomers and collagen breakdown products.

Our study aimed to modify the microtensile testing methodology to avoid the pre-test failures and to investigate the bond strength of self-adhesive resin cements versus adhesive-based resin cements when they are used to bond lithium disilicate glass ceramic material and composite resin material to the underlying dentin and composite resin substrates. Also, it was our aim to compare the effect of two surface treatments on lithium disilicate glass ceramic.
3. Materials and Methods

3.1 Materials

All the materials used in the study and their composition (as reported by the manufacturers) are listed in Table 1.

3.2 Methods:

3.2.1 Pilot study:

The current study was preceded by an external pilot study to detect any problems with the proposed methods of testing, where 2 samples per group were used in the pilot study. Following the completion of the pilot study, 240 specimens were used in the main study.

3.2.2 Main study

Twenty-four groups were formed (n=10)(Figure 1). The number of specimens per group was calculated using web-based software® for sample size analysis at α=0.05 and 80% power and effect size equal to 0.32 which yields a sample size of 7 samples per group. We increased sample size to 10 per group to enhance power. The effect size (f) was calculated by using the following formula, developed by Cohen (1999):

\[ f = d \times \frac{1}{2} \sqrt{\frac{k+1}{3(k-1)}} \]

Where \( k \) is the number of groups and \( d \) is the difference in means (\( x_1 - x_2 \)) divided by common standard deviation (\( \sigma \)). Based on values obtained from the pilot study, \( x_1 = 18.23 \), \( x_2 = 15.76 \), \( \sigma = 2.32 \), the \( d \) value was calculated (\( d = 1.064 \)), and \( f \) was calculated (0.32).

*Power Analysis For ANOVA Designs [http://www.math.yorku.ca/SCS/Online/power/]
Table 1: Materials and their composition (as reported by manufacturers)

<table>
<thead>
<tr>
<th>Material</th>
<th>Classification</th>
<th>Brand / Manufacturer</th>
<th>Composition</th>
<th>Lot #</th>
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<tbody>
<tr>
<td>CAD/CAM Materials</td>
<td>Lithium disilicate</td>
<td>IPS e.max CAD/ Ivoclar Vivadent, Liechtenstein</td>
<td>SiO₂ (57 – 80 wt.%), Li₂O (11 – 19 wt.%), K₂O (0 – 13 wt.%), P₂O₅ (0 – 11 wt.%), ZrO₂ (0 – 8 wt.%), ZnO (0 – 8 wt.%), Other and coloring oxides (0-12 wt.%).</td>
<td>P63946</td>
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<tr>
<td></td>
<td>ceramic</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>Indirect</td>
<td>Composite resin</td>
<td>Paradigm™ MZ100/ 3M ESPE, St Paul, MN, USA</td>
<td>85 wt.% ultrafine zirconia-silica ceramic particles that reinforce a highly cross-linked polymeric matrix. The polymer matrix consists of BisGMA and TEGDMA, and employs a ternary initiator system.</td>
<td>N250997</td>
</tr>
<tr>
<td>Resin Cements</td>
<td>Self-adhesive cement</td>
<td>RelyX™ Unicem/ 3M ESPE, St Paul, MN, USA</td>
<td>It consists of two pastes: Base paste: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, and stabilizers. Catalyst paste: methacrylate monomers, alkaline (basic) fillers, silanated fillers, initiator components, stabilizers, and pigments. (Filler=50% vol., 72 wt.%; avg. &lt; 12.5 μm)</td>
<td>427554</td>
</tr>
<tr>
<td></td>
<td></td>
<td>RelyX Ceramic primer / 3M ESPE, St Paul, MN, USA</td>
<td>Stable solution of a prehydrolyzed silane-coupling agent, alcohol and water.</td>
<td>N234072</td>
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<tr>
<td>Material</td>
<td>Classification</td>
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</tbody>
</table>
| Resin Cements             | Self-adhesive resin cement system     | Clearfil SA/ Kuraray Medical Inc., Sakazu, Kurashiki, Okayama, Japan | It consists of two pastes:  
Paste A: 10-MDP, Bis-GMA, TEGDMA, Hydrophobic aromatic dimethacrylate, dl-Camphorquinone, BPO Initiator, Silanated barium glass filler and Silanated colloidal silica.  
Paste B: Bis-GMA, Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic dimethacrylate, Accelerators, Pigments, and Surface treated sodium fluoride, Silanated barium glass filler and Silanated colloidal silica.  
(Filler = 45 % vol., 66 wt. %; avg. 2.5 μm)                                                                 | 011AAA |
<p>| Silane coupling agent     |                                       | Clearfil Ceramic primer/ Kuraray Medical Inc., Sakazu, Kurashiki, Okayama, Japan | Mixture of 3-trimethoxysilylpropyl methacrylate (&lt;5 wt.%), ethanol (&gt;80 wt. %) and 10-Methacryloyloxydecyl dihydrogen phosphate                                                                                      | 00019D |</p>
<table>
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<th>Material</th>
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<th>Brand / Manufacturer</th>
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</thead>
<tbody>
<tr>
<td>Resin Cements</td>
<td>Self-etching bonding agent</td>
<td>Multilink Primer A/B /Ivoclar Vivadent, Liechtenstein</td>
<td>Two Bottle self-etching adhesive. Multilink Primer A: water (85.7 wt.%) and initiators (14.3 wt.%). Multilink Primer B: Phosphonic acid acrylate (48.1 wt.%), HEMA (48.1 wt.%), Methacrylate mod. polyacrylic acid (3.8 wt.%) and Stabilizers (&lt;0.02 wt.%).</td>
<td>Multilink Primer A: P63825 Multilink Primer B: P65434</td>
</tr>
<tr>
<td></td>
<td>Adhesive-based resin cement</td>
<td>Multilink Automix/ Ivoclar Vivadent, Liechtenstein</td>
<td>It consists of two pastes: Base paste: Dimethacrylate and HEMA (30.5 wt.%), Barium glass filler and Silica filler (45.5 wt.%), Ytterbiumtrifluoride (23 wt.%), Catalysts and Stabilizers (1 wt.%), Pigments (&lt;0.01 wt.%). Catalyst paste: Dimethacrylate and HEMA (30.2 wt.%), Barium glass filler and Silica filler (45.5 wt.%), Ytterbiumtrifluoride (23 wt.%), Catalysts and Stabilizers (1.3 wt.%). (Filler=45.5 wt.%; avg. = 0.9 μm)</td>
<td>Multilink Cement: P67924</td>
</tr>
<tr>
<td></td>
<td>Silane coupling agent</td>
<td>Monobond Plus primer /Ivoclar Vivadent, Liechtenstein</td>
<td>Solution of adhesive monomers (4 wt.%) and ethanol (96 wt.%), the adhesive monomers combine three different functional groups – silane methacrylate, phosphoric acid methacrylate and sulfide methacrylate.</td>
<td>Primer: P59584</td>
</tr>
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<td>Classification</td>
<td>Brand / Manufacturer</td>
<td>Composition</td>
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<tr>
<td>Resin Cement</td>
<td>Acid etch</td>
<td>Scotchbond™ Phosphoric Etchant / 3M ESPE, St Paul, MN, USA</td>
<td>The etchant is 35% phosphoric acid by weight. It has a pH of approximately 0.6</td>
<td>N252067</td>
</tr>
<tr>
<td></td>
<td>Bonding agent</td>
<td>Adper™ Single Bond Plus / 3M ESPE, St Paul, MN, USA</td>
<td>One-bottle adhesive containing ethanol, 2-HEMA, BisGMA, other dimethacrylate resins, methacrylate-modified polycarboxylic acid copolymer, silica nanofiller, a small amount of water and a photoinitiator system.</td>
<td>N241285</td>
</tr>
<tr>
<td>Adhesive-based resin cement</td>
<td>Adhesive-based resin cement</td>
<td>RelyX™ ARC / 3M ESPE, St Paul, MN, USA</td>
<td>It is composed of BisGMA and TEGDMA polymer, it consists of two pastes: Paste A: zirconia/silica filler (68 wt.%), pigments, amine and photo initiator system. Paste B: zirconia/silica filler (67 wt.%), BPO activator (Filler=67.5 wt.%; avg. = 1.5 μm)</td>
<td>N247407</td>
</tr>
<tr>
<td></td>
<td>Silane coupling agent</td>
<td>RelyX Ceramic primer / 3M ESPE, St Paul, MN, USA</td>
<td>Stable solution of a prehydrolyzed silane-coupling agent, alcohol and water.</td>
<td>N234072</td>
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<tr>
<td>Material</td>
<td>Classification</td>
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<tr>
<td>Mechanical surface treatment material</td>
<td>Grit-etching</td>
<td>Aluminum Oxide-50 Micron/ Danville Engineering Inc., California</td>
<td>50 microns aluminum oxide powder.</td>
<td>25671</td>
</tr>
<tr>
<td></td>
<td>Acid etching</td>
<td>IPS Ceramic Etching Gel /Ivoclar Vivadent, Liechtenstein</td>
<td>The etchant is &lt;5% hydrofluoric acid. It has a pH of approximately 2</td>
<td>P72319</td>
</tr>
<tr>
<td>Composite filling material</td>
<td>Composite resin</td>
<td>Z100™ Restorative /3M ESPE, St Paul, MN, USA</td>
<td>The matrix of this composite consists of BisGMA and TEGDMA polymer. The filler is zirconia/silica. The inorganic filler loading is 66% by volume with a particle size range of 3.5 to 0.01 micron.</td>
<td>N225470</td>
</tr>
</tbody>
</table>

Abbreviations: BIS-GMA (bisphenol a diglycidyl ether dimethacrylate), BPO (benzoyl peroxide), HEMA (hydroxyethyl methacrylate), MDP (methacryloyloxydecyl dihydrogen phosphate), and TEGDMA (tetraethyleneglycol dimethacrylate).
Figure 1: Study groups, the total number of specimens are 240 specimens (10 per group x 24 group)
3.2.2.1 Specimen Preparation:

- Preparing IPS e.max CAD and Paradigm™ MZ100 specimens:

  IPS e.max CAD Blocks (Ivoclar Vivadent, Liechtenstein) and Paradigm™ MZ100 Blocks (Ivoclar Vivadent, Liechtenstein)(Figure 2) were prepared according to a new technique presented in Figure 3. The blocks were cut perpendicular to the long axis with a low speed diamond saw (Isomet, Buehler, Lake Bluff, IL) under copious water to expose flat freshly-cut material surface. Each block was then cut perpendicular to the flat surface into slabs of 1mm thickness using the same saw. The block of slabs was then rotated 90° and again cut perpendicular to the surface. IPS e.max CAD and Paradigm™ MZ100 microbars of cross sectional area $1 \pm 0.1 \text{ mm}^2$ (Figure 4), were separated to be utilized for microtensile bond strength test according to the “non-trimming” method of the μTBS test. The IPS e.max CAD microbars were crystallized in a ceramic furnace (EP 600 Combi, Ivoclar Vivadent, Liechtenstein) according to the manufacturer instructions presented in Table 2.

Figure 2: Crown materials, IPS e.max CAD (left), Paradigm MZ100 (right).
Figure 3: Conventional microtensile technique aims at bonding the specimen first then cutting it. The new technique proposed in this study aims at cutting specimens first then bonding them to avoid premature failures.
The IPS e.max CAD and Paradigm™ MZ100 microbars were embedded in a plastic rubbery base to expose the surface only for the surface treatment procedure. 50 μm aluminum oxide particles (Figure 5) were used for grit-etching the microbars using an air abrasion device (Microetcher ERC, Danville Engineering Inc., California) from a distance of approximately 10 mm for 15 seconds under 2 bar pressure at the nozzle (Figure 6). The microbars were then cleaned with compressed air for 30 seconds and then placed in distilled water in an ultrasonic cleaner (FS5 Fisher Scientific, Sheboygan, Wisconsin, United States) for 5 minutes and air-dried. Another group of IPS e.max CAD microbars was treated with 5 % hydrofluoric acid (IPS Ceramic Etching Gel, Ivoclar Vivadent, Liechtenstein) (Figure 5) for 20 seconds, and then washed thoroughly for 1 min under tap water and air-dried. Then, the grit-etched lithium disilicate ceramic (IPS. e.max CAD GE), the hydrofluoric acid etched lithium disilicate ceramic (IPS e.max CAD HF) and the grit-etched composite resin (Paradigm MZ100) were chemically treated with silane coupling agent (Table 1) according to manufacturer instructions (Table 3). The surface that was utilized in adhesion and exposed to surface treatment is the surface that was cut by the low speed saw.
Table 2: Crystallization program for the IPS e.max CAD.

<table>
<thead>
<tr>
<th>Material</th>
<th>Stand-by temperature (°C)</th>
<th>Closing time (min.)</th>
<th>Heating rate (°C/min)</th>
<th>Firing temperature T1 (°C)</th>
<th>Holding time (min.)</th>
<th>Heating rate (°C/min)</th>
<th>Final firing temperature T2 (°C)</th>
<th>Holding time (min.)</th>
<th>Vacuum 1 (°C)</th>
<th>Vacuum 2 (°C)</th>
<th>Long term cooling (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>IPS e.max CAD</td>
<td>403</td>
<td>6:00</td>
<td>60</td>
<td>770</td>
<td>0:10</td>
<td>30</td>
<td>850</td>
<td>10:00</td>
<td>550</td>
<td>770</td>
<td>700</td>
</tr>
</tbody>
</table>
Figure 5: Surface treatment materials, Aluminum oxide 50 micron powder (left), IPS Ceramic etching gel (right)

Figure 6: The Grit-etching procedure; a) The microetcher and dust collector cabinet, b) microbars embedded in plastic rubbery base ready for grit-etching, c) Grit-etching the microbar
- **Dentin microbars:**

Intact caries-free human molars were collected and sterilized with gamma radiation (Gamma cell 220, Atomic Energy Ltd, Mississauga, Canada) at the Department of Chemical Engineering and Applied Chemistry, University of Toronto. Teeth were placed in a glass jar that was placed in a cobalt chamber 5.5x8 inches, and exposed for 4 hours. The radiation dose rate was 0.3 KGy/h. This method of sterilization has been proved to not alter the tooth tissue structure and mechanical or physical properties.\(^{202,203}\)

Teeth were kept, whenever possible, in distilled water in a refrigerator during and between all experimental procedures, in order to preserve their optimum mechanical and physical properties. Roots were then embedded in chemically-cured acrylic resin bases (Ivolen, Ivoclar Vivadent, Liechtenstein, Germany) to facilitate handling during cutting procedures.

The crown was cut perpendicularly to the long axis at approximately 4 mm from the cemento-enamel junction with a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL) under copious water to expose a flat dentin surface. If needed, the surface was further ground with the same saw until complete removal of enamel was achieved. Each tooth was cut perpendicular to the flat dentin surface into slabs of 1 mm thickness using the low speed cutting saw under copious water. The block of slabs was then rotated 90° and cut perpendicular to the surface. Dentin rods \(1 \pm 0.1 \text{ mm}^2\) in cross-sectional area were separated (Figure 4) to be utilized for microtensile bond strength test according to the “non-trimming” method of the μTBS test. The coronal surface of dentin to be utilized in adhesion was adjusted with a wheel diamond bur (#2909 314 040, Brassler, Komet, USA) similar to the one used in crown preparation listed above to induce clinically relevant standardized surface roughness.
Composite Z100™ Restorative Microbars:

Z100™ Restorative Composite (3M ESPE, St Paul, MN, USA) (Figure 7) was used to make a composite block in 2 mm increments. A Demi LED (light-emitting-diode) light polymerization unit (Kerr Corporation, Middleton, WI, USA, 1100-1200 mW/cm²) was used for light-curing each increment for 40 seconds. The surface layer of the block was cut using the low-speed cutting saw under copious water to produce a flat composite surface. The block was cut perpendicular to the flat composite surface into slabs of 1 mm thickness using a low speed cutting saw (Isomet, Buehler, Lake Bluff, IL) under copious water. The block of slabs was then rotated 90° and again cut perpendicular to the surface. Composite microbars 1± 0.1 mm² in cross-sectional area were separated, to be utilized for μTBS test according to the “non-trimming” method. The surface to be utilized in adhesion was adjusted with a wheel diamond bur (#2909 314 040, Brassler, Komet, USA) to induce clinically relevant standardized surface roughness.

Figure 7: Composite Z100 Restorative

3.2.2.2 Bonding procedure:

Dimensions of microbars to be bonded were measured using a digital caliper (Mastercraft, Toronto, Canada) and the surface area was calculated. The microbars were
randomly distributed among the groups. CAD/CAM microbars (IPS e.max CAD GE, IPS e.max CAD HF and Paradigm MZ100) were bonded to either dentin or composite resin (composite Z100 restorative) microbars. The microbars to be bonded to each other were placed in the attachment device (Figure 8) for trial positioning, a 3x magnifying loupes were used to make sure that the microbars were well adapted to each other. Once the position of the rods was satisfactory, the bonding procedures were started. For groups to be bonded using RelyX ARC cement, the surface was etched by phosphoric acid for 15 seconds and bonding agent was applied according to the manufacturer instructions (Table 3). For groups to be bonded using Multilink cement, the surface was treated by self-etching Multilink primer according to the manufacturer instructions (Table 3).

Four cements were used for the cementation procedures: two adhesive-based cements (RelyX-ARC, 3M-ESPE and Multilink Automix, Ivoclar-Vivadent) and two self-adhesive resin cements (RelyX-Unicem, 3M-ESPE; Clearfil SA, Kuraray), each cement was mixed according to the manufacturer instructions (Table 3) and applied to the microbar specimens using microbrushes. The microbars were pressed against each other using a fixed pressure (146 gram) applied on the attachment device to reach a minimal film thickness. Excess resin was removed from around the microbars with microbrushes. Luting resins were polymerized in 20 seconds stages in two opposite sides of the specimens using a Demi LED (light-emitting-diode) light polymerization unit (Kerr Corporation, Middleton, WI, USA, 1100-1200 mW/cm²). The microbar specimens were inspected under stereomicroscope at 10x magnification for the interface integrity (i.e. no voids or bubbles at the interface).
3.2.2.3 **Thermocycling:**

Bonded specimens were stored in distilled water at 37°C for 24 hours. They were then subjected to artificial thermal ageing (Thermocycling procedure) according to the ISO (The International Organization for Standardization, ISO TR 11450 standard, 1994) recommendations. Thermocycles were performed using a dwell time of 30 seconds in each bath and a transfer time of 15 seconds between baths for 500 cycles between 5°C and 55°C. Then specimens underwent the bond testing procedures immediately after thermocycling.
Table 3: Materials and sequence followed for cement application.

<table>
<thead>
<tr>
<th>Classification</th>
<th>Materials (Manufacturers)</th>
<th>Etchant</th>
<th>primer</th>
<th>Adhesive</th>
<th>Silane coupling agent (ceramic primer)</th>
<th>Luting Resin Cement</th>
</tr>
</thead>
<tbody>
<tr>
<td>Self-adhesive resin cements</td>
<td>RelyX Unicem (3M ESPE, St Paul, USA)</td>
<td>------</td>
<td>------</td>
<td>------</td>
<td>Apply 3M™ RelyX™ ceramic primer to the roughened crown material and allow to dry for 5 seconds.</td>
<td>Dispense the cement onto a mixing pad and mix for 10 seconds, then apply the cement in a thin layer on the bonding surfaces with micro brush, remove excess, light-cure (20s).</td>
</tr>
<tr>
<td></td>
<td>Clearfil SA (Kuraray Medical Inc, Okayama, Japan)</td>
<td>------</td>
<td>------</td>
<td>------</td>
<td>Apply Clearfil ceramic primer to the roughened crown material and dry gently with oil-free air.</td>
<td>Dispense and mix the cement using the automixtips, then apply the cement in a thin layer on the bonding surfaces with micro brush, remove excess, light-cure (20s).</td>
</tr>
<tr>
<td>Classification</td>
<td>Materials (Manufacturers)</td>
<td>Etchant</td>
<td>primer</td>
<td>Adhesive</td>
<td>Silane coupling agent (ceramic primer)</td>
<td>Luting Resin Cement</td>
</tr>
<tr>
<td>----------------</td>
<td>--------------------------</td>
<td>---------</td>
<td>--------</td>
<td>----------</td>
<td>-------------------------------------</td>
<td>-------------------</td>
</tr>
<tr>
<td>Adhesive-based resin cements</td>
<td><strong>Multilink Automix</strong> <em>(Ivoclar Vivadent, Liechtenstein)</em></td>
<td>-----</td>
<td><strong>Mix Multilink Primer liquids A and B in a 1:1 mixing ratio, apply with a micro-brush on the bonding substrate surface, allow 15 seconds reaction time, then gently dry with air. The primer is self-curing.</strong></td>
<td>-----</td>
<td><strong>Apply Monobond Plus primer to the roughened crown material, let to react for 60 sec, then dry gently with water- and oil-free air.</strong></td>
<td>Dispense and mix the cement using the automixtips, then apply the cement in a thin layer on the bonding surfaces with micro brush, remove excess, light-cure (20s).</td>
</tr>
<tr>
<td></td>
<td><strong>RelyX™ ARC</strong> <em>(3M ESPE, St Paul, USA)</em></td>
<td><strong>Apply Scotchbond ™ Etchant for 15 seconds, water rinse for 10 seconds. Blot excess water with a moist cotton pellet.</strong></td>
<td>-----</td>
<td><strong>Apply 2 coats of 3M™ Single Bond Adhesive to the bonding substrate using microbrushes, let to dry for 5 seconds. Then light-cured for 10 seconds.</strong></td>
<td><strong>Apply 3M™ RelyX™ ceramic primer to the roughened crown material and allow to dry for 5 seconds.</strong></td>
<td><strong>Dispense the cement onto a mixing pad and mix for 10 seconds, then apply the cement in a thin layer on the bonding surfaces with micro brush, remove excess, light-cure (20s).</strong></td>
</tr>
</tbody>
</table>
3.2.2.4 Microtensile bond strength testing (µTBS):

Cyanoacrylate glue (Krazy glue, Columbus, OH, USA) was used to attach the microtensile specimens to the opposing free-sliding halves of a testing jig. The testing jig was specially designed to transfer tensile forces to the specimen purely without any torque and to fit the microtensile testing machine (BISCO Microtensile Tester, BISCO Inc., Figure 9). The specimens were loaded in tension to failure at a crosshead speed of 0.5 mm /min, and the µTBS was expressed in MPa. If the adhesive interface of any specimen was contaminated with cyanoacrylate glue, the specimen was discarded because the bond strength would not have been represented correctly.

Figure 9: Testing jig placed in the microtensile-testing machine.
Figure 10: Bonded microbars glued using cyanoacrylate glue to the free sliding parts of a testing jig, and loaded in tension till failure at cross head speed 0.5 mm/min.

3.2.2.5 Evaluation of the mode of failure:

A single operator inspected the two halves of each specimen under a stereomicroscope (Wild/Leica M3Z Stereo Microscope, Heerbrugg, Switzerland) at 40x magnification. The modes of failure were evaluated according to the following scheme:

1= Adhesive (crown material/cement interface): when at least 75% of the cement layer exists on the substrate material (dentin or composite)

2= Adhesive failure (substrate/cement interface): When at least 75% of the cement layer exists on the crown material (IPS e.max CAD or Paradigm MZ100)

3= Mixed: when adhesive failure occurs at both interfaces, the cement layer exists on less than 75 % of the crown material and substrate.

4= Cohesive failure of the crown material

5= Cohesive failure of the substrate (dentin or composite)

6= Mixed cohesive/adhesive: when adhesive failure is accompanied with any cohesive failure
3.2.2.6 **Statistical analysis:**

Statistical analysis was computed using SPSS (PC+ version 20 software, Chicago, IL, USA). Means and standard deviation values of the µTBS were calculated. This data is quantitative (continuous) data. Statistical analysis was carried out using Factorial analysis of variance (Factorial ANOVA) to explore the impact of CAD/CAM material, type of cement and substrate on the µTBS. CAD/CAM material was divided in three levels according to the material/surface treatment combination (Group 1: IPS e.max CAD GE; Group 2: IPS e.max HF; Group 3: Paradigm MZ 100). Type of cement was divided in four levels (Group 1: Rely X Unicem; Group 2: Clearfil SA; Group 3: Multilink Automix; Group 4: Rely X ARC). Substrate was divided in two levels (Group 1: Dentin; Group 2: Composite resin). The significance level was set at $P \leq 0.05$ and 95% confidence level.
4. Results

The results of factorial ANOVA are presented in Table 4. Interaction effect between CAD/CAM material, type of cement and substrate was statistically significant, $F(6, 216) = 9.547, (p<0.001)$.

Table 4: Factorial ANOVA (Dependent variable: Bond strength; Independent variables: CAD/CAM material, type of cement and type of the substrate)

<table>
<thead>
<tr>
<th>Source</th>
<th>Type III Sum of Squares</th>
<th>df</th>
<th>Mean Square</th>
<th>F</th>
<th>Sig.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Corrected Model</td>
<td>9478.270$^a$</td>
<td>23</td>
<td>412.099</td>
<td>124.094</td>
<td>.000</td>
</tr>
<tr>
<td>Intercept</td>
<td>87225.426</td>
<td>1</td>
<td>87225.426</td>
<td>26265.911</td>
<td>.000</td>
</tr>
<tr>
<td>CAD/CAM material</td>
<td>3823.080</td>
<td>2</td>
<td>1911.540</td>
<td>575.616</td>
<td>.000</td>
</tr>
<tr>
<td>Substrate</td>
<td>1642.325</td>
<td>1</td>
<td>1642.325</td>
<td>494.548</td>
<td>.000</td>
</tr>
<tr>
<td>Type of Cement</td>
<td>3452.245</td>
<td>3</td>
<td>1150.748</td>
<td>346.521</td>
<td>.000</td>
</tr>
<tr>
<td>CAD/CAM material * Substrate</td>
<td>178.997</td>
<td>2</td>
<td>89.499</td>
<td>26.950</td>
<td>.000</td>
</tr>
<tr>
<td>CAD/CAM material * Type of Cement</td>
<td>134.965</td>
<td>6</td>
<td>22.494</td>
<td>6.774</td>
<td>.000</td>
</tr>
<tr>
<td>Substrate * Type of Cement</td>
<td>56.427</td>
<td>3</td>
<td>18.809</td>
<td>5.664</td>
<td>.001</td>
</tr>
<tr>
<td>CAD/CAM material * Substrate * Type of Cement</td>
<td>190.230</td>
<td>6</td>
<td>31.705</td>
<td>9.547</td>
<td>.000</td>
</tr>
<tr>
<td>Error</td>
<td>717.306</td>
<td>216</td>
<td>3.321</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>97421.001</td>
<td>240</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Corrected Total</td>
<td>10195.576</td>
<td>239</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

a. R Squared = .930 (Adjusted R Squared = .922)

4.1 Comparison between resin cements

Since a significant interaction was detected, comparison between types of cements was performed within levels of the other independent factors; CAD/CAM material (Group 1: IPS e.max CAD GE; Group 2: IPS e.max HF; Group 3: Paradigm MZ 100) and substrate (Group 1: Dentin; Group 2: Composite resin). The follow-up analysis was carried out using one-way
analysis of variance (one-way ANOVA) for every level of comparison. One-way ANOVA showed significance at $p<0.001$ at all levels of comparison. The results of the one-way ANOVA are presented in Table 5. Post-hoc comparisons were carried using Tukey’s test; the significance level was set at $P \leq 0.05$ and 95 % confidence level. The means and the standard deviation (SD) values are presented in Table 6.

<table>
<thead>
<tr>
<th>CAD/CAM material</th>
<th>Substrate</th>
<th>Sum of Squares</th>
<th>df</th>
<th>Mean Square</th>
<th>F</th>
<th>Sig.</th>
</tr>
</thead>
<tbody>
<tr>
<td>IPS e.max CAD GE</td>
<td>Dentin</td>
<td>Between Groups</td>
<td>410.171</td>
<td>3</td>
<td>136.724</td>
<td>56.145</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Within Groups</td>
<td>87.667</td>
<td>36</td>
<td>2.435</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Composite resin</td>
<td>Between Groups</td>
<td>424.279</td>
<td>3</td>
<td>141.426</td>
<td>75.720</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Within Groups</td>
<td>67.239</td>
<td>36</td>
<td>1.868</td>
<td></td>
</tr>
<tr>
<td>IPS e.max CAD HF</td>
<td>Dentin</td>
<td>Between Groups</td>
<td>1020.627</td>
<td>3</td>
<td>340.209</td>
<td>100.513</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Within Groups</td>
<td>121.851</td>
<td>36</td>
<td>3.385</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Composite resin</td>
<td>Between Groups</td>
<td>346.209</td>
<td>3</td>
<td>115.403</td>
<td>41.820</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Within Groups</td>
<td>99.342</td>
<td>36</td>
<td>2.760</td>
<td></td>
</tr>
<tr>
<td>Paradigm MZ100</td>
<td>Dentin</td>
<td>Between Groups</td>
<td>843.215</td>
<td>3</td>
<td>281.072</td>
<td>55.920</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Within Groups</td>
<td>180.946</td>
<td>36</td>
<td>5.026</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Composite resin</td>
<td>Between Groups</td>
<td>789.367</td>
<td>3</td>
<td>263.122</td>
<td>59.106</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Within Groups</td>
<td>160.261</td>
<td>36</td>
<td>4.452</td>
<td></td>
</tr>
</tbody>
</table>
Table 6: Descriptive showing mean and standard deviation and the pairwise comparison of the type of cements

<table>
<thead>
<tr>
<th>CAD/CAM material</th>
<th>Substrate</th>
<th>Cement</th>
<th>N</th>
<th>Mean (SD) (MPa)*</th>
<th>95% Confidence Interval for Mean</th>
</tr>
</thead>
<tbody>
<tr>
<td>IPS e.max CAD GE</td>
<td>Dentin</td>
<td>Rely X Unicem</td>
<td>10</td>
<td>10.87 (2.06) (c)</td>
<td>9.39-12.34</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Clearfil SA</td>
<td>10</td>
<td>8.61 (1.34) (o)</td>
<td>7.64-9.57</td>
</tr>
<tr>
<td></td>
<td></td>
<td>MultiLink Automix</td>
<td>10</td>
<td>14.47 (0.90) (w)</td>
<td>13.82-15.11</td>
</tr>
<tr>
<td></td>
<td>Composite resin</td>
<td>Rely X Unicem</td>
<td>10</td>
<td>11.59 (1.28) (d)</td>
<td>10.67-12.50</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Clearfil SA</td>
<td>10</td>
<td>13.91 (0.82) (c)</td>
<td>13.32-14.49</td>
</tr>
<tr>
<td></td>
<td></td>
<td>MultiLink Automix</td>
<td>10</td>
<td>17.6 (1.69) (b)</td>
<td>16.38-18.81</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Rely X ARC</td>
<td>10</td>
<td>20.03 (1.50) (a)</td>
<td>18.95-21.10</td>
</tr>
<tr>
<td>IPS e.max CAD HF</td>
<td>Dentin</td>
<td>Rely X Unicem</td>
<td>10</td>
<td>11.15 (1.38) (c)</td>
<td>10.16-12.14</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Clearfil SA</td>
<td>10</td>
<td>10.58 (1.95) (c)</td>
<td>9.18-11.97</td>
</tr>
<tr>
<td></td>
<td></td>
<td>MultiLink Automix</td>
<td>10</td>
<td>16.2 (1.48) (b)</td>
<td>15.13-17.26</td>
</tr>
<tr>
<td></td>
<td>Composite resin</td>
<td>Rely X Unicem</td>
<td>10</td>
<td>22.48 (1.82) (d)</td>
<td>21.17-23.78</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Clearfil SA</td>
<td>10</td>
<td>18.43 (0.92) (w)</td>
<td>17.76-19.09</td>
</tr>
<tr>
<td></td>
<td></td>
<td>MultiLink Automix</td>
<td>10</td>
<td>22.6 (2.12) (b)</td>
<td>21.08-24.11</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Rely X ARC</td>
<td>10</td>
<td>26.75 (1.52) (b)</td>
<td>25.66-27.83</td>
</tr>
<tr>
<td>Paradigm MZ100</td>
<td>Dentin</td>
<td>Rely X Unicem</td>
<td>10</td>
<td>17.78 (2.04) (c)</td>
<td>16.31-19.24</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Clearfil SA</td>
<td>10</td>
<td>17.37 (2.75) (c)</td>
<td>15.39-19.34</td>
</tr>
<tr>
<td></td>
<td></td>
<td>MultiLink Automix</td>
<td>10</td>
<td>21.45 (1.70) (b)</td>
<td>20.23-22.66</td>
</tr>
<tr>
<td></td>
<td>Composite resin</td>
<td>Rely X Unicem</td>
<td>10</td>
<td>23.16 (1.43) (c)</td>
<td>22.13-24.18</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Clearfil SA</td>
<td>10</td>
<td>21.8 (1.11) (c)</td>
<td>21.00-22.59</td>
</tr>
<tr>
<td></td>
<td></td>
<td>MultiLink Automix</td>
<td>10</td>
<td>29.03 (3.24) (b)</td>
<td>26.71-31.35</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Rely X ARC</td>
<td>10</td>
<td>32.78 (1.99) (a)</td>
<td>31.35-34.20</td>
</tr>
</tbody>
</table>

* Significant at P ≤ 0.05, Groups with different letters show statistically significant difference according to Tukey’s test results.
4.1.1 Grit-etched lithium disilicate glass ceramic (IPS e.max CAD GE)

Comparison between different types of cement was carried out when these cements were used to bond the IPS e.max CAD grit-etched to dentin and composite resin substrates. The means and the standard deviation (SD) values are presented in Figure 11 and Table 6.

Figure 11: Bar chart of the mean values of µTBS for different cements with IPS e.max CAD grit-etched. The error bars represent standard deviation. For every level of comparison, groups with different letters show a statistically significant difference according to Tukey's test results.
4.1.1.1 Dentin substrate:

One-way ANOVA was conducted to explore the effect of the type of cement on $\mu$TBS when these cements were used to bond the IPS e.max CAD grit-etched to dentin. There was a statistically significant difference ($p<0.001$) (Table 5). Post-hoc comparisons using Tukey’s test indicated that Rely X Arc had the highest mean $\mu$TBS, followed by Multilink Automix cement then Rely X Unicem. Clearfil SA showed the lowest mean bond strength.

4.1.1.2 Composite resin substrate:

One-way ANOVA was conducted to explore the effect of the type of cement on the $\mu$TBS when these cements were used to bond the IPS e.max CAD GE to composite resin. There was a statistically significant difference ($p<0.001$) (Table 5). Post-hoc comparisons using Tukey’s test indicated that Rely X ARC had the highest statistically significant mean $\mu$TBS, followed by Multilink Automix cement then Clearfil SA. Rely X Unicem showed statistically significant lowest mean bond strength.
4.1.2 Hydroflouric acid etched lithium disilicate glass ceramic (IPS e.max CAD HF)

Comparison between different types of cements was carried when these cements were used to bond the IPS e.max CAD HF etched to dentin and composite resin substrates. The means and the standard deviation (SD) values are presented in Figure 12 and Table 6.

Figure 12: Bar chart of the mean values of µTBS for different cements with IPS e.max CAD HF. The error bars represent standard deviation. For every level of comparison, groups with different letters are statistically significant different according to Tukey’s test results.
4.1.2.1 Dentin Substrate:

One-way ANOVA was conducted to explore the effect of the type of cement on $\mu$TBS, when these cements were used to bond IPS e.max CAD HF to dentin. There was a statistically significant difference ($p < 0.001$)(Table 5). Post-hoc comparisons using Tukey’s test indicated that Rely X ARC had the highest mean $\mu$TBS, followed by Multilink Automix cement. There was no statistically significant difference between Clearfil SA and Rely X Unicem, which had the lowest mean bond strength.

4.1.2.2 Composite Substrate:

One-way ANOVA was conducted to explore the effect of type of cement on the $\mu$TBS, when these cements were used to bond IPS e.max CAD HF to composite resin. There was a statistically significant difference at $p < 0.001$ (table 5). Post-hoc comparisons using the Tukey’s test indicated that Rely X ARC has the highest mean $\mu$TBS, followed by Multilink Automix cement which showed no statistically significant difference from Rely X Unicem. Clearfil SA showed the lowest mean bond strength.
4.1.3 Indirect composite resin (Paradigm MZ100)

Comparison between different types of cements was carried when these cements were used to bond Paradigm MZ100 to dentin and composite substrates. The means and standard deviation (SD) values are presented in Figure 13 and Table 6.

Figure 13: Bar chart of the mean values of μTBS for different cements with Paradigm MZ100. The error bars represent standard deviation. For every level of comparison, groups with different letters are statistically significant different according to Tukey’s test results.
4.1.3.1 Dentin substrate:

One-way ANOVA was conducted to explore the effect of type of cement on µTBS, when these cements were used to bond Paradigm MZ100 to dentin. There was a statistically significant difference ($p < 0.001$) (Table 5). Post-hoc comparisons using Tukey’s test indicated that Rely X ARC had the highest statistically significant mean µTBS, followed by Multilink Automix cement. There was no statistically significant difference between Clearfil SA and RelyX Unicem, which showed the lowest mean bond strength.

4.1.3.2 Composite resin substrate:

One-way ANOVA was conducted to explore the effect of type of cement on the µTBS, when these cements were used to bond Paradigm MZ100 to composite resin. There was a statistically significant difference ($p < 0.001$) (Table 5). Post-hoc comparisons using Tukey’s test indicated that Rely X ARC had the highest mean µTBS. This was followed by Multilink Automix cement. There was no statistically significant difference between Clearfil SA and RelyX Unicem which showed the lowest mean bond strength.

A summary of the graphs presented in this section comparing different resin cements is presented in Figure 21.
4.2 **Comparison between substrates:**

Due to the significant interaction among different factors detected by the factorial ANOVA, further comparison between the substrates was carried within levels of the other independent factors: Type of Cement (Group 1: Rely X Unicem; Group 2: Clearfil SA; Group 3: Multilink Automix; Group 4: Rely X ARC) and CAD/CAM material (Group 1: IPS e.max CAD grit-etched; Group 2: IPS e.max HF; Group 3: Paradigm MZ 100). The follow-up analysis to a significant interaction in factorial ANOVA was carried out using independent sample t-test. The results of the independent sample t-test are presented in Table 7. The significance level was set at $P \leq 0.05$ and 95% confidence level. The means and the standard deviation (SD) values are presented in Table 6.
<table>
<thead>
<tr>
<th>Type OF Cement</th>
<th>CAD/CAM material</th>
<th>Levene’s Test for Equality of Variances</th>
<th>t-test for Equality of Means</th>
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</thead>
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<tr>
<td></td>
<td></td>
<td>F</td>
<td>Sig.</td>
</tr>
<tr>
<td>Rely X Unicem</td>
<td>EMX GE</td>
<td>E.V.A</td>
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</tr>
<tr>
<td></td>
<td></td>
<td>E.V.N.A</td>
<td></td>
</tr>
<tr>
<td></td>
<td>EMX HF</td>
<td>E.V.A</td>
<td>0.495</td>
</tr>
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<td></td>
<td></td>
<td>E.V.N.A</td>
<td></td>
</tr>
<tr>
<td></td>
<td>PZ</td>
<td>E.V.A</td>
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</tr>
<tr>
<td></td>
<td></td>
<td>E.V.N.A</td>
<td></td>
</tr>
<tr>
<td>Clearfil SA</td>
<td>EMX GE</td>
<td>E.V.A</td>
<td>0.957</td>
</tr>
<tr>
<td></td>
<td></td>
<td>E.V.N.A</td>
<td></td>
</tr>
<tr>
<td></td>
<td>EMX HF</td>
<td>E.V.A</td>
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</tr>
<tr>
<td></td>
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<td>E.V.N.A</td>
<td></td>
</tr>
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<td></td>
<td>PZ</td>
<td>E.V.A</td>
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<td>E.V.N.A</td>
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<td>EMX GE</td>
<td>E.V.A</td>
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<td></td>
<td>E.V.N.A</td>
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<td>EMX HF</td>
<td>E.V.A</td>
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<td>E.V.N.A</td>
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<td></td>
<td>PZ</td>
<td>E.V.A</td>
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<td>E.V.N.A</td>
<td></td>
</tr>
<tr>
<td>Rely X ARC</td>
<td>EMX GE</td>
<td>E.V.A</td>
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<td></td>
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<td>E.V.N.A</td>
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</tr>
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<td>EMX HF</td>
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<td>E.V.N.A</td>
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</tr>
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</table>

*EMX GE: IPS. E.max CAD grit-etched; EMX HF: IPS e.max CAD HF; PZ: Paradigm MZ100; E.V.A: equal variance assumed; E.V.N.A: equal variance not assumed*
4.2.1 Self-adhesive resin cement (Rely X Unicem)

Comparison between different types of substrates was carried when Rely X Unicem cement was used to bond different crown materials. The means and standard deviation (SD) values are presented in Figure 14 and Table 6.

Figure 14: Bar chart of the mean values of µTBS for different substrates with Rely X Unicem cement. The error bars represent standard deviation. For every level of comparison, groups with different letters show a statistically significant difference according to Tukey’s test results.
4.2.1.1 Grit-etched lithium disilicate glass ceramic (IPS e.max CAD GE):

Independent sample t-test was conducted to explore the effect of the type of substrate on μTBS when Rely X Unicem cement was used to bond IPS e.max grit-etched. The results of the analysis show that there was no statistically significant difference between dentin and composite resin in the mean bond strength (p=0.364) (Table 7).

4.2.1.2 Hydrofluoric acid etched lithium disilicate glass ceramic (IPS e.max CAD HF):

Independent sample t-test was conducted to explore the effect of the type of substrate on μTBS, when Rely X Unicem cement was used to bond IPS e.max HF. There was statistical difference (p <0.001) (Table 7), the results of the analysis showed that composite resin had significantly higher mean μTBS compared to dentin.

4.2.1.3 Indirect composite resin (Paradigm MZ100):

Independent sample t-test was conducted to explore the effect of the type of substrate on μTBS, when Rely X Unicem cement was used to bond Paradigm MZ100. There was statistical difference (p <0.001) (Table 7), the results of the analysis showed that composite resin had significantly higher mean μTBS compared to dentin.
4.2.2 Self-adhesive resin cement (Clearfil SA)

Comparison between different types of substrates was carried when Clearfil SA cement was used to bond different crown materials. The means and standard deviation (SD) values are presented in Figure 15 and Table 6.

![Bar chart of the mean values of µTBS for different substrates with Clearfil SA cement. The error bars represent standard deviation. For every level of comparison, groups with different letters show a statistically significant difference according to Tukey’s test results.](image)

Figure 15: Bar chart of the mean values of µTBS for different substrates with Clearfil SA cement. The error bars represent standard deviation. For every level of comparison, groups with different letters show a statistically significant difference according to Tukey’s test results.
4.2.2.1 Grit-etched lithium disilicate glass ceramic (IPS e.max CAD GE):

Independent sample t-test was conducted to explore the effect of the type of substrate on \( \mu \text{TBS} \), when Clearfil SA cement was used to bond IPS e.max grit-etched. There was statistical difference at the \( p < 0.001 \) (Table 7), the results of the analysis showed that composite resin has higher mean \( \mu \text{TBS} \) compared to dentin.

4.2.2.2 Hydrofluoric acid etched lithium disilicate glass ceramic (IPS e.max CAD HF):

Independent sample t-test was conducted to explore the effect of the type of substrate on \( \mu \text{TBS} \), when Clearfil SA cement was used to bond IPS e.max HF. There was statistical difference at \( p < 0.001 \) (Table 7), the results of the analysis showed that resin has higher mean \( \mu \text{TBS} \) compared to dentin.

4.2.2.3 Indirect composite resin (Paradigm MZ100):

Independent sample t-test was conducted to explore the effect of the type of substrate on \( \mu \text{TBS} \), when Clearfil SA cement was used to bond Paradigm MZ100. There was statistical difference \( (p=0.001) \) (Table 7). The results of the analysis showed that composite resin has higher mean \( \mu \text{TBS} \) compared to dentin.
4.2.3 Self-etch adhesive-based resin cement (Multilink Automix)

Comparison between different types of substrates was carried when Multilink Automix cement was used to bond different crown materials. The means and standard deviation (SD) values are presented in Figure 16 and Table 6.

Figure 16: Bar chart of the mean values of μTBS for different substrates with Multilink cement. The error bars represent standard deviation. For every level of comparison, groups with different letters show a statistically significant difference according to Tukey’s test results.
4.2.3.1 Grit-etched lithium disilicate glass ceramic (IPS e.max CAD GE):

Independent sample t-test was conducted to explore the effect of the type of substrate on μTBS, when Multilink Automix cement was used to bond IPS e.max grit-etched. There was statistical difference ($p < 0.001$) (Table 7), the results of the analysis showed that composite resin had significantly higher mean μTBS compared to dentin.

4.2.3.2 Hydrofluoric acid etched lithium disilicate glass ceramic (IPS e.max CAD HF):

Independent sample t-test was conducted to explore the effect of the type of substrate on μTBS, when Multilink Automix cement was used to bond IPS e.max HF. There was statistical difference ($p < 0.001$) (Table 7), the results of the analysis showed that composite resin had significantly higher mean μTBS compared to dentin.

4.2.3.3 Indirect composite resin (Paradigm MZ100):

Independent sample t-test was conducted to explore the effect of the type of substrate on μTBS, when Multilink Automix cement was used to bond Paradigm MZ100. There was statistical difference ($p < 0.001$) (Table 7), the results of the analysis showed that composite resin had significantly higher mean μTBS compared to dentin.
4.2.4 Etch-and-rinse adhesive-based resin cement (Rely X ARC)

Comparison between different types of substrates was carried when Rely X Arc cement was used to bond different crown materials. The means and standard deviation (SD) values are presented in Figure 17 and Table 6.

Figure 17: Bar chart of the mean values of µTBS for different substrates with Rely X Arc cement. The error bars represent standard deviation. For every level of comparison, groups with different letters show a statistically significant difference according to Tukey’s test results.
4.2.4.1 Grit-etched lithium disilicate glass ceramic (IPS e.max CAD GE):

Independent sample t-test was conducted to explore the effect of the type of substrate on µTBS, when Rely X ARC cement was used to bond IPS e.max sandblasted. There was statistical difference ($p < 0.001$) (Table 7), the results of the analysis showed that composite resin had higher mean µTBS compared to dentin.

4.2.4.2 Hydrofluoric acid etched lithium disilicate glass ceramic (IPS e.max CAD HF):

Independent sample t-test was conducted to explore the effect of the type of substrate on µTBS, when Rely X ARC cement was used to bond IPS e.max HF. There was statistical difference ($p=0.001$) (Table 7). The results of the analysis showed that composite resin had higher mean µTBS compared to dentin.

4.2.4.3 Indirect composite resin (Paradigm MZ100):

Independent sample t-test was conducted to explore the effect of the type of substrate on µTBS, when Rely X ARC cement was used to bond Paradigm MZ100. There was statistical difference ($p=0.001$)(table 7). The results of the analysis showed that composite resin had higher mean µTBS compared to dentin.

A summary of the graphs presented in this section comparing different substrates is presented in Figure 22.
4.3 Comparison between CAD/CAM materials

Due to the significant interaction among different factors detected by factorial ANOVA, comparison between CAD/CAM materials was carried within levels of the other independent factors: Substrate (Group 1: Dentin; Group 2: Composite resin) and Type of Cement (Group 1: Rely X Unicem; Group 2: Clearfil SA; Group 3: Multilink Automix; Group 4: Rely X ARC). The follow-up analysis to the significant interaction in factorial ANOVA was carried out using one-way analysis of variance (one way ANOVA). The results of the one-way ANOVA are presented in Table 8. Post-hoc comparisons were carried using the Tukey’s test. The significance level was set at $P \leq 0.05$ and 95% confidence level. The means and standard deviation (SD) values are presented in Table 6.
Table 8: One way ANOVA (Dependent variable: Bond strength; Independent variable: CAD/CAM material)

<table>
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<tr>
<th>Substrate</th>
<th>Type of cement</th>
<th>Sum of Squares</th>
<th>df</th>
<th>Mean Square</th>
<th>F</th>
<th>Sig.</th>
</tr>
</thead>
<tbody>
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<td>Rely X</td>
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<td></td>
<td>Unicem</td>
<td>Within Groups</td>
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<td>3.454</td>
<td></td>
</tr>
<tr>
<td>Clearfil</td>
<td>Sa</td>
<td>Between Groups</td>
<td>422.409</td>
<td>2</td>
<td>211.204</td>
<td>47.926</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Within Groups</td>
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<td>27</td>
<td>4.407</td>
<td></td>
</tr>
<tr>
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<td>Automix</td>
<td>Between Groups</td>
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<td></td>
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<td>ARC</td>
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<td>76.55</td>
</tr>
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<td></td>
<td></td>
<td>Within Groups</td>
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<td>27</td>
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<td>Between Groups</td>
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<td>421.532</td>
<td>179.152</td>
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<td>Within Groups</td>
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<td>2.353</td>
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<tr>
<td>Composite</td>
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<td>Between Groups</td>
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<td>Within Groups</td>
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4.3.1 Dentin substrate

Comparison between different CAD/CAM materials was carried when bonded to dentin using different cements. The means and standard deviation (SD) values are presented in Figure 18 and Table 6.

![Bar chart of the mean values of µTBS for different CAD/CAM materials with Dentin. The error bars represent standard deviation. For every level of comparison, groups with different letters show a statistically significant difference according to Tukey’s test results.](image)

Figure 18: Bar chart of the mean values of µTBS for different CAD/CAM materials with Dentin. The error bars represent standard deviation. For every level of comparison, groups with different letters show a statistically significant difference according to Tukey’s test results.
4.3.1.1 **Self-adhesive resin cement (Rely X Unicem):**

One-way analysis of variance was conducted to explore the effect of the type of CAD/CAM material on $\mu$TBS, when bonded to dentin using Rely X Unicem cement. There was a statistically significant difference ($p < 0.001$) (Table 8). Post-hoc comparisons using Tukey’s test indicated that Paradigm MZ100 showed the highest mean bond strength. There was no statistically significant difference between IPS e.max CAD grit-etched and IPS e.max CAD HF etched, which showed the lowest mean bond strength.

4.3.1.2 **Self-adhesive resin cement (Clearfil SA):**

One-way analysis of variance was conducted to explore the effect of the type of CAD/CAM material on $\mu$TBS, when bonded to dentin using Clearfil SA cement. There was a statistically significant difference ($p <0.01$) (Table 8). Post-hoc comparisons using Tukey’s test indicated that Paradigm MZ100 showed the highest mean bond strength. This was followed by IPS e.max CAD HF etched. Finally, IPS e.max CAD grit-etched showed the lowest mean bond strength.

4.3.1.3 **Self-etch adhesive-based resin cement (Multilink Automix):**

One-way analysis of variance was conducted to explore the effect of the type of CAD/CAM material on $\mu$TBS, when bonded to dentin using Multilink Automix cement. There was a statistically significant difference ($p < 0.001$) (Table 8). Post-hoc comparisons using Tukey’s test indicated that Paradigm MZ100 showed the highest mean bond strength. This was followed by IPS e.max CAD HF etched. Finally, IPS e.max CAD grit-etched showed the lowest mean bond strength.
4.3.1.4 Etch-and-rinse adhesive-based resin cements (Rely X ARC):

One-way analysis of variance was conducted to explore the effect of the type of CAD/CAM material on μTBS, when bonded to dentin using Rely X ARC cement. There was a statistically significant difference (p < 0.001) (Table 8). Post-hoc comparisons using Tukey’s test indicated that Paradigm MZ100 showed the highest mean bond strength, followed by IPS e.max CAD HF etched. Finally, IPS e.max CAD grit-etched showed the lowest mean bond strength.

4.3.2 Composite resin substrate

Comparison between different CAD/CAM materials was carried out when bonded to composite Z100 restorative using different cements. The means and standard deviation (SD) values are presented in Figure 19 and Table 6.

4.3.2.1 Self-adhesive resin cement (Rely X Unicem):

One-way analysis of variance was conducted to explore the effect of the type of CAD/CAM material on μTBS, when bonded to composite resin using Rely X Unicem cement. There was a statistically significant difference (p < 0.001) (Table 8). Post-hoc comparisons using Tukey’s test indicated no statistically significant difference between Paradigm MZ100 and IPS e.max CAD HF etched which showed the highest mean bond strength. This was followed by IPS e.max CAD grit-etched that showed the lowest mean bond strength.

4.3.2.2 Self-adhesive resin cement (Clearfil SA):

One-way analysis of variance was conducted to explore the effect of the type of CAD/CAM material on μTBS, when bonded to composite resin using Clearfil SA cement. There was a statistically significant difference (p < 0.001) (Table 8). Post-hoc comparisons using Tukey’s test indicated that Paradigm MZ100 showed the highest mean bond strength, followed
by IPS e.max CAD HF etched. Finally, IPS e.max CAD grit-etched showed the lowest mean bond strength.

Figure 19: Bar chart of the mean values of μTBS for different CAD/CAM materials with Dentin. The error bars represent standard deviation. For every level of comparison, groups with different letters show a statistically significant difference according to Tukey’s test results.
4.3.2.3  **Self-etch adhesive-based resin cement (Multilink Automix):**

One-way analysis of variance was conducted to explore the effect of the type of CAD/CAM material on µTBS, when bonded to composite resin using Multilink Automix cement. There was a statistically significant difference ($p < 0.001$) (Table 8). Post-hoc comparisons using Tukey’s test indicated that Paradigm MZ100 showed the highest mean bond strength, followed by IPS e.max CAD HF etched. Finally, IPS e.max CAD grit-etched showed the lowest mean bond strength.

4.3.2.4  **Etch-and-rinse adhesive-based resin cement (Rely X ARC):**

One-way analysis of variance was conducted to explore the effect of the type of CAD/CAM material on µTBS, when bonded to composite resin using Rely X ARC cement. There was a statistically significant difference ($p < 0.001$) (Table 8). Post-hoc comparisons using Tukey’s test indicated that Paradigm MZ100 showed the highest mean bond strength, followed by IPS e.max CAD HF etched. Finally, IPS e.max CAD grit-etched showed the lowest mean bond strength.

A summary of the graphs presented in this section comparing CAD/CAM materials is presented in Figure 23.

4.4  **Modes of failure**

The distribution of the modes of failure in specimens of the microtensile test is shown in Figure 20. The majority of the failures were adhesive failure (crown material/cement interface) 57.08%, followed by adhesive failure (substrate/cement interface) 28.75%; and finally mixed failures 14.17%. 

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Figure 20: Pie chart showing distribution of the modes of failure in μTBS specimens.
Figure 21: Summary Bar chart of the mean values of μTBS for different types of cements. The error bars represent standard deviation. For every level of comparison, groups with different letters show a statistically significant difference according to Tukey’s test results.
Figure 22: Summary Bar chart of the mean values of μTBS for different Substrates. The error bars represent standard deviation. For every level of comparison, groups with different letters show a statistically significant difference according to Tukey's test results.
Figure 23: Summary Bar chart of the mean values of µTBS for different CAD/CAM materials. The error bars represent standard deviation. For every level of comparison, groups with different letters show a statistically significant difference according to Tukey’s test results.
5. Discussion

The microtensile bond strength (µTBS) test has been used to evaluate adhesion of dental restorations to the underlying tooth substrate. It offers several advantages over conventional tensile bond strength tests.\textsuperscript{16} The configuration of the samples concentrates the stresses at the adhesive interface as shown by the number of adhesive failures in the specimens of interest.\textsuperscript{151} Also, the smaller the surface area of the specimens the lower is the amount of defects and flaws of critical size at the adhesive interface giving better chance at evaluating the bonding performance at this interface.\textsuperscript{152} Also, µTBS test is considered more conservative compared to conventional tensile test, since more specimens are produced per tooth.\textsuperscript{172}

On the other hand, the current methodology for µTBS testing suffers from different flaws that can affect the reliability of the test. The cutting procedures of the bonded specimens induce stresses that cause several pretest failures in addition to weakening of the survived specimens.\textsuperscript{22,199} These pretest failures create a statistical problem when dealing with it. In the literature there are three approaches of addressing these pretest failures, and each approach can lead to a different result and conclusion from the same study.\textsuperscript{199}

In the current study a new methodology was developed for µTBS testing, where specimens were cut first and then bonded, thus avoiding the induction of unnecessary stresses to the bonded specimens. The efficacy of this method is demonstrated by the low standard deviation presented by our samples and the similar bond strength values between our study and those of others which made use of the traditional method.\textsuperscript{198,199} Moreover, this methodology has the advantage of preventing pretest failures thus avoiding the statistical problem observed with the conventional microtensile methodology.\textsuperscript{199} This methodology was also capable of
concentrating the stresses at the adhesive interface as presented by the prevalence of adhesive failures.

In this study our aim was to investigate the bond strength of two self-adhesive resin cements (Rely X Unicem and Clearfil SA) and two adhesive-based resin cements (Multilink Automix and Rely X Arc), when used to bond lithium disilicate glass ceramic (IPS e.max CAD) and composite resin CAD/CAM material (Paradigm MZ100) to two types of substrates (dentin and composite Z100 restorative). Comparing the performance of different cements, the etch-and-rinse adhesive-based resin cement (Rely X ARC) prevailed in all groups with the highest mean µTBS. The self-etch adhesive-based resin cement (Multilink Automix) was ranked second highest regarding µTBS in almost all groups. The self-adhesive resin cements (Rely X Unicem and Clearfil SA) both had the lowest bond strength in all groups. For dentin substrate, this can be explained by the better demineralization ability of the etch-and-rinse system with the maximum smear layer removal, exposing a greater quantity of dentinal tubules and collagen fibers to resin infiltration, yielding the well documented hybrid layer. The self-etch system only dissolves the smear layer, but doesn’t remove the dissolved calcium phosphates which become embedded within the interfacial transition zone and encapsulated by resin. These calcium phosphates are soluble and may explain the lower laboratory and clinical bonding performance. Also, the high concentrations of acidic resin monomers can make self-etch adhesives behave like hydrophilic permeable membranes which allow water movement from dentin to the restoration-adhesive interface and mediate hydrolytic reactions. Moreover, the residual solvent (water) that remains within the adhesive interface can weaken the bond. For these reasons the self-etching system showed less bond strength values compared to etch-and-rinse resin cement systems. However, self-etching adhesive-based resin
cements have some advantages over self-adhesive resin cements. The low bonding performance of self-adhesive resin cements could be explained by the weak infiltration of these cements into dentin, as suggested by De Munk et al (2004), who stated that self-adhesive resin cements were only capable of tangible infiltration of the dental surface, hence their inferior bonding performance. Furthermore, Monticelli et al (2008) stated that there was no demineralization of dentin with self-adhesive resin cements and no resin penetration. Also, the acidic component of the self-adhesive resin cements may interfere with the polymerization reaction as a result of improper neutralization of the acidic group, which in turn results in lower mechanical properties of the set cement and lower bond strength values. It is well known that acidic monomers affect the free radical polymerization initiators, reducing the degree of polymerization as well as the rate of polymerization of light and self-cured resin formulations. For these reasons self-adhesive resin cements had weaker bond strength compared to adhesive-based resin systems irrespective to the CAD/CAM material employed.

For composite resin substrate, this bonding performance could be related to the chemical composition of the cement systems. The unreacted acidic monomers in self-etching systems and self-adhesive systems might have negatively affected the polymerization reaction and jeopardize adhesion. These acidic monomers are hydrophilic and can mediate hydrolytic reactions that can negatively affect the bond strength. Therefore, the two-step self-etching system is advantageous in bond strength as a result of the two-step procedure that allows applying the acidic monomers first then curing it followed by placement of the mixed cement. The advantage of adhesive-based resin cements over self-adhesive resin cements has already been demonstrated by De Angelis et al (2004), who found higher bond strength for etch-and-rinse adhesive-based resin compared to self-etch adhesive-based resin cement, and both types
of the multistep adhesive-based resin cements showed higher bond strength values compared with self-adhesive resin cements.\textsuperscript{199}

When hydrofluoric acid etched lithium disilicate glass ceramic (IPS e.max CAD HF) was bonded to composite resin, Rely X Unicem (self-adhesive resin cement) results were similar to Multilink Automix (self-etch adhesive-based resin cement) which ranked second in bond strength, and was significantly higher than Clearfil SA (self-adhesive resin cement), with the lowest bond strength. This could be explained by the strong etching of the hydrofluoric acid that increased the surface roughness on the ceramic surface, which in turn is reflected in an increase in the micromechanical interlocking, and increase in the surface area that was silanated resulting in extra chemical bonding. The increased surface area and roughness may have been more beneficial to Rely X Unicem which has higher filler loading (72 wt.%\%) and bigger filler particle size (<12.5 μm) compared to other resin cement materials, which leads to high mechanical properties of the cement, and thus stronger engagement of the micro-roughness on the etched ceramic surface without fracture of the resin cement. Multilink filler loading is (45.5 wt.%\%) and filler particle size (0.9 μm) and Clearfil SA whose filler loading is (66 wt.%\%) and filler size (2.5 μm). The efficacy of etching lithium disilicate glass ceramic with hydrofluoric acid have been reported by De Menezes et al who found significant increase in the surface roughness for micromechanical bonding compared to grit-etched lithium disilicate glass ceramic.\textsuperscript{197}

However, Rely X Unicem and Clearfil SA presented similar results when hydrofluoric acid etched lithium disilicate ceramic (IPS e.max CAD HF) was bonded to dentin. This could be due to the 10-MDP monomer (10-methacryloyloxydecyl dihydrogen phosphate) groups in the Clearfil SA that enhance the chemical bonding to dentin by forming strong and
hydrolytically stable ionic bonds with the hydroxyapatite. Rely X Unicem has methacrylated phosphoric esters to chemically bind to the hydroxyapatite, but these esters are hydrolytically unstable with the release of phosphoric acid as by-product of hydrolysis which can cause further demineralization of dentin and weakening of the adhesive interface. On the other hand, Rely X Unicem has higher filler content and thus was expected to have higher mechanical properties that would positively affect the bond strength values, therefore, resulting in a similar performance for the two materials.

Interestingly, when Rely X Unicem was used to bond grit-etched lithium disilicate glass ceramic (IPS e.max CAD GE) to composite resin, the results were inferior to those presented by Clearfil SA. The literature indicates that grit-etching of lithium disilicate glass ceramic results in lesser roughness than when the material is treated with hydrofluoric acid. The reduced micromechanical interlocking may have compromised the bond strength between Rely X Unicem and lithium disilicate glass ceramic. However, Rely X Unicem had significantly higher bond strength than Clearfil SA when used to bond IPS e.max CAD GE to dentin. This could be related to the stress distribution and concentration at the adhesive interface as well as the resin cement mechanical properties. Van Noort et al (1989) stated that the modulus of elasticity of the materials forming the interface affects the stress distribution within the interface, and Tam et al (2000) indicated that fracture toughness of the interface is affected by the degree of interaction whether mechanical or chemical between the constituents of the interface. Clearfil SA is capable of achieving good chemical adhesion to dentin through its 10-MDP monomers. However, it has low modulus of elasticity 4.8 GPa compared to Rely X Unicem 7.9 GPa. This could lead to unfavorable stress concentration at the crown material/cement interface and lower fracture toughness of this interface.
In all other groups, Rely X Unicem behaved similarly to Clearfil SA with no statistical difference. This could be due to balance between factors affecting the bond strength for the two cements, like the stress distribution at the interfaces and the degree of mechanical and chemical interaction between the cement and the crowns material together with the underlying substrate.

Comparing different substrates, composite resin substrate prevailed in almost all groups with the highest $\mu$TBS, while dentin showed the lowest bond strength. This could be explained by the copolymerization between monomers of the resin cement and residual monomers of the composite substrate resulting in strong chemical bond. This explanation is in agreement with that of Kallio et al (2001) who stated that the unreacted monomer groups in a composite substrate could allow formation of covalent bonds through copolymerization between the substrate and repair resin or intermediary resin. Composite resin substrate showed no statistical difference in bond strength from dentin substrate when bonded to IPS e.max CAD GE using Rely X Unicem. This could be explained by the weak adhesion of Rely X Unicem to the grit-etched lithium disilicate glass ceramic either mechanically due to the shallow irregularities pattern created by grit-etching or chemically due to the chemical composition of the cement, which makes the resin cement/ceramic interface a weak point and decreases the fracture toughness of this interface and causes both substrates to behave similarly in bond strength.

Comparing different crown materials, Paradigm MZ100, a composite resin material, prevailed in almost all groups with the highest $\mu$TBS. The degree of conversion of Paradigm MZ100 is 84%, thus providing 16% of unreacted monomers for copolymerization with the resin cement monomers, resulting in strong chemical bond. Also, the silane-coupling agent is capable of increasing the bond strength between indirect composite restorations and resin
cements.\textsuperscript{123} Moreover, the mechanical difference between composite and ceramic is an important factor affecting the stress concentration at the adhesive interfaces.\textsuperscript{198} The more brittle ceramic material tends to fracture at the adhesive interface at lower values than the more resilient composite.\textsuperscript{198} This is in accordance with Van Noort et al. (1989) who showed that the higher the elastic modulus of the material, the higher the stress generated at the edge of the bonding interface.\textsuperscript{155} IPS e.max CAD HF presented similar results to Paradigm MZ100, when bonded to composite resin substrate using Rely X Unicem cement. The high filler loading of Rely X Unicem as well as the larger filler size leads to high mechanical properties of the cement resulting in more resistant micromechanical interlocking with the hydrofluoric acid etched IPS e.max CAD material to be strong, while the other interface has a strong chemical bond between the unreacted monomers of Rely X Unicem and the composite substrate, a situation that result in two strong interfaces and stress distribution that enhances the bond strength of the IPS e.max HF.

In all the other groups the hydrofluoric acid etched IPS e.max CAD showed superior bond strength to the grit-etched IPS e.max CAD. As previously mentioned the increased surface roughness created by the hydrofluoric acid etching over the grit-etched surface treatment may have enhanced the micromechanical interlocking.\textsuperscript{197} Hydrofluoric acid etched IPS e.max CAD and the grit-etched IPS e.max CAD presented similar results when bonded to dentin using Rely X Unicem cement. This could be explained by weak adhesion of Rely X Unicem to the dentin, so stress concentration at this interface could lead to inferior bond strength results of the hydrofluoric acid etched IPS e.max CAD. This explanation is in agreement with Brunzel et al (2010) who stated that the adhesive interface between Rely X Unicem and dentin is a weak link and was the cause of the recorded low tensile bond strength values.\textsuperscript{212}
The modes of failure evaluated in this study were mostly adhesive failure at the (crown material interface) 57.08%, followed by adhesive failure at the (substrate interface) 28.75%; and finally mixed failures 14.17%. This indicates that the stresses were concentrated at the adhesive interface with no cohesive failures. Cohesive failures are known to preclude measurement of the interfacial bond strength. This is in agreement with Salz et al (2010) who mentioned that below 2mm$^2$ of adhesive area, all specimens fail adhesively at the interface.\textsuperscript{151}
6. Limitations of the study

This study has the following limitations:

1. The study is an in-vitro study, which helps in rapid assessment of dental biomaterials bonding properties, but cannot substitute in-vivo studies.

2. Thermocycling in a water bath between 5 °C and 55 °C for 500 cycles is a minimal effect aging procedure. However, based on the ISO TR 11405 (1994), thermocycling for 500 cycles between 5 °C and 55 °C is a recommended protocol for aging dental interfaces prior to adhesion testing.

3. This study was performed using the new microtensile specimen preparation methodology while the conventional microtensile specimen preparation methodology was not performed in this study, which limits direct comparison of both methodologies. However, the results of this study are comparable with previous studies but with lower standard deviations. The absence of pre-test failures gives this methodology an extra advantage.

7. Conclusions

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

- Etch-and-rinse adhesive-based resin cements are capable of achieving the highest μTBS between CAD/CAM materials and different substrates.

- Self-adhesive resin cements showed the lowest bond strength irrespective of the crown material or bonding substrate.

- Composite resin crown material presents better bond strength results than lithium disilicate glass ceramic.

- Composite substrate showed better bonding performance than dentin substrate.


- Hydrofluoric acid etching of lithium disilicate glass ceramic resulted in better bond strength to different substrates than grit-etching of lithium disilicate glass ceramic.
Chapter 4 Microshear bond strength of self-adhesive resin cements to non-metallic crown CAD/CAM materials, composite and dentin
1. Abstract

Objectives: To determine microshear bond strength (μSBS) of two CAD/CAM crown materials when bonded to dentin or composite-resin substrate using two self-adhesive, etch-and-rinse and self-etch resin cement; and to compare the effect of two surface treatments of the ceramic crown material on μSBS. Methods: Lithium disilicate glass ceramic (IPS e.max CAD, Ivoclar-Vivadent/EMX), and composite-resin (Paradigm MZ-100, 3M-ESPE/PZ) blocks were sectioned into microbars approximately 1×1×5 mm³. Custom-made composite-resin (Z100 restorative, 3M-ESPE/ZC) blocks, and coronal dentin from freshly extracted molars were sectioned into slices 2mm ± 0.1mm thickness. EMX rods were either grit-etched (50μm Al₂O₃) or etched with HF acid and silanated. PZ rods were only grit-etched and silanated. EMX and PZ specimens were then bonded to either ZC or dentin slices using two self-adhesive cements (RelyX Unicem, 3M-ESPE; Clearfil SA, Kuraray) and two adhesive-based cements (Multilink Automix, Ivoclar-Vivadent;RelyX-ARC, 3M-ESPE) using custom-made device (n=10). Static load 146 g was maintained until initial setting of cement. Specimens were stored in water (37°C, 24 h), thermocycled (500 cycles, 5-55°C), then subjected to μSBS test. Means and SDs were calculated in MPa and data statistically analyzed by factorial ANOVA (α=0.05). Results: ANOVA revealed significant differences among the groups (P<0.05). Comparing cements, highest mean μSBS (8.65 ±0.3) was obtained with etch-and-rinse adhesive based cement (RelyX-ARC), while lowest mean μSBS (6.25 ±0.3) with self-adhesive resin cement (Rely-X Unicem). Comparing substrates, highest mean μSBS (7.5±1.5) was obtained with ZC, while lowest mean μSBS (6.9±1.6) with dentin. Comparing crown materials, highest mean μSBS was (7.59 ±0.24) with PZ, while lowest mean μSBS (6.57 ±0.24) with grit-etched EMX.

Conclusions: In overall adhesive-based cements resulted in higher mean μSBS, whereas self-
adhesive cements resulted in lower mean μSBS, irrespective of the substrate or crown material. For EMX, HF-treatment resulted in higher mean μSBS as compared to grit-etching.

2. Introduction

Clinically, the use of resin cements to bond esthetic restorations to the underlying substrate is growing substantially, due to their strong bonding and esthetic shades. Self-adhesive resin cements were recently introduced as a potential and equivalent substitute for the conventional adhesive-based resin cements since they are capable of achieving good adhesion to the tooth structure without the need to apply any adhesive system prior to cement application. Factors that can affect the bonding of indirect restorations to the underlying substrate include the type of cement, the type of the indirect restorative material, the surface treatment of the indirect restorative material and the nature of the bonding substrate. Several studies have been performed to determine the efficiency of self-adhesive resin cements and to investigate if they were possible replacements for current conventional adhesive resin cement systems. The results of these studies are controversial. Several in-vitro bond strength testing methods have been used to evaluate dental adhesion, such as shear, tensile, microshear and microtensile bond strength tests. The “micro” tests give the advantages of preparing several samples per tooth with conservative amount of tooth structure and materials.

Micro-shear bond strength (μSBS) testing is capable of concentrating the stresses at the adhesive interface compared to conventional shear bond strength testing, which in turn reflects on the modes of the adhesive failure within the test to be mainly adhesive and reflect a true interfacial debonding. In addition, smaller specimens are less likely to have critical
size defects that can affect the outcome of the test. This is the reason why “micro” tests are having relatively higher bond strength values compared to the “macro” test methods.\textsuperscript{152,172}

\(\mu\text{SBS}\) testing has an advantage over the conventional methodology used for microtensile testing, since there are no sectioning and trimming procedures for the bonded specimens, thus avoiding the pretest failures and the misinterpretation of the results that can be associated with conventional \(\mu\text{TBS}\) testing.\textsuperscript{172} Also, specimen preparation for \(\mu\text{TBS}\) testing is difficult to conduct and time-consuming compared to microshear.\textsuperscript{171} Conventional microshear testing involves only one adhesive interface where the cement is bonded directly to the tooth substrate and a blade or wire is applied to the interface inducing shearing forces.\textsuperscript{170} In this study our aim was to modify the conventional microshear testing methodology to involve two adhesive interfaces, where the crown material is bonded to the underlying substrate using resin cement, thus creating two interfaces: crown material/cement interface and substrate/cement interface, which is a more clinically relevant situation. Stress distribution at the adhesive interfaces is dependent on the elastic moduli of the components constituting the adhesive interfaces, which can alter the crack propagation within the adhesive interfaces and affect the recorded bond strength.\textsuperscript{155,207}

Another aim of this study was to investigate the bond strength of self adhesive resin cements versus adhesive-based resin cements when they are used to bond lithium disilicate glass ceramic material and composite resin material to the underlying dentin and composite resin substrates, and to compare the effect of two surface treatments on lithium disilicate ceramic.
3. Materials and Methods

3.1 Materials

All the materials used in the study and their composition (as reported by manufacturers) are listed in Table 9.

3.2 Methods:

3.2.1 Pilot study:

The current study was preceded by an external pilot study to detect any problems with the proposed methods of testing, where 2 samples per group were used in the pilot study. Following the completion of pilot study, 240 specimens were used for the main study.

3.2.2 Main study

Twenty-four groups were formed (n=10)(Figure 24). The number of specimens per group was calculated using web-based software® for sample size analysis at α=0.05 and 80% power and effect size equal to 0.307 which yields a sample size of 7 samples per group. We used 10 samples per group for this study to have more power. The effect size (f) was calculated by using the following formula, developed by Cohen (1999).

\[
f = d \times \frac{1}{2} \sqrt{\frac{k+1}{3(k-1)}}
\]

Where \(k\) is the number of groups and \(d\) is the difference in means \((x_1-x_2)\) divided by common standard deviation \((\sigma)\). Based on values obtained from the pilot study, \(x_1=6.8, x_2=5.43, \sigma=1.34\), the \(d\) value was calculated \((d=1.022)\), and \(f\) was calculated \((0.307)\).

® Power Analysis For ANOVA Designs [http://www.math.yorku.ca/SCS/Online/power/](http://www.math.yorku.ca/SCS/Online/power/)
Table 9: Materials and their composition (as reported by manufacturers).

<table>
<thead>
<tr>
<th>Material</th>
<th>Classification</th>
<th>Brand / Manufacturer</th>
<th>Composition</th>
<th>Lot #</th>
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<tbody>
<tr>
<td>CAD/CAM Materials</td>
<td>Lithium disilicate ceramic</td>
<td>IPS e.max CAD/ Ivoclar Vivadent, Liechtenstein</td>
<td>SiO₂ (57 – 80 wt.%), Li₂O (11 – 19 wt.%), K₂O (0 – 13 wt.%), P₂O₅ (0 – 11 wt.%), ZrO₂ (0 – 8 wt.%), ZnO (0 – 8 wt.%), Other and coloring oxides (0-12 wt.%)</td>
<td>P63946</td>
</tr>
<tr>
<td></td>
<td>Indirect composite resin</td>
<td>Paradigm™ MZ100/ 3M ESPE, St Paul, MN, USA</td>
<td>85 wt.% ultrafine zirconia-silica ceramic particles that reinforce a highly cross-linked polymeric matrix. The polymer matrix consists of BisGMA and TEGDMA, and employs a ternary initiator system.</td>
<td>N250997</td>
</tr>
<tr>
<td>Resin Cements</td>
<td>Self-adhesive resin cement system</td>
<td>RelyX™ Unicem/ 3M ESPE, St Paul, MN, USA</td>
<td>It consists of two pastes: Base paste: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, and stabilizers. Catalyst paste: methacrylate monomers, alkaline (basic) fillers, silanated fillers, initiator components, stabilizers, and pigments. (Filler=50% vol., 72 wt.%; avg. &lt; 12.5 μm)</td>
<td>427554</td>
</tr>
<tr>
<td></td>
<td>Self-adhesive resin cement system</td>
<td>RelyX Ceramic primer / 3M ESPE, St Paul, MN, USA</td>
<td>Stable solution of a prehydrolyzed silane-coupling agent, alcohol and water.</td>
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<tr>
<td>Material</td>
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<tr>
<td>Resin Cements</td>
<td>Self-adhesive resin cement system</td>
<td>Clearfil SA/Kuraray Medical Inc., Sakazu, Kurashiki, Okayama, Japan</td>
<td>It consists of two pastes: Paste A: 10-MDP, Bis-GMA, TEGDMA, Hydrophobic aromatic dimethacrylate, dl-Camphorquinone, BPO Initiator, Silanated barium glass filler and Silanated colloidal silica. Paste B: Bis-GMA, Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic dimethacrylate, Accelerators, Pigments, and Surface treated sodium fluoride, Silanated barium glass filler and Silanated colloidal silica. (Filler = 45% vol., 66 wt.%; avg. 2.5 μm)</td>
<td>011AAA</td>
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<tr>
<td>Silane coupling agent</td>
<td></td>
<td>Clearfil Ceramic primer/Kuraray Medical Inc., Sakazu, Kurashiki, Okayama, Japan</td>
<td>Mixture of 3-trimethoxysilylpropyl methacrylate (&lt;5 wt.%), ethanol (&gt;80 wt.%) and 10-Methacryloyloxydecyl dihydrogen phosphate</td>
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<td>Material</td>
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<tr>
<td>Resin Cements</td>
<td>Adhesive-based resin cement system</td>
<td>Multilink Primer A/B /Ivoclar Vivadent, Liechtenstein</td>
<td>Two Bottle self-etching adhesive. Multilink Primer A: water (85.7 wt.%) and initiators (14.3 wt%) Multilink Primer B: Phosphonic acid acrylate (48.1 wt.%), HEMA (48.1 wt.%), Methacrylate mod. polyacrylic acid (3.8 wt.%) and Stabilizers (&lt;0.02 wt.%)</td>
<td>Multilink Primer A: P63825 Multilink Primer B: P65434</td>
</tr>
</tbody>
</table>
| Adhesive-based resin cement | Self-etching bonding agent | Multilink Automix/ Ivoclar Vivadent, Liechtenstein | It consists of two pastes: Base paste: Dimethacrylate and HEMA (30.5 wt.%), Barium glass filler and Silica filler (45.5 wt.%), Ytterbiumtrifluoride (23 wt.%), Catalysts and Stabilizers (1 wt.%), Pigments (<0.01 wt.%)
Catalyst paste: Dimethacrylate and HEMA (30.2 wt.%), Barium glass filler and Silica filler (45.5 wt.%), Ytterbiumtrifluoride (23 wt.%), Catalysts and Stabilizers (1.3 wt.%) (Filler=45.5 wt%; avg. = 0.9 μm) | Multilink Cement: P67924 |
<p>| Silane coupling agent    | Monobond Plus primer /Ivoclar Vivadent, Liechtenstein | Solution of adhesive monomers (4 wt.%) and ethanol (96 wt.%), the adhesive monomers combine three different functional groups – silane methacrylate, phosphoric acid methacrylate and sulfide methacrylate. | Primer: P59584 |</p>
<table>
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<th>Material</th>
<th>Classification</th>
<th>Brand / Manufacturer</th>
<th>Composition</th>
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<td>Resin Cement</td>
<td>Adhesive-based resin cementsystem</td>
<td>Adhesive</td>
<td>Acid etch</td>
<td>Scotchbond&lt;sup&gt;TM&lt;/sup&gt; Phosphoric Etchant / 3M ESPE, St Paul, MN, USA</td>
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<td>Bonding agent</td>
<td>Adhesive</td>
<td>Bonding agent</td>
<td>Adper&lt;sup&gt;TM&lt;/sup&gt; Single Bond Plus / 3M ESPE, St Paul, MN, USA</td>
</tr>
<tr>
<td>Adhesive-based resin cement</td>
<td></td>
<td></td>
<td>RelyX&lt;sup&gt;TM&lt;/sup&gt; ARC / 3M ESPE, St Paul, MN, USA</td>
<td>It is composed of BisGMA and TEGDMA polymer, it consists of two pastes: Paste A: zirconia/silica filler(68 wt.%), pigments, amine and photo initiator system. Paste B: zirconia/silica filler (67 wt.%), BPO activator (Filler=67.5 wt.%; avg. = 1.5 μm)</td>
</tr>
<tr>
<td>Silane coupling agent</td>
<td></td>
<td></td>
<td>RelyX Ceramic primer / 3M ESPE, St Paul, MN, USA</td>
<td>Stable solution of a prehydrolyzed silane-coupling agent, alcohol and water.</td>
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<td>Material</td>
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<td></td>
<td>Mechanical Surface treatment material</td>
<td>Grit-etching Aluminum Oxide-50 Micron/ Danville Engineering Inc., California</td>
<td>50 microns aluminum oxide powder.</td>
<td>25671</td>
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<td></td>
<td>Acid etching</td>
<td>IPS Ceramic Etching Gel /Ivoclar Vivadent, Liechtenstein</td>
<td>The etchant is &lt;5% hydrofluoric acid. It has a pH of approximately 2</td>
<td>P72319</td>
</tr>
<tr>
<td></td>
<td>Composite filling material</td>
<td>Composite resin Z100™ Restorative /3M ESPE, St Paul, MN, USA</td>
<td>The matrix of this composite consists of BisGMA and TEGDMA polymer. The filler is zirconia/silica. The inorganic filler loading is 66% by volume with a particle size range of 3.5 to 0.01 micron.</td>
<td>N225470</td>
</tr>
</tbody>
</table>

Abbreviations: BIS-GMA (bisphenol a diglycidyl ether dimethacrylate), BPO (benzoyl peroxide), HEMA (hydroxyethyl methacrylate), MDP (methacryloyloxydecyl dihydrogen phosphate), and TEGDMA (tetraethyleneglycol dimethacrylate).
Figure 24: Study groups, the total number of specimens are 240 specimens (10 per group x 24 group)
3.2.2.1 Specimen Preparation:

- Preparing IPS e.max CAD and Paradigm™ MZ100 specimens:

IPS e.max CAD Blocks (Ivoclar Vivadent, Liechtenstein) and Paradigm™ MZ100 Blocks (Ivoclar Vivadent, Liechtenstein)(Figure 2) were cut perpendicular to the long axis of the block with a low speed diamond saw (Isomet, Buehler, Lake Bluff, IL) under copious water to expose flat freshly-cut material surface. Each block was then cut perpendicular to the flat surface into slabs of 1mm thickness using the same saw. The block of slabs was then rotated 90° and again cut perpendicular to the surface. IPS e.max CAD and Paradigm™ MZ100 microbars of cross sectional area $1\text{mm}^2 \pm 0.1 \text{mm}^2$ and 5-6 mm in length (Figure 26), were separated to be utilized for microshear bond strength test. The IPS e.max CAD microbars were crystallized in a ceramic furnace (EP 600 Combi, Ivoclar Vivadent, Liechtenstein) according to the manufacturer instructions.

![Figure 25: Crown materials, IPS e.max CAD (left), Paradigm MZ100 (right).](image-url)
Figure 26: Sectioning Paradigm MZ100 crown material in a diamond cutting saw (Isomet-top), Microbars(bottom): 1: IPS e.max CAD, 2: Paradigm MZ100.

The IPS e.max CAD microbars and Paradigm™ MZ100 microbars were embedded in a plastic rubbery base to expose the surface only for the surface treatment procedure, 50 μm aluminum oxide particles (Figure 27) were used for grit-etching the microbars using an air abrasion device (Microetcher ERC, Danville Engineering Inc., California)(Figure 28) from a distance of approximately 10 mm for 15 seconds under 2 bar pressure at the nozzle. The
microbars were then cleaned with compressed air for 30 seconds and then further cleaned in distilled water in an ultrasonic cleaner (FS5 Fisher Scientific, Sheboygan, Wisconsin, United States) for 5 minutes and air-dried. Another group of IPS e.max CAD microbars was treated with 5 % hydrofluoric acid (IPS Ceramic Etching Gel, Ivoclar Vivadent, Liechtenstein)(Figure 5) for 20 seconds, washed thoroughly for 1 min under tap water and air-dried. Then, the grit-etched lithium disilicate ceramic (IPS e.max CAD GE), the hydrofluoric acid etched lithium disilicate ceramic (IPS e.max CAD HF) and the grit-etched composite resin (Paradigm MZ100) were chemically treated with silane coupling agent presented in Table 9 according to manufacturer instructions presented in Table 10. The surface that was utilized in adhesion and exposed to surface treatment is the surface that was cut by the low speed saw.

Figure 27: Surface treatment materials, Aluminum oxide 50 micron powder (left), IPS Ceramic etching gel (right)
Dentin substrate:

Intact caries-free human molars were collected and sterilized with gamma radiation (Gamma cell 220, Atomic Energy Ltd, Mississauga, Canada) at the Department of Chemical Engineering and Applied Chemistry, University of Toronto. Teeth were placed in a glass jar that was placed in a cobalt chamber 5.5x8 inches, and exposed for 4 hours. The radiation dose rate was 0.3 KGY/h. This method of sterilization has been proved to not alter the tooth tissue mechanical or the physical properties. Teeth were kept, wherever possible, in distilled water in a refrigerator during and between all experimental procedures, in order to preserve their optimum mechanical and physical properties. Roots were then embedded in chemically cured acrylic resin bases (Ivolen, Ivoclar Vivadent, Liechtenstein, Germany) to facilitate handling during cutting procedures.

The crown was cut perpendicular to the long axis at approximately 4 mm from the cemento-enamel junction with a low speed diamond saw (Isomet, Buehler, Lake Bluff, IL)
under copious water to expose flat dentin surfaces. If needed, the surfaces were further ground with the same saw until complete removal of enamel was achieved. This flat coronal surface of dentin was further ground using a wheel diamond bur (#2909 314 040, Brassler, Komet, USA) similar to the one used in crown preparation to obtain a clinically relevant surface roughness. A tooth slice was obtained by a second cut parallel to the first one, approximately 2 mm towards the pulp chamber. The slice was embedded in self-cured acrylic resin circular blocks (Ivolen, Ivoclar Vivadent, Liechtenstein, Germany) with coronal dentin facing upwards, on which the bonding procedure was performed.

- **Composite Z100™ Restorative substrate:**

  Z100™ Restorative Composite (3M ESPE, St Paul, MN, USA) was used to make composite block in 2 mm increments. A Demi LED (light-emitting-diode) light polymerization unit (Kerr Corporation, Middleton, WI, USA, 1100-1200 mW/cm²) was used for light-curing each increment for 40 seconds. The surface layer of the block was cut using the low speed-cutting saw under copious water to produce a flat composite resin surface.

  The block was cut parallel to the flat composite surface into slices of 2 mm thickness using the low speed cutting saw under copious water. For every slice, the flat surface to be utilized in adhesion was further ground using a wheel diamond bur (#2909 314 040, Brassler, Komet, USA) to obtain standardized roughness. The slices were embedded in self-cured acrylic resin circular blocks (Ivolen, Ivoclar Vivadent, Liechtenstein, Germany) on which the bonding procedure was performed.
3.2.2.2 Bonding procedure:

Dimensions of microbars to be used for bonding were measured using a digital caliper (Mastercraft, Toronto, Canada) and the surface area was calculated. The microbars were randomly distributed over the groups. Each microbar was placed in the sleeve of the attachment device to standardize the vertical position of the microbars and trial placement of the microbar on the slice was performed prior to every bonding procedure (Figure 29). Once the position of the rod on the substrate slice was satisfactory, bonding procedures were started. For the groups to be bonded using RelyX ARC cement, the surface was etched with phosphoric acid and bonding agent was applied according to the manufacturer instructions presented in Table 3. For the groups to be bonded using Multilink cement, the surface was treated by self-etching multilink primer according to the manufacturer instructions presented in Table 3.

Four cements were used for the cementation procedures; each cement was mixed according to the manufacturer instructions (Table 3) and applied to the microbar specimens using microbrushes. The microbars were pressed against the underlying substrate slice using a fixed pressure (146 gram) applied on the attachment device to reach a minimal film thickness (Figure 29). Excess resin was removed from around the rods using microbrushes. Luting resins were polymerized in 20 seconds stages in two opposite sides of the specimens using a Demi LED (light-emitting-diode) light polymerization unit (Kerr Corporation, Middleton, WI, USA, 1100-1200 mW/cm²).
Figure 29: Attachment device (top), specimen pressed over the substrate (middle), bonded specimen (bottom)
3.2.2.3 **Thermocycling:**

Bonded specimens were stored in distilled water at 37°C for 24 hours. They were then subjected to artificial thermal ageing (Thermocycling procedure) according to the ISO (The International Organization for Standardization, ISO TR 11450 standard, 1994) recommendations. Thermocycles were performed using a dwell time of 30 seconds in each bath and a transfer time of 15 seconds between baths for 500 cycles between 5°C and 55°C. Then specimens were subjected to the bond testing procedures immediately after thermocycling.

3.2.2.4 **Microshear bond strength testing:**

The specimens were inspected for the interface quality (i.e., no voids or bubbles at the interface). The specimens, which were mounted in a self-cured acrylic circular block, were fitted in the clamp of the microshear testing machine (BISCO Microshear Tester, BISCO Inc.) (Figure 30). The blade of the machine was modified using self-cured acrylic resin to make the blade flat in shape to fit the bonded ceramic and composite rods. Mechanical loading with speed of 0.5 mm/min was applied. The bonded area was calculated and the shear bond strength was expressed in MPa.
Figure 30: Microshear bond strength testing; specimen fitted in the clamp of the microshear testing machine (top), specimen loaded in shear till failure (bottom)
3.2.2.5 Evaluation of the mode of failure:

A single operator inspected the two halves of each specimen under a stereomicroscope (Wild/Leica M3Z Stereo Microscope, Heerbrugg, Switzerland) at 40x magnification to fully assess the quality of the interfacial bonding. The modes of failure were assessed according to the following scheme:

1= Adhesive (crown material/cement interface): when at least 75% of the cement layer exists on the substrate material (dentin or composite)

2= Adhesive failure (substrate/cement interface): when at least 75% of the cement layer exists on the crown material (IPS e.max CAD or Paradigm MZ100)

3= Mixed: when adhesive failure occurs at both interfaces, the cement layer exists on less than 75 % of the crown material and substrate.

4= Cohesive failure of the crown material

5= Cohesive failure of the substrate (dentin or composite)

6= Mixed cohesive/adhesive: when adhesive failure is accompanied with any cohesive failure.

3.2.2.6 Statistical analysis:

Statistical analysis was computed using SPSS (PC+ version 20 software, Chicago, IL, USA). Data were presented as mean and standard deviation values of the microshear bond strength(μSBS). This data is quantitative (continuous) data. Statistical analysis was carried out using Factorial ANOVA. The significance level was set at P ≤ 0.05 and 95 % confidence level. Factorial analysis of variance was conducted to explore the impact of CAD/CAM material, type of cement and substrate on μSBS. CAD/CAM materials were divided into three groups according to their material /surface treatment combination: Group 1: IPS e.max CAD GE;
Group 2: IPS e.max HF; Group 3: Paradigm MZ 100. Types of cement were divided into four groups: Group 1: Rely X Unicem; Group 2: Clearfil SA; Group 3: Multilink Automix; Group 4: Rely X ARC. Substrates were divided into two groups: Group 1: Dentin; Group 2: Composite resin.
4. Results

The results of factorial ANOVA are presented in Table 10. All the investigated main effects were statistically significant and all the interactions were not statistically significant.

<table>
<thead>
<tr>
<th>Source</th>
<th>Type III Sum of Squares</th>
<th>df</th>
<th>Mean Square</th>
<th>F</th>
<th>Sig.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Corrected Model</td>
<td>303.200a</td>
<td>23</td>
<td>13.183</td>
<td>10.603</td>
<td>0.000</td>
</tr>
<tr>
<td>Intercept</td>
<td>12518.904</td>
<td>1</td>
<td>12518.904</td>
<td>10068.979</td>
<td>0.000</td>
</tr>
<tr>
<td>CAD/CAM material</td>
<td>50.827</td>
<td>2</td>
<td>25.414</td>
<td>20.44</td>
<td>0.000</td>
</tr>
<tr>
<td>Substrate</td>
<td>20.91</td>
<td>1</td>
<td>20.91</td>
<td>16.818</td>
<td>0.000</td>
</tr>
<tr>
<td>Type of Cement</td>
<td>210.259</td>
<td>3</td>
<td>70.086</td>
<td>56.37</td>
<td>0.000</td>
</tr>
<tr>
<td>CAD/CAM material * Substrate</td>
<td>0.786</td>
<td>2</td>
<td>0.393</td>
<td>0.316</td>
<td>0.729</td>
</tr>
<tr>
<td>CAD/CAM material* Type of Cement</td>
<td>15.412</td>
<td>6</td>
<td>2.569</td>
<td>2.066</td>
<td>0.058</td>
</tr>
<tr>
<td>Substrate * Type of Cement</td>
<td>0.176</td>
<td>3</td>
<td>0.059</td>
<td>0.047</td>
<td>0.986</td>
</tr>
<tr>
<td>CAD/CAM material * Substrate * Type of Cement</td>
<td>4.831</td>
<td>6</td>
<td>0.805</td>
<td>0.648</td>
<td>0.692</td>
</tr>
<tr>
<td>Error</td>
<td>268.556</td>
<td>216</td>
<td>1.243</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>13090.66</td>
<td>240</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Corrected Total</td>
<td>571.756</td>
<td>239</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

a. R Squared = .530 (Adjusted R Squared = .480)
4.1 Comparison between resin cements

Following detection of a significant main effect without significant interaction in factorial ANOVA Table 10, comparisons between the types of cements were carried without the levels of the other independent factors. The follow-up analysis to factorial ANOVA was carried out using one-way analysis of variance between-groups (one-way ANOVA) to explore the effect of the type of cement on μSBS. One-way ANOVA showed significance at $p<0.001$. The results of the one-way ANOVA are presented in Table 11. Post-hoc comparisons were carried out using the Tukey’s test; the significance level was set at $P \leq 0.05$ and 95% confidence level. The means and the standard deviation (SD) values are presented in Table 12. Homogenous subsets for Tukey’s test are presented in Table 13.

Table 11: One way ANOVA (Dependent variable: Bond strength; Independent variable: type of cement)

<table>
<thead>
<tr>
<th></th>
<th>Sum of Squares</th>
<th>df</th>
<th>Mean Square</th>
<th>F</th>
<th>Sig.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Between Groups</td>
<td>410.171</td>
<td>3</td>
<td>136.724</td>
<td>56.145</td>
<td>.000</td>
</tr>
<tr>
<td>Within Groups</td>
<td>87.667</td>
<td>36</td>
<td>2.435</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>571.756</td>
<td>239</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 12: Descriptive showing mean and standard deviation and the pairwise comparison of the type of cements

<table>
<thead>
<tr>
<th>Type of cement</th>
<th>N</th>
<th>Mean(SD) (MPa) *</th>
<th>95% Confidence Interval for Mean</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rely X Unicem</td>
<td>60</td>
<td>6.53 (1.17) c</td>
<td>6.23 - 6.83</td>
</tr>
<tr>
<td>Clearfil SA</td>
<td>60</td>
<td>6.25 (1.17) b</td>
<td>5.95 - 6.55</td>
</tr>
<tr>
<td>Multilink</td>
<td>60</td>
<td>7.45 (1.24) b</td>
<td>7.12 - 7.77</td>
</tr>
<tr>
<td>Rely X ARC</td>
<td>60</td>
<td>8.65 (1.34) a</td>
<td>8.30 - 8.99</td>
</tr>
</tbody>
</table>

*: Groups with different letters are statistically significant different according to Tukey’s test.
Table 13: Means for Cement groups in homogeneous subsets are displayed according to Tukey HSD

<table>
<thead>
<tr>
<th>Type of Cement</th>
<th>N</th>
<th>Subset for alpha = 0.05</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>1</td>
</tr>
<tr>
<td>Clearfil Sa</td>
<td>60</td>
<td>6.2533</td>
</tr>
<tr>
<td>Rely X Unicem</td>
<td>60</td>
<td>6.5350</td>
</tr>
<tr>
<td>Multilink</td>
<td>60</td>
<td>7.4500</td>
</tr>
<tr>
<td>Rely X ARC</td>
<td>60</td>
<td>8.6510</td>
</tr>
<tr>
<td>Sig.</td>
<td></td>
<td>.598</td>
</tr>
</tbody>
</table>

Post-hoc comparisons using the Tukey’s test indicated that etch-and-rinse adhesive-based resin cement (Rely X ARC) had the highest mean µSBS (p<0.001). This was followed by self-etch adhesive-based resin cement (Multilink) (p<0.001). There was no statistically significant difference between self-adhesive resin cement (Rely X Unicem) and self-adhesive resin cement (Clearfil SA) (p=0.598), which showed the lowest mean µSBS. The means and standard deviation (SD) values are presented in Figure 31.
Figure 31: Bar chart of the mean values of microshear bond strength (µSBS) for different types of cements. The error bars represent standard deviation. Groups with different letters show a statistically significant difference according to Tukey's test results.
4.2 **Comparison between Substrates:**

Following detection of a significant main effect without significant interaction in factorial ANOVA Table 10, comparison between substrates was carried without the levels of the other independent factors. The follow-up analysis to a significant interaction in factorial ANOVA was carried out using independent sample t-test. The means and the standard deviation (SD) values are presented in Table 14. The results of the independent sample t-test are presented in Table 15. The significance level was set at \( P \leq 0.05 \) and 95% confidence level.

**Table 14:** Descriptive analysis showing mean and standard deviation and the pairwise comparison of the type of cements

<table>
<thead>
<tr>
<th>Bond Strength</th>
<th>Substrate</th>
<th>N</th>
<th>Mean (MPa)*</th>
<th>Std. Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dentin</td>
<td>120</td>
<td>6.9272(^{(b)})</td>
<td>1.57612</td>
<td></td>
</tr>
<tr>
<td>Composite resin</td>
<td>120</td>
<td>7.5175(^{(a)})</td>
<td>1.46452</td>
<td></td>
</tr>
</tbody>
</table>

*: Groups with different letters are statistically significant different.

**Table 15:** Independent Samples Test (Dependent variable: Bond strength; Independent variable: type of the substrate)

<table>
<thead>
<tr>
<th>Levene's Test for Equality of Variances</th>
<th>BondStrength</th>
</tr>
</thead>
<tbody>
<tr>
<td>Equal variances assumed</td>
<td>Equal variances not assumed</td>
</tr>
<tr>
<td>F</td>
<td>.793</td>
</tr>
<tr>
<td>Sig.</td>
<td>.374</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>t-test for Equality of Means</th>
<th>BondStrength</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Equal variances assumed</td>
</tr>
<tr>
<td>t</td>
<td>-3.006</td>
</tr>
<tr>
<td>df</td>
<td>238</td>
</tr>
<tr>
<td>Sig. (2-tailed)</td>
<td>.003</td>
</tr>
<tr>
<td>Mean Difference</td>
<td>-.59033</td>
</tr>
<tr>
<td>Std. Error Difference</td>
<td>.19640</td>
</tr>
<tr>
<td>95% Confidence Interval of the Difference</td>
<td>Lower</td>
</tr>
<tr>
<td></td>
<td>Upper</td>
</tr>
</tbody>
</table>
T-test for independent samples indicated that composite resin substrate showed the highest mean µSBS (p=0.003). The means and the standard deviation (SD) values are presented in Figure 32.

Figure 32: Bar chart of the mean values of µSBS for different Substrates. The error bars represent standard deviation. Groups with different letters show a statistically significant difference according to Tukey’s test results.
4.3 Comparison between CAD/CAM materials

Following detection of a significant main effect without significant interaction in factorial ANOVA Table 10, comparison between CAD/CAM materials was carried without the levels of the other independent factors. The follow-up analysis to factorial ANOVA was carried out using one-way analysis of variance between-groups (one-way ANOVA) to explore the effect of the CAD/CAM material on µSBS. One-way ANOVA showed significance at \( p<0.001 \). The results of the one-way ANOVA are presented in Table 16. Post-hoc comparisons were carried using the Tukey’s test; the significance level was set at \( P \leq 0.05 \) and 95% confidence level. The means and the standard deviation (SD) values are presented in Table 17. Homogenous subsets for Tukey’s test are presented in Table 18.

<table>
<thead>
<tr>
<th>Table 16: One way ANOVA (Dependent variable: Bond strength; Independent variable: CAD/CAM material)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sum of Squares</td>
</tr>
<tr>
<td>Between Groups</td>
</tr>
<tr>
<td>Within Groups</td>
</tr>
<tr>
<td>Total</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Table 17: Descriptive showing mean and standard deviation and the pairwise comparison of the type of cements</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type of cement</td>
</tr>
<tr>
<td>IPS e.max CAD GE</td>
</tr>
<tr>
<td>IPS e.max CAD HF</td>
</tr>
<tr>
<td>Paradigm MZ100</td>
</tr>
</tbody>
</table>

*: Significant at \( P \leq 0.05 \), Groups with different letters are statistically significant different according Tukey's test results.
Table 18: Means for Cement groups in homogeneous subsets are displayed according to Tukey HSD

<table>
<thead>
<tr>
<th>CAD/CAM material</th>
<th>N</th>
<th>Subset for alpha = 0.05</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>IPS e.max CAD GE</td>
<td>80</td>
<td>6.5737</td>
</tr>
<tr>
<td>IPS e.max CAD HF</td>
<td>80</td>
<td>7.5000</td>
</tr>
<tr>
<td>Paradigm MZ100</td>
<td>80</td>
<td>7.5932</td>
</tr>
<tr>
<td>Sig.</td>
<td>1.000</td>
<td>.917</td>
</tr>
</tbody>
</table>

Post-hoc comparisons using the Tukey’s test indicated that there was no statistically significant difference between hydrofluoric acid etched lithium disilicate glass ceramic (IPS e.max CAD HF) and composite resin CAD/CAM material (Paradigm MZ100) (p=0.917), which showed the highest $\mu$SBS. This was followed by IPS e.max CAD GE, which showed the lowest $\mu$SBS (p<0.001). The means and standard deviation (SD) values are presented in Figure 33.
Figure 33: Bar chart of the mean values of microshear bond strength (µSBS) for different CAD/CAM materials. The error bars represent standard deviation. Groups with different letters show a statistically significant difference according to Tukey’s test results.

4.4 Modes of failure

The distribution of the modes of failure in the specimens of the microshear test is shown in Figure 34, the majority of the failures were adhesive in nature at substrate interface 53.75%, followed by adhesive failure at crown material interface 35%, and finally mixed failures 11.25%.
Figure 34: Pie chart showing distribution of the modes of failure in the microshear bond strength (μSBS) specimens.
5. Discussion

The microshear bond strength test has been introduced to evaluate adhesion of dental restorations to the underlying tooth substrate as an alternative to conventional shear bond strength testing, sharing with the microtensile bond strength testing the advantages of the “micro” tests. Microshear test is capable of better evaluation of the bond strength at adhesive interfaces compared to conventional shear test as a consequence to the small surface area specimens with decreased amount of defects and critical size flaws, and to the better stress distribution at the adhesive interface. Also, in microshear test, more specimens were produced per tooth and per restoration, which is advantage over conventional shear test. The main advantage of microshear test over the microtensile test (conventional methodology) is that no sectioning of the bonded specimen is needed, thus avoiding the undue stresses that are introduced to the microtensile specimens (conventional methodology) that lead to pretest failures and may also affect the final bond strength results. On the other hand, in the current methodology for microshear testing, only one adhesive interface can be examined, where the cement is bonded directly to the tooth substrate and a blade is applied to the interface inducing shearing forces. In this study we modified the conventional microshear testing methodology to involve two adhesive interfaces where the crown material is bonded to the underlying substrate using resin cement in-between, thus creating a more clinically relevant situation.

Thermocycling was used in this study as a clinically relevant aging methodology. Several studies have mentioned that 10,000 thermocycles correspond to one year of in-vivo serviceability, which means that 500 cycles correspond to 18 day of in-vivo serviceability, thus 500 cycles is considered to be minimal aging effect. However, thermocycling for 500 cycles between 5 °C and 55 °C is in agreement with the International Organization for
Standardization, ISO TR 11450 standard, 1994, which considers thermocycling protocol as an appropriate aging methodology for adhesion testing.

Comparing different cements, etch-and-rinse adhesive-based resin cement (Rely X ARC) showed the highest statistically significant µSBS. Self-etch adhesive-based resin cement (Multilink cement) ranked second regarding µSBS. Self-adhesive resin cements (Rely X Unicem and Clearfil SA) were third rank with the lowest bond strength in all groups. These results are in agreement with Ozcan et al (2012) who found that etch-and-rinse adhesive-based resin cements have higher shear bond strength to dentin compared to self-etching adhesive resin cements. Also, in agreement with our findings, Zhang et al (2010) found that etch-and-rinse adhesive-based resin cements and self-etch adhesive-based resin cement systems were capable of achieving higher shear bond strength compared with self-adhesive resin cements, when used to bond lithium disilicate ceramic restorative material and composite resin restorative material to dentin. The bonding performance of etch-and-rinse adhesive-based resin cements with dentin substrate can be explained by the better etching ability of the etch-and-rinse system that causes microporosities within the intertubular dentin, exposes collagen fibrils and removes the smear layer. Also, the bonding performance of etch-and-rinse adhesive-based resin cement with composite substrate could be related to the more hydrolytically stable chemical composition of this cement in relation to other cements included in this study, since high concentrations of acidic resin monomers can make self-etch adhesives behave like hydrophilic permeable membranes and mediate hydrolytic reactions at the adhesive interface. Also, the residual solvent (water) that remains within the adhesive interface can weaken the bond. Moreover, self-etching adhesive based resin cements are only capable of dissolving the smear layer without removing it, thus calcium phosphates of the smear layer become entrapped in the
interfacial zone between the self-etch adhesive based resin cement and dentin and negatively affect the bond strength. \(^{20}\) This could explain the lower \(\mu\)SBS performance of self-etching adhesive-based resin cement (Multilink Automix) in relation to etch-and-rinse adhesive-based resin cement (Rely X ARC).

Self-etching adhesive-based resin cement (Multilink Automix) presented higher microshear bond strength values compared to the two self-adhesive resin cements Rely X Unicem and Clearfil SA. This could be explained by the incomplete neutralization of the acidic groups in self-adhesive resin cements, which negatively affects the mechanical properties of these cements as a result of interfering with the polymerization reaction initiators causing lower degree of polymerization. \(^{5,204,205}\) Also, it is well known that self-adhesive resin cements are very weak in etching and infiltrating dentin with no hybrid layer formation, \(^{110,137}\) while self-etching adhesive-based resin cements are capable of etching the dentin and forming hybrid layer. \(^{218}\)

Comparing different substrates, composite resin showed the highest mean microshear bond strength, while dentin showed the lowest mean microshear bond strength. This could be related to the chemical bonding between unreacted monomers of the resin cement and that of the composite resin substrate which has a 74% degree of conversion. \(^{210}\) It has been reported by Kallio et al (2001) that chemical covalent bonding could occur between unsaturated double bonds of composite resin substrate and repair resin or intermediary resin. \(^{209}\)

Comparing different crown materials, there was no statistically significant difference between IPS e.max CAD HF etched and Paradigm MZ100, where both showed the highest mean \(\mu\)SBS, while IPS e.max CAD GE showed the lowest mean \(\mu\)SBS. This could be explained by the weak irregularities created at the surface of the ceramic when grit-etching is employed that compromises the mechanical interlocking to this surface, \(^{197}\) resulting in lower
bond strength values for the IPS e.max CAD GE. De Menezes et al demonstrated that hydrofluoric acid etching of lithium disilicate ceramic rendered the surface rougher and better for micromechanical retention as opposed to grit-etching of the same ceramic.\textsuperscript{197}

The modes of failure evaluated in this study were mostly adhesive failure at the substrate/cement interface 53.75\%, followed by adhesive failure at the crown material/cement interface 35\%, and finally mixed failures 11.25\%. This indicates that the stresses were concentrated at the adhesive interface with no cohesive failures. Cohesive failures are known to preclude measurement of the interfacial bond strength.\textsuperscript{16} This is in agreement with Salz et al (2010) who mentioned that below 2mm\textsuperscript{2} of adhesive area, all specimens fail adhesively at the interface.\textsuperscript{151} The modes of failure for the microtensile test mentioned in chapter 3 of this thesis were mainly adhesive at the crown material/cement interface, while for the microshear test they were primarily adhesive failures at the substrate/cement interface. This could be related to the difference in stress distribution for every test because of the different test settings and the difference in the direction of loading.
6. Limitations of the study

This study has the following limitations:

1. As an in-vitro study, it helps in primary investigation of dental biomaterials bonding properties but can’t substitute in-vivo studies.

2. Thermocycling in a water bath between 5 °C and 55 °C for 500 can be considered of minimal aging effect.\textsuperscript{213,214}\textsuperscript{213,214} However, the ISO TR 11405 (1994) states that this thermocycling protocol is recommended for aging dental interfaces prior to adhesion testing.\textsuperscript{215}\textsuperscript{215}

3. This study was performed using the new microshear specimen preparation methodology while the conventional microshear specimen preparation methodology was not performed in this study as it tests only one adhesive interface, which limits direct comparison of both methodologies.
7. Conclusions

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

- Etch-and-rinse adhesive resin cements are capable of achieving the highest $\mu$SBS between CAD/CAM materials and different substrates.
- Self-adhesive resin cements showed the lowest $\mu$SBS between CAD/CAM materials and different substrates.
- Composite resin CAD/CAM materials and hydrofluoric acid etched lithium disilicate ceramic showed similar $\mu$SBS to different substrates.
- Composite substrate showed better bonding performance to CAD/CAM materials as opposed to dentin substrate.
- Hydrofluoric acid etching of lithium disilicate glass ceramic resulted in better bond strength to different substrates than grit-etching of lithium disilicate glass ceramic.
Chapter 5 Retention Of CAD/CAM Crowns Using Self-adhesive Resin Cement
1. Abstract

Objectives: To determine retention strength (RS) of etch-and-rinse and self-adhesive resin cements when used to bond IP-e.max-CAD and Lava-Ultimate crowns to different substrates: dentin, amalgam and composite. Methods: Ninety-six freshly extracted molars were prepared to receive CAD/CAM crowns using special paralleling-device. After occlusal reduction, molars were divided into three equal groups. One control group with no restorations, second restored with mesio-occluso-distal amalgam (Permite-C, SDI Inc.) and third restored with mesio-occluso-distal composite (Z100-restorative, 3M-ESPE). After crown preparations teeth were scanned in 3d scanner (3shape-D810, 3shape) to determine surface area. Each group was further divided into 4 equal subgroups. IPS- e.max-CAD (EMX) crowns were made for teeth in two subgroups while composite crowns (Lava Ultimate) (LU) were made for the other two subgroups. Crowns were milled using CEREC-3D system. Crowns of one EMX subgroup and one LU subgroup were cemented to their respective teeth using (RelyX-Ultimate) (RXL). Crowns of other two subgroups were cemented with (Rely-Unicem-2) (RXU). Crowns were then subjected to compressive cyclic-loading (500,000 cycle, 80N, 1mm vertical, 1mm horizontal, in water 37°C, 2Hz) in chewing-simulator. Crowns were then pulled away from teeth in Instron universal testing machine at cross head speed 0.5 mm/min. RS was calculated and means and SDs determined. Data were statistically analyzed with multi-factorial ANOVA.

Results: ANOVA revealed significant difference in mean RS among subgroups (P<0.05). Highest mean RS 3.88 (0.56) MPa was obtained when RXL was used to cement EMX crowns to specimens of control group, while lowest mean RS 1.92 (0.2) MPa was obtained when RXU was used to cement EMX crowns to specimens with amalgam fillings. Conclusions: Irrespective of substrate, use of RXL etch-and –rinse cement resulted in significantly higher
mean RS whereas use of RXU resulted in significantly lower mean RS. Mean RS for composite subgroups was higher than that of amalgam subgroups.

2. Introduction

The increasing demand for esthetics in the posterior region of the mouth and environmental concerns about restorations containing metals were behind the evolution of new techniques for fabrication of non-metallic posterior inlays, onlays, and crowns.219

The introduction of computer-aided design/computer-aided manufacturing (CAD/CAM) technology in dentistry enabled dentists to use new treatment modalities. CAD/CAM machines are now gaining popularity and are clinically proven.220 The clinical success of these indirect esthetic restorations depends in part on the adequate adhesion between the restoration and the underlying bonding substrate.220,221

Resin cement systems have been successfully used to achieve adequate adhesion between the indirect esthetic restorations and the underlying tooth substrate.61 Resin cements were divided into two subgroups according to the adhesive system utilized to prepare the tooth surface prior to cementation.61 One group used etch-and-rinse adhesive-based systems, while the other group used self-etching primers.61 Recently, self-adhesive resin cements were introduced as a new subgroup of resin cements and were designed to bond directly to the tooth structure without any pre-treatment to the tooth surface.105 Several studies have been performed to determine the efficiency of self-adhesive resin cements and to determine if they are a possible replacement for current conventional adhesive resin cement systems, results being controversial.63,104,105 All these studies tested adhesion of resin cements to tooth substrate, yet information about the effect of different substrates such as intact natural teeth and teeth with
composite or amalgam restorations on the retention of esthetic crowns are sparse in the literature.

Several factors have been described that influence the bond strength of the indirect esthetic restorations to the underlying bonding substrate. Some of these factors include the type of the restoration, the type of the resin cement and the type of the bonding substrate. Also, the outcomes of the bond strength testing showed variations with different methods of bond strength measurement.\textsuperscript{4,13,14}

In-vitro bond strength measurement tests provide immediate information on the bonding effectiveness of new materials. The most commonly used bond strength tests are tensile and shear tests. However, they were replaced recently by the micro tests, which allow the production of multiple specimens from the same tooth.\textsuperscript{172}

Currently, the most common approach in micro bond strength testing is to load multiple test specimens from each tooth in either a tensile (\(\mu\)TBS) or shear (\(\mu\)SBS) manner. However, in microtensile bond strength testing procedures, sectioning and trimming steps may introduce early micro cracking within the specimen and affect the recorded results, which may in turn affect the validity of the results. Also, in microtensile bond strength testing procedures, a large number of specimens show pre-test failures, and different studies used different statistical approaches to deal with these pre-test failures either by excluding them from the results or assigning them half of the lowest recorded bond strength value or assigning them a value of zero, which also may undermine the validity of the results. In microshear bond strength testing, the micro-cracking introduced during microtensile testing is avoided. However, highly non-uniform stress distribution at the interface occurs and flashes of excess resin cements may exist beyond the restoration which may affect the results.\textsuperscript{172,199}
In spite of the considerable advances regarding adhesion testing, shortcomings of present methods and the lack of an accepted standard protocol necessitate new adhesion testing procedures. Currently there is very little evidence reporting the amount of force necessary to remove a cemented crown. Lack of adequate retention can lead to the dislodgement, loss or fracture of the crown.\textsuperscript{151,183}

This in-vitro study was designed to evaluate the retention force of an etch-and-rinse and a self-adhesive resin cement system when used to bond IPS e.max CAD and Lava Ultimate crowns to sound natural teeth, natural teeth with amalgam filling and natural teeth with composite resin filling. Non-metallic crowns were gripped by a new technique and pulled away from the underlying teeth to measure their resistance to dislodgment.

3. Materials and Methods

3.1 Materials

All the material used in the study and their composition (as reported by manufacturers) are listed in Table 19.

3.2 Methods:

3.2.1 Pilot study:

The current study was preceded by an external pilot study to detect any problems with the proposed methods of testing, where 2 samples per group were used in the pilot study. Following the completion of the pilot study, 96 specimens were used for the main study.
3.2.2 Main study

Twelve groups were formed (n=8)(Figure 35). The number of specimens per group was calculated using web-based software® for sample size analysis at $\alpha=0.05$ and 80% power and effect size equal to 0.356 which yields a sample size of 5 samples per group. We used 8 samples per group for this study to have more power. The effect size ($f$) was calculated by using the following formula, developed by Cohen (1999).

$$f = d \times \frac{1}{2} \sqrt{\frac{k+1}{3(k-1)}}$$

Where $k$ is the number of groups and $d$ is the difference in means ($x_1-x_2$) divided by common standard deviation ($\sigma$). Based on values obtained from the pilot study, $x_1=3.04$, $x_2=2.45$, $\sigma=0.52$, the $d$ value was calculated ($d=1.134$), and $f$ was calculated (0.356).

® Power Analysis For ANOVA Designs [http://www.math.yorku.ca/SCS/Online/power/](http://www.math.yorku.ca/SCS/Online/power/)
Table 19: Material used in the study and their composition as reported by the manufacturer.

<table>
<thead>
<tr>
<th>Material</th>
<th>Classification</th>
<th>Brand / Manufacturer</th>
<th>Composition</th>
<th>Lot #</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lithium disilicate ceramic</td>
<td>CAD/CAM Materials</td>
<td>IPS e.max CAD/ Ivoclar Vivadent, Liechtenstein</td>
<td>SiO₂ (57 – 80 wt.%), Li₂O (11 – 19 wt.%), K₂O (0 – 13 wt.%), P₂O₅ (0 – 11 wt.%), ZrO₂ (0 – 8 wt.%), ZnO (0 – 8 wt.%), Other and coloring oxides (0-12 wt.)</td>
<td>P63946</td>
</tr>
<tr>
<td>Indirect composite resin</td>
<td>CAD/CAM Materials</td>
<td>Lava™ Ultimate/ 3M ESPE, St Paul, MN, USA</td>
<td>Composite material contains: Zirconia-silica nanocluster: Synthesized via a proprietary process from 20 nm silica particles and 4 to 11 nm zirconia particles; Nanomere particles: 20 nm silica nanomer particles and 4-11 nm zirconia nanomere particles The average nanocluster particle size is 0.6 to 10 micrometers. Resin matrix composition was not mentioned by the manufacturer Total filler loading is 80% wt.</td>
<td>N481731</td>
</tr>
<tr>
<td>Resin Cements</td>
<td>Self-adhesive resin cement system</td>
<td>RelyX™ Unicem 2/3M ESPE, St Paul, MN, USA</td>
<td>Self adhesive resin cement consists of two pastes: Base paste: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers, rheological additives. Catalyst paste: methacrylate monomers, alkaline (basic) fillers, silanated fillers, initiator components, stabilizers, pigments and rheological additives (Filler=50% vol., 72 wt.%; avg. &lt; 12.5 μm)</td>
<td>515337</td>
</tr>
<tr>
<td>---</td>
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<td>---</td>
</tr>
<tr>
<td>Silane coupling agent</td>
<td>Self-adhesive cement</td>
<td>RelyX Ceramic primer / 3M ESPE, St Paul, MN, USA</td>
<td>Stable solution of a prehydrolyzed silane-coupling agent, alcohol and water.</td>
<td>N470024</td>
</tr>
<tr>
<td>Resin Cement</td>
<td>Adhesive-based resin cement system</td>
<td>Description</td>
<td>Notes</td>
<td></td>
</tr>
<tr>
<td>--------------</td>
<td>----------------------------------</td>
<td>-------------</td>
<td>-------</td>
<td></td>
</tr>
<tr>
<td>Acid etch</td>
<td>Scotchbond™ Phosphoric Etchant / 3M ESPE, St Paul, MN, USA</td>
<td>The etchant is 35% phosphoric acid by weight. It has a pH of approximately 0.6</td>
<td>509513</td>
<td></td>
</tr>
<tr>
<td>Bonding agent</td>
<td>Scotchbond™ Universal Adhesive/ 3M ESPE, St Paul, MN, USA</td>
<td>One-bottle adhesive containing MDP Phosphate Monomer, Dimethacrylate resins, HEMA, Vitrebond™ Copolymer, Filler, ethanol, water, initiators and silane</td>
<td>514135</td>
<td></td>
</tr>
<tr>
<td>Adhesive-based resin cement</td>
<td>RelyX™ Ultimate/ 3M ESPE, St Paul, MN, USA</td>
<td>Consists of two pastes: Base paste: methacrylate monomers, radiopaque silanated fillers, initiator components, stabilizers, and rheological additives. Catalyst paste: methacrylate monomers, radiopaque alkaline (basic) fillers, initiator components, stabilizers, pigments, rheological additives, Fluorescence dye and Dark cure activator for Scotchbond Universal (Filler=43 vol.%; avg. = 13 μm)</td>
<td>514665</td>
<td></td>
</tr>
<tr>
<td>Silane coupling agent</td>
<td>RelyX Ceramic primer / 3M ESPE, St Paul, MN, USA</td>
<td>Stable solution of a prehydrolyzed silane-coupling agent, alcohol and water.</td>
<td>N470024</td>
<td></td>
</tr>
<tr>
<td>Material</td>
<td>Classification</td>
<td>Brand / Manufacturer</td>
<td>Composition</td>
<td>Lot #</td>
</tr>
<tr>
<td>----------</td>
<td>----------------</td>
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<td>-------------</td>
<td>------</td>
</tr>
<tr>
<td>Mechanical Surface treatment material</td>
<td>Grit-etching</td>
<td>Aluminum Oxide-50 Micron/ Danville Engineering Inc., California</td>
<td>50 microns aluminum oxide powder.</td>
<td>25671</td>
</tr>
<tr>
<td></td>
<td>Acid etching</td>
<td>IPS Ceramic Etching Gel /Ivoclar Vivadent, Liechtenstein</td>
<td>The etchant is &lt;5% hydrofluoric acid. It has a pH of approximately 2</td>
<td>P72319</td>
</tr>
<tr>
<td>Tooth filling material</td>
<td>Composite resin</td>
<td>Z100™ Restorative /3M ESPE, St Paul, MN, USA</td>
<td>The matrix of this composite consists of BisGMA and TEGDMA polymer. The filler is zirconia/silica. The inorganic filler loading is 66% by volume with a particle size range of 3.5 to 0.01 micron.</td>
<td>N464768</td>
</tr>
<tr>
<td></td>
<td>Amalgam</td>
<td>Permite / SDI Inc., Itasca, USA</td>
<td>Non-gamma two admix alloy consists of: Ag 56%, Sn 27.9%, Cu 15.4%, In 0.5%, Zn 0.2%, Hg 47.9% Alloy particles are spherical and lath cut.</td>
<td>110615102</td>
</tr>
</tbody>
</table>
Figure 35: Study groups, the total number of specimens is 96 (8 per group x 12 group)
3.2.2.1 Specimen collection and storage

Intact caries-free human molars were collected and cleaned with periodontal scalers and curettes, then sterilized with gamma radiation (Gamma cell 220, Atomic Energy Ltd, Mississauga, Canada) at the Department of Chemical Engineering and Applied Chemistry, University of Toronto. Teeth were placed in a cobalt chamber 5.5x8 inches, and exposed for 4 hours. The radiation dose rate was 0.3 KGy/h. This method of sterilization has been proved to not alter the mechanical or physical properties of the tooth tissue. Teeth were kept wet, wherever possible, in distilled water in a refrigerator during and between all experimental procedures, in order to preserve their optimum mechanical and physical properties.

3.2.2.2 Specimen preparation:

The dimensions of the teeth were measured mesio-distally and bucco-lingually at the cemento-enamel junction using a digital caliper (Mastercraft, Toronto, Canada) at approximately the mid-point of every surface. An average width for every tooth was calculated based on the bucco-lingual and the mesio-distal readings. Teeth were then divided into three groups based on size: small (less than 8mm), medium (8-10 mm) and large (more than 10mm). The teeth were randomly assigned to the study groups in such a way to have even distribution of different sizes of the teeth. Every group included two small molars, five medium-sized molars and one large molar (n=8). The roots of each tooth were notched and 4-5 restorative pins (TMS Link, Coltène/Whaledent Inc., Cuyahoga Falls, USA) were inserted horizontally into the roots. The occlusal surface of the teeth was attached to the vertical arm of a dental surveyor (Figure 36), using thermoplastic dental compound (Impression Compound, Kerr Inc., USA), so that the long axis of the tooth coincides with the vertical arm of the surveyor and the cemento-enamel
junction was parallel to the horizontal plane. Teeth were then mounted in acrylic resin (Ivolen, Ivoclar Vivadent, Liechtenstein, Germany), at a level of 2 mm apical to the cemento-enamel junction (CEJ), using a metal threaded cylinder creating a base. Teeth were then separated from the surveyor arm.

![Figure 36: Molar tooth inserted in acrylic resin inside the threaded metal base.](image)

Occlusal reduction of the teeth was carried out to a vertical height of $4 \pm 0.25$ mm using wheel diamond bur (#2909, Komet, USA) mounted in a high-speed air turbine handpiece with profuse water coolant. Specimens were then divided into three groups: a control consisting of unrestored teeth, a group with teeth having mesio-occluso-distal (M.O.D) amalgam restorations (3 mm width and 2 mm depth), and a group with teeth having similar M.O.D composite restorations. For teeth having M.O.D restorations, cavity preparation was done with tungsten carbide burs (#330, SS White, Great White Series, Lakewood, NJ, USA) in a high speed air turbine handpiece with copious water coolant and all the line angles were rounded to have a cavity with dimensions of $3 \text{mm} \pm 0.25 \text{ mm width}$
and 2 mm±0.25 depth. Bur was changed every eight-cavity preparations to ensure sharpness of the bur. For amalgam restorations, permite amalgam capsules were trititurated for 9 seconds in an amalgamator (VARI-MIX III, Dentsply, USA) and amalgam was condensed into the cavity with the aid of a tofflemire matrix retainer and band. For composite resin restoration, the teeth were restored with composite Z100 restorative in two increments, and each increment was light-cured for 40 seconds. Finishing of the restorations was done after 24 hours storage of the teeth in water before crown preparations.

Crown preparations were made with a 1 mm shoulder margin positioned just occlusal to the cement-enamel junction and 10± 2 degree axial wall convergence for every wall (20± 4degree total angle of convergence mesio-distally and 20± 4degree total angle of convergence bucco-lingually) using flat-end taper diamond bur (#847, Brassler, USA) in a high-speed air turbine handpiece with copious water coolant mounted in a parallelometer to standardize the taper of the preparation (Figure 37). Bur was changed every eight preparations to ensure good cutting efficiency. The degree of taper was verified using measuring software (Onde rulers v1.13.1, Ondesoft Computing Inc.) when optical impressions were subsequently taken by the CAD/CAM camera (Figure 40). Occlusal surface was finished to create a non-anatomical occlusal table. 3D scanning was performed using a 3D scanner (D810 3D Scanner, 3Shape Inc., New Jersey, USA) in order to calculate the teeth surface area (Figure 38).
Figure 37: a) Parallelogram used to prepare teeth, b) Prepared natural molar tooth, C) Prepared natural tooth with composite restoration, D) Prepared natural tooth with amalgam restoration.
Figure 38: Top: 3D scanner, Bottom: scanned tooth for surface area determination.

3.2.2.3 Impressions and restoration design

Optical reflective medium spray (IPS contrast spray, Ivoclar Vivadent, Liechtenstein) was used to cover the prepared teeth with a thin layer (Figure 39). An optical impression of the prepared tooth was captured using the CEREC 3D intraoral camera. CEREC software was used to design crowns. Each crown was made with anatomical features of lower second molar readily available on a computer program. Minimum occlusal thickness (at central fossa) was designed to be 1.5 mm. Thickness of crown at various points was verified with Boley gauge after milling.
Figure 39: Prepared tooth sprayed with the IPS contrast medium to be ready for optical impression.

Figure 40: Angle verification on the optical impression.
3.2.2.4 Crown fabrication

Forty-eight crowns were fabricated from each crown material: IPS e.max CAD and Lava Ultimate blocks, using the CEREC 3D milling unit (Figure 41). Cutting diamonds were changed after milling eight crowns. IPS e.max CAD crowns were crystallized in a ceramic furnace (EP 600 Combi, Ivoclar Vivadent, Liechtenstein) according to the manufacturer instructions (Table 2) (Figure 42).

Figure 41: a) Milling machine, b) IPS e.max CAD Block in the milling machine, C) Cerec 3 Machine.
Figure 42: IPS e.max Cad crowns in the EB600 Combi furnace.

Figure 43: Left: IPS e.max CAD crowns before crystallization, right: IPS e.max CAD crowns after crystallization.
3.2.2.5 Crowns surface treatment

The intaglio surfaces of the IPS e.max CAD crowns were etched with 5% hydrofluoric acid etching gel (IPS ceramic etching gel, Ivoclar Vivadent, Liechtenstein) for 20 sec then washed using water spray for 60 seconds, air-dried then silanated (RelyX ceramic primer, 3M ESPE). The intaglio surfaces of Lava Ultimate crowns were grit-etched with 50 µm aluminum oxide powder in an air abrasion device (Microetcher ERC, Danville Engineering Inc., California) under 2 bar pressure at the nozzle. They were then cleaned with compressed air-water spray for 30 seconds and further cleaned in distilled water in an ultrasonic cleaner for 5 minutes, air-dried then silanated (RelyX ceramic primer, 3M ESPE).

3.2.2.6 Cementation

All crowns were divided into two main groups, one group was cemented with etch-and-rinse adhesive cement (Rely X Ultimate, 3M ESPE), the other group was cemented with self-adhesive resin cement (Rely X Unicem 2, 3M ESPE). The cements were mixed and applied to the intaglio surfaces of the crowns according to the manufacturer's instructions(Table 20). The crowns were initially seated on their respective abutments using finger pressure, then the crowns were placed under a static load of 22 N for 5 minutes (Figure44), the excess cement was briefly light-cured then removed. After load removal, each crown was light-cured for 20 seconds on each surface.
**Table 20: Materials and sequence for their respective application (according to the manufacturer)**

<table>
<thead>
<tr>
<th></th>
<th>Etchant</th>
<th>Adhesive</th>
<th>Ceramic primer</th>
<th>Luting Resin Cement</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>RelyX Unicem 2</strong>&lt;br&gt; <em>(3M ESPE, St Paul, USA)</em></td>
<td>------</td>
<td>------</td>
<td>Apply RelyX™ adhesive primer to the roughened crown material and allow to dry for 5 seconds.</td>
<td>Dispense and mix the cement using the automix tips, then apply the cement in a thin layer on the bonding surfaces with micro brush, light cure for 2 seconds and remove excess, then light cure for 20 seconds per surface.</td>
</tr>
<tr>
<td></td>
<td><strong>Apply</strong> Scotchbond ™ Etchant for 15 seconds to the tooth substrate, water rinse for 10 seconds. Dry the surface with gentle air stream.</td>
<td><strong>Apply</strong> 2 consecutive coats of Scotchbond™ Universal Adhesive to the tooth substrate using microbrush and rub it in for 20 seconds, and then dry the surface with gentle air stream for 5 seconds. Then light-cure for 10 seconds.</td>
<td><strong>Apply</strong> RelyX™ ceramic primer to the roughened crown material and allow to dry for 5 seconds.</td>
<td>**Dispense and mix the cement using the automix tips, then apply the cement in a thin layer on the bonding surfaces with micro brush, light cure for 2 seconds and remove excess, then light cure for 20 seconds per surface.</td>
</tr>
<tr>
<td><strong>RelyX™ Ultimate</strong>&lt;br&gt; <em>(3M ESPE, St Paul, USA)</em></td>
<td><strong>Apply</strong> Scotchbond ™ Etchant for 15 seconds to the tooth substrate, water rinse for 10 seconds. Dry the surface with gentle air stream.</td>
<td><strong>Apply</strong> 2 consecutive coats of Scotchbond™ Universal Adhesive to the tooth substrate using microbrush and rub it in for 20 seconds, and then dry the surface with gentle air stream for 5 seconds. Then light-cure for 10 seconds.</td>
<td><strong>Apply</strong> RelyX™ ceramic primer to the roughened crown material and allow to dry for 5 seconds.</td>
<td>**Dispense and mix the cement using the automix tips, then apply the cement in a thin layer on the bonding surfaces with micro brush, light cure for 2 seconds and remove excess, then light cure for 20 seconds per surface.</td>
</tr>
</tbody>
</table>
3.2.2.7 Cyclic loading

Specimens with their metal threaded bases were then transferred to the plastic chamber of a chewing simulator (chewing simulator CS-4, SD Mechatronik Co., Germany) and secured in their place using putty rubber base impression material (Provil novo putty, Heraeus Kulzer, Germany) and custom made acrylic ring (Ivolen, Ivoclar Vivadent, Liechtenstein, Germany). Crowns were then subjected to compressive cyclic loading for 500,000 cycles at speed 2 Hz in a water bath of 37 C°. The chewing simulator stainless steel antagonist cone, with 30° total taper and its end machined to a curvature equivalent to
3mm diameter, was adjusted to contact the central fossa of the crowns and preload the crowns with 80 N force by 0.2 mm vertical displacement, it was adjusted to move in two directions, vertical movement of 1 mm and horizontal movement 1mm, to induce both vertical and horizontal forces during the chewing cycles (Figure 45).

Figure 45: Crowns being aged in the chewing simulator.

3.2.2.8 Retention testing

The outer surface of the e.max crowns were acid etched with hydrofluoric acid and silanated, while the Lava Ultimate crowns outer surfaces were grit-etched and silanated. Pink wax was used to protect and cover the restoration/tooth interface then acrylic resin was built up over and around the crown inside a threaded metal cylinder to create a base through which it can be attached to the universal testing machine (Figure 46). A dental surveyor was used to adjust the alignment of the threaded metal base. The whole assembly was attached to a universal testing machine (Instron 4301 universal tester, Instron, USA) using special custom made attachment (Figure 47). Tensile force was applied to dislodge the crown away from the tooth along the long axis with cross head
speed of 0.5 mm/min. The force of retention was recorded and then divided by the surface area and retention stress was calculated in MPa.

Figure 46: a) Protecting the restoration tooth interface with wax, b) building acrylic over the crown, c) adding the threaded metal base with acrylic over the crown, d) the attachment device; 1) the threaded metal base for crown, 2) the threaded metal base for tooth.
Figure 47: Top: Pulling the crowns out using Instron universal testing machine, Bottom: crown dislodged from its abutment.
3.2.2.9 Evaluation of the mode of failure:

A single operator inspected the dislodged crowns and their corresponding teeth under a stereomicroscope (Wild/Leica M3Z Stereo Microscope, Heerbrugg, Switzerland) at 10x magnification to fully assess the interfacial area. The modes of failure were inspected according to the following scheme:

1) Adhesive (crown material/cement interface): when at least 75% of the cement layer exists on the substrate material (dentin or composite)

2) Adhesive failure (substrate/cement interface): when at least 75% of the cement layer exists on the Crown material (IPS e.mac CAD or Lava Ultimate)

3) Mixed: when adhesive failure occurs at both interfaces, the cement layer exists on less than 75% of the crown material and substrate.

4) Cohesive failure of the crown material

5) Cohesive failure of the substrate (dentin or composite)

6) Mixed cohesive/adhesive: when adhesive failure is accompanied with any cohesive failure.

3.2.2.10 Statistical analysis:

Statistical analysis was computed using SPSS (PC+ version 20 software, Chicago, IL, USA). Data were presented as mean and standard deviation values of the retention strength. This data is quantitative (continuous). Statistical analysis was carried out using Factorial ANOVA. The significance level was set at $P \leq 0.05$ and 95% confidence level. Factorial analysis of variance was conducted to explore the impact of CAD/CAM material, type of cement and substrate on the retention strength. CAD/CAM material was divided into two groups: Group 1: IPS e.max CAD; Group 2: Lava Ultimate. Types of cement were divided into
two groups: Group 1: Rely X Ultimate; Group 2: RelyX Unicem 2. Substrates were divided into three groups: Group 1: Natural teeth; Group 2: Natural teeth with amalgam filling; Group 3: Natural teeth with composite resin filling.
4. Results

All the investigated main effects were statistically significant at p<0.01, Table 21.

Interaction effect between CAD/CAM material and substrate was statistically significant, $F (2, 84) = 3.275, p=0.043$. Interaction effect between CAD/CAM material and type of cement was statistically significant, $F (1, 84) = 4.416, p=0.039$.

Table 21: Factorial ANOVA (Dependent variable: Bond strength; Independent variables: CAD/CAM material, type of cement and type of the substrate)

<table>
<thead>
<tr>
<th>Source</th>
<th>Type III Sum of Squares</th>
<th>df</th>
<th>Mean Square</th>
<th>F</th>
<th>Sig.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Corrected Model</td>
<td>37.316a</td>
<td>11</td>
<td>3.392</td>
<td>36.934</td>
<td>0.000</td>
</tr>
<tr>
<td>Intercept</td>
<td>766.253</td>
<td>1</td>
<td>766.253</td>
<td>8342.439</td>
<td>0.000</td>
</tr>
<tr>
<td>CAD/CAM material</td>
<td>4.403</td>
<td>1</td>
<td>4.403</td>
<td>47.94</td>
<td>0.000</td>
</tr>
<tr>
<td>Substrate</td>
<td>30.724</td>
<td>2</td>
<td>15.362</td>
<td>167.249</td>
<td>0.000</td>
</tr>
<tr>
<td>Type of Cement</td>
<td>0.781</td>
<td>1</td>
<td>0.781</td>
<td>8.505</td>
<td>0.005</td>
</tr>
<tr>
<td>CAD/CAM material * Substrate</td>
<td>0.602</td>
<td>2</td>
<td>0.301</td>
<td>3.275</td>
<td>0.043</td>
</tr>
<tr>
<td>CAD/CAM material * Type of Cement</td>
<td>0.406</td>
<td>1</td>
<td>0.406</td>
<td>4.416</td>
<td>0.039</td>
</tr>
<tr>
<td>Substrate * Type of Cement</td>
<td>0.091</td>
<td>2</td>
<td>0.045</td>
<td>0.494</td>
<td>0.612</td>
</tr>
<tr>
<td>CAD/CAM material * Substrate * Type of Cement</td>
<td>0.31</td>
<td>2</td>
<td>0.155</td>
<td>1.687</td>
<td>0.191</td>
</tr>
<tr>
<td>Error</td>
<td>7.715</td>
<td>84</td>
<td>0.092</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>811.284</td>
<td>96</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Corrected Total</td>
<td>45.031</td>
<td>95</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

a. R Squared = .829 (Adjusted R Squared = .806)
4.1 Comparison between resin cements

Following detection of a significant interaction, comparison between the types of cements was carried out within levels of the other independent factors: Substrate (Group 1: Natural teeth; Group 2: Natural teeth with amalgam filling; Group 3: Natural teeth with composite resin filling) and CAD/CAM material (Group 1: IPS e.max CAD; Group 2: Lava Ultimate). The follow-up analysis to a significant interaction in factorial ANOVA was carried out using independent sample t-test. The results of the independent sample t-test are presented in Table 22. The significance level was set at \( P \leq 0.05 \) and 95 % confidence level. The means and the standard deviation (SD) values are presented in Table 23.

<table>
<thead>
<tr>
<th>CAD/CAM material</th>
<th>Substrate</th>
<th>Levene’s Test for Equality of Variances</th>
<th>t-test for Equality of Means</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>F</td>
<td>Sig.</td>
</tr>
<tr>
<td>IPS e.max CAD</td>
<td>Natural teeth</td>
<td>E.V.A</td>
<td>0.324</td>
</tr>
<tr>
<td></td>
<td>Natural teeth</td>
<td>E.V.N.A</td>
<td>4.054</td>
</tr>
<tr>
<td></td>
<td>Natural teeth + Amalgam</td>
<td>E.V.A</td>
<td>0.139</td>
</tr>
<tr>
<td></td>
<td>Natural teeth + Composite</td>
<td>E.V.A</td>
<td>2.891</td>
</tr>
<tr>
<td></td>
<td>Natural teeth + Composite</td>
<td>E.V.N.A</td>
<td>2.416</td>
</tr>
<tr>
<td>Lava Ultimate</td>
<td>Natural teeth</td>
<td>E.V.A</td>
<td>1.173</td>
</tr>
<tr>
<td></td>
<td>Natural teeth</td>
<td>E.V.N.A</td>
<td>2.644</td>
</tr>
<tr>
<td></td>
<td>Natural teeth + Amalgam</td>
<td>E.V.A</td>
<td>0.488</td>
</tr>
<tr>
<td></td>
<td>Natural teeth + Composite</td>
<td>E.V.A</td>
<td>2.565</td>
</tr>
<tr>
<td></td>
<td>Natural teeth + Composite</td>
<td>E.V.N.A</td>
<td>2.489</td>
</tr>
</tbody>
</table>

E.V.A: equal variance assumed; E.V.N.A: equal variance not assumed
Table 23: Descriptive showing mean and standard deviation and the pairwise comparison of the type of cements

<table>
<thead>
<tr>
<th>CAD/CAM material</th>
<th>Substrate</th>
<th>N</th>
<th>Mean(SD) *</th>
<th>95% Confidence Interval for Mean</th>
</tr>
</thead>
<tbody>
<tr>
<td>IPS e.max</td>
<td>Natural teeth</td>
<td>8</td>
<td>3.88(0.56) (a)</td>
<td>3.40-4.35</td>
</tr>
<tr>
<td></td>
<td>Rely X Ultimate</td>
<td>8</td>
<td>2.94(0.31) (b)</td>
<td>2.67-3.21</td>
</tr>
<tr>
<td></td>
<td>Rely X Unicem 2</td>
<td>8</td>
<td>2.24(0.23) (c)</td>
<td>2.04-2.43</td>
</tr>
<tr>
<td></td>
<td>Rely X Unicem 2</td>
<td>8</td>
<td>1.92(0.20) (d)</td>
<td>1.75-2.09</td>
</tr>
<tr>
<td></td>
<td>Natural teeth + Amalgam</td>
<td>8</td>
<td>2.91(0.22) (e)</td>
<td>2.72-3.10</td>
</tr>
<tr>
<td></td>
<td>Rely X Ultimate</td>
<td>8</td>
<td>2.49(0.44) (f)</td>
<td>2.12-2.86</td>
</tr>
<tr>
<td></td>
<td>Rely X Unicem 2</td>
<td>8</td>
<td>2.35(0.14) (g)</td>
<td>2.23-2.47</td>
</tr>
<tr>
<td></td>
<td>Rely X Unicem 2</td>
<td>8</td>
<td>2.13(0.20) (h)</td>
<td>1.96-2.30</td>
</tr>
<tr>
<td></td>
<td>Natural teeth + Composite</td>
<td>8</td>
<td>2.98(0.18) (i)</td>
<td>2.82-3.14</td>
</tr>
<tr>
<td></td>
<td>Rely X Ultimate</td>
<td>8</td>
<td>2.66(0.30) (j)</td>
<td>2.40-2.92</td>
</tr>
<tr>
<td></td>
<td>Rely X Unicem 2</td>
<td>8</td>
<td>2.13(0.20) (k)</td>
<td>1.96-2.30</td>
</tr>
</tbody>
</table>

*: Significant at P ≤ 0.05, Groups with different letters are statistically significant different according Tukey’s test results.
4.1.1 Lithium disilicate glass ceramic (IPS e.max CAD)

Comparison between different types of cements was carried when these cements were used to bond lithium disilicate glass ceramic crowns (IPS e.max CAD) to different substrates (Natural teeth, Natural teeth + Amalgam, Natural Teeth + Composite). The means and standard deviation (SD) values are presented in Figure 48 and Table 23.

4.1.1.1 Natural teeth

T-test for independent samples indicated that etch-and-rinse adhesive-based resin cement (RelyX Ultimate) showed the highest mean retention strength (p=0.001), while self-adhesive resin cement (RelyX Unicem 2), showed the lowest retention strength. The means and standard deviation (SD) values are presented in Figure 48.

4.1.1.2 Natural teeth + Amalgam

T-test for independent samples indicated that etch-and-rinse adhesive-based resin cement (RelyX Ultimate) showed the highest mean retention strength (p=0.012), while self-adhesive resin cement (RelyX Unicem 2), showed the lowest retention strength. The means and standard deviation (SD) values are presented in Figure 48.

4.1.1.3 Natural teeth + Composite

T-test for independent samples indicated that etch-and-rinse adhesive-based resin cement (RelyX Ultimate) showed the highest mean retention strength (p=0.035), while self-adhesive resin cement (RelyX Unicem 2), showed the lowest retention strength. The means and standard deviation (SD) values are presented in Figure 48.
Figure 48: Bar chart of the mean values of retention strength (RS) for different cements with IPS e.max CAD. The error bars represent standard deviation. For every level of comparison, groups with different letters shows a statistically significant difference according to Tukey's test results.
4.1.2 Composite resin (Lava Ultimate)

Comparison between different types of cements was carried when these cements were used to bond composite resin crowns (Lava Ultimate) to different substrates (Natural teeth, Natural teeth + Amalgam, Natural Teeth + Composite). The means and the standard deviation (SD) values are presented in Figure 49 and Table 23.

4.1.2.1 Natural teeth

T-test for independent samples indicated that etch-and-rinse adhesive-based resin cement (RelyX Ultimate) showed the highest mean retention strength (p=0.019), while self-adhesive resin cement (RelyX Unicem 2), showed the lowest retention strength. The means and standard deviation (SD) values are presented in Figure 49.

4.1.2.2 Natural teeth + Amalgam

T-test for independent samples indicated that etch-and-rinse adhesive-based resin cement (RelyX Ultimate) showed the highest statistically significant mean retention strength (p=0.022), while self-adhesive resin cement (RelyX Unicem 2), showed the lowest retention strength. The means and standard deviation (SD) values are presented in Figure 49.

4.1.2.3 Natural teeth + Composite

T-test for independent samples indicated that etch-and-rinse adhesive-based resin cement (RelyX Ultimate) showed the highest mean retention strength (p=0.026), while self-adhesive resin cement (RelyX Unicem 2), showed the lowest retention strength. The means and standard deviation (SD) values are presented in Figure 49.
Figure 49: Bar chart of the mean values of retention strength (RS) for different cements with Lava Ultimate. The error bars represent standard deviation. For every level of comparison, groups with different letters show a statistically significant difference according to Tukey’s test results.
4.2 Comparison between substrates:

Following detection of a significant interactions, comparison between the substrates was carried out within levels of the other independent factors: CAD/CAM material (Group 1: IPS e.max CAD; Group 2: Lava Ultimate) and type of cement (Group 1: Rely X Ultimate; Group 2: RelyX Unicem 2). The Follow-up analysis to a significant interaction in factorial ANOVA was carried out using one-way analysis of variance between-groups (one-way ANOVA) for every level of comparison. One-way ANOVA show significance at p<0.001 at all levels of comparison. The results of the one-way ANOVA are presented in Table 24. Post-hoc comparisons were carried using Tukey’s test; the significance level was set at P ≤ 0.05 and 95 % confidence level. The means and the standard deviation (SD) values are presented in Table 23.

<p>| Table 24: One way ANOVA (Dependent variable: Retention strength; Independent variable: type of cement) |</p>
<table>
<thead>
<tr>
<th>CAD/CAM material</th>
<th>Substrate</th>
<th>Sum of Squares</th>
<th>df</th>
<th>Mean Square</th>
<th>F</th>
<th>Sig.</th>
</tr>
</thead>
<tbody>
<tr>
<td>IPS e.max CAD</td>
<td>RelyX</td>
<td>Between Groups</td>
<td>10.851</td>
<td>2</td>
<td>5.426</td>
<td>37.882</td>
</tr>
<tr>
<td></td>
<td>Ultimate</td>
<td>Within Groups</td>
<td>3.008</td>
<td>21</td>
<td>0.143</td>
<td></td>
</tr>
<tr>
<td></td>
<td>RelyX</td>
<td>Between Groups</td>
<td>4.169</td>
<td>2</td>
<td>2.084</td>
<td>18.485</td>
</tr>
<tr>
<td></td>
<td>Unicem 2</td>
<td>Within Groups</td>
<td>2.368</td>
<td>21</td>
<td>0.113</td>
<td></td>
</tr>
<tr>
<td>Paradigm MZ100</td>
<td>RelyX</td>
<td>Between Groups</td>
<td>9.078</td>
<td>2</td>
<td>4.539</td>
<td>85.385</td>
</tr>
<tr>
<td></td>
<td>Ultimate</td>
<td>Within Groups</td>
<td>1.116</td>
<td>21</td>
<td>0.053</td>
<td></td>
</tr>
<tr>
<td></td>
<td>RelyX</td>
<td>Between Groups</td>
<td>7.628</td>
<td>2</td>
<td>3.814</td>
<td>65.467</td>
</tr>
<tr>
<td></td>
<td>Unicem 2</td>
<td>Within Groups</td>
<td>1.223</td>
<td>21</td>
<td>0.058</td>
<td></td>
</tr>
</tbody>
</table>
4.2.1 Lithium disilicate glass ceramic (IPS e.max CAD)

Comparison between different substrates was carried out when etch-and-rinse adhesive-based resin cement (RelyX Ultimate) and self-adhesive resin cement (RelyX Unicem 2) were used to bond the lithium disilicate glass ceramic crowns (IPS e.max CAD). The means and standard deviation (SD) values are presented in Figure 50 and Table 23.

4.2.1.1 Etch-and-rinse adhesive-based resin cement (RelyX Ultimate)

One-way analysis of variance was conducted to explore the effect of the substrate on the retention strength (RS) when etch-and-rinse adhesive-based resin cement (RelyX Ultimate) was used to bond lithium disilicate glass ceramic crowns (IPS e.max CAD). There was a statistically significant difference at p<0.001 (Table 24). Post-hoc comparisons using the Tukey’s test indicated that natural teeth had the highest mean retention strength. This was followed by natural teeth + composite, which came in the second rank. Finally, natural teeth + amalgam had the lowest retention strength.

4.2.1.2 Self-adhesive resin cement (RelyX Unicem 2)

One-way analysis of variance was conducted to explore the effect of the substrate on the retention strength (RS) when self-adhesive resin cement (RelyX Unicem 2) was used to bond lithium disilicate glass ceramic crowns (IPS e.max CAD). There was a statistically significant difference at p<0.001 (Table 24). Post-hoc comparisons using Tukey’s test indicated that natural teeth had the highest mean retention strength. This was followed by natural teeth + composite, which came in the second rank. Finally, natural teeth + amalgam had the lowest retention strength.
Figure 50: Bar chart of the mean values of retention strength (RS) for different substrates with IPS e.max CAD. The error bars represent standard deviation. For every level of comparison, groups with different letters shows a statistically significant difference according to Tukey’s test results.
4.2.2 Composite resin (Lava Ultimate)

Comparison between different substrates was carried when etch-and-rinse adhesive-based resin cement (RelyX Ultimate) and self-adhesive resin cement (RelyX Unicem 2) were used to bond the composite resin crowns (Lava Ultimate). The means and standard deviation (SD) values are presented in Figure 51 and Table 23.

4.2.2.1 Etch-and-rinse (RelyX Ultimate)

One-way analysis of variance was conducted to explore the effect of the substrate on the retention strength (RS) when etch-and-rinse adhesive-based resin cement (RelyX Ultimate) was used to bond the composite resin crowns (Lava Ultimate). There was a statistically significant difference at $p<0.001$ (Table 24). Post-hoc comparisons using Tukey’s test indicated that natural teeth had the highest mean retention strength. This was followed by natural teeth + composite, which came in the second rank. Finally, natural teeth + amalgam had the lowest retention strength.

4.2.2.2 Self-adhesive resin cement (RelyX Unicem 2)

One-way analysis of variance was conducted to explore the effect of the substrate on the retention strength (RS) when self-adhesive resin cement (RelyX Unicem 2) was used to bond the composite resin crowns (Lava Ultimate). There was a statistically significant difference at $p<0.001$ (Table 24). Post-hoc comparisons using Tukey’s test indicated that natural teeth had the highest mean retention strength. This was followed by natural teeth + composite, which came in the second rank. Finally, natural teeth + amalgam had the lowest retention strength.
Figure 51: Bar chart of the mean values of retention strength (RS) for different cements with Lava Ultimate. The error bars represent standard deviation. For every level of comparison, groups with different letters shows a statistically significant difference according to Tukey’s test results.
4.3 Comparison between CAD/CAM materials

Following detection of a significant interaction, comparison between the CAD/CAM materials was carried within levels of the other independent factors: Substrate (Group 1: Natural teeth; Group 2: Natural teeth with amalgam filling; Group 3: Natural teeth with composite resin filling) and Type of cement (Group 1: RelyX Ultimate; Group 2: RelyX Unicem 2). The follow up analysis to a significant interaction in factorial ANOVA was carried out using independent sample t-test. The results of the independent sample t-test are presented in Table 25. The significance level was set at $P \leq 0.05$ and 95% confidence level. The means and the standard deviation (SD) values are presented in Table 23.

<table>
<thead>
<tr>
<th>CAD/CAM material</th>
<th>Substrate</th>
<th>Levene’s Test for Equality of Variances</th>
<th>t-test for Equality of Means</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>F</td>
<td>Sig.</td>
</tr>
<tr>
<td>RelyX Ultimate</td>
<td>Natural teeth</td>
<td>E.V.A</td>
<td>0.449</td>
</tr>
<tr>
<td></td>
<td></td>
<td>E.V.N.A</td>
<td>0.113</td>
</tr>
<tr>
<td></td>
<td>Natural teeth + Amalgam</td>
<td>E.V.A</td>
<td>2.272</td>
</tr>
<tr>
<td></td>
<td></td>
<td>E.V.N.A</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Natural teeth + Composite</td>
<td>E.V.A</td>
<td>0.495</td>
</tr>
<tr>
<td></td>
<td></td>
<td>E.V.N.A</td>
<td></td>
</tr>
<tr>
<td>RelyX Unicem 2</td>
<td>Natural teeth</td>
<td>E.V.A</td>
<td>2.355</td>
</tr>
<tr>
<td></td>
<td></td>
<td>E.V.N.A</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Natural teeth + Amalgam</td>
<td>E.V.A</td>
<td>0.119</td>
</tr>
<tr>
<td></td>
<td></td>
<td>E.V.N.A</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Natural teeth + Composite</td>
<td>E.V.A</td>
<td>2.686</td>
</tr>
<tr>
<td></td>
<td></td>
<td>E.V.N.A</td>
<td></td>
</tr>
</tbody>
</table>

_E.V.A: equal variance assumed; E.V.N.A: equal variance not assumed_
4.3.1 Etch-and-rinse adhesive-based resin cement (RelyX Ultimate)

Comparison between different CAD/CAM materials was carried when bonded to different substrates using etch-and-rinse adhesive-based resin cement (RelyX Ultimate). The means and the standard deviation (SD) values are presented in Figure 52 and Table 23.

4.3.1.1 Natural teeth

T-test for independent samples indicated that there was no significant difference in retention strength between lithium disilicate glass ceramic (IPS e.max CAD) and composite resin (Lava Ultimate) crowns (p=0.911). The means and the standard deviation (SD) values are presented in Figure 52.

4.3.1.2 Natural teeth + Amalgam

T-test for independent samples indicated that there was no significant difference in retention strength between lithium disilicate glass ceramic (IPS e.max CAD) and composite resin (Lava Ultimate) crowns (p=0.261). The means and the standard deviation (SD) values are presented in Figure 52.

4.3.1.3 Natural teeth + Composite

T-test for independent samples indicated that there was no significant difference in retention strength between lithium disilicate glass ceramic (IPS e.max CAD) and composite resin (Lava Ultimate) crowns (p=0.543). The means and the standard deviation (SD) values are presented in Figure 52.
Figure 52: Bar chart of the mean values of retention strength (RS) for different CAD/CAM materials with RelyX Ultimate. The error bars represent standard deviation. For every level of comparison, groups with different letters shows a statistically significant difference according to Tukey's test results.
4.3.2 Self-adhesive resin cement (RelyX Unicem 2)

Comparison between different CAD/CAM materials was carried when bonded to different substrates using self-adhesive resin cement (RelyX Unicem 2). The means and the standard deviation (SD) values are presented in Figure 53 and Table 23.

4.3.2.1 Natural teeth

T-test for independent samples indicated that composite resin crowns (Lava Ultimate) showed the highest mean retention strength (p<0.01), lithium disilicate glass ceramic crowns (IPS e.max CAD) showed the lowest retention strength. The means and standard deviation (SD) values are presented in Figure 53.

4.3.2.2 Natural teeth + Amalgam

T-test for independent samples indicated that there was no significant difference in retention strength between lithium disilicate glass ceramic (IPS e.max CAD) and composite resin (Lava Ultimate) crowns (p=0.064). The means and the standard deviation (SD) values are presented in Figure 53.

4.3.2.3 Natural teeth + Composite

T-test for independent samples indicated that there was no significant difference in retention strength between lithium disilicate glass ceramic (IPS e.max CAD) and composite resin (Lava Ultimate) crowns (p=0.381). The means and the standard deviation (SD) values are presented in Figure 53.
Figure 53: Bar chart of the mean values of retention strength (RS) for different CAD/CAM materials with RelyX Unicem. The error bars represent standard deviation. For every level of comparison, groups with different letters shows a statistically significant difference according to Tukey's test results.
4.4 Modes of failure

The distribution of the modes of failure of the specimens of the retention strength test is shown in figure (54), the majority of the failures were adhesive in nature at the crown/cement interface 42.71%, followed by adhesive failure at substrate/cement interface 36.46%; followed by cohesive failures of substrate 15.63% and finally mixed failures 5.21%.

Figure 54: Pie chart showing distribution of the modes of failure in the retention strength (RS) Test.
Figure 55: Summary Bar chart of the mean values of Retention strength (RS) for different types of cements. The error bars represent standard deviation. For every level of comparison, groups with different letters show statistically significant differences according to Tukey’s test results.
Figure 56: Summary Bar chart of the mean values of Retention strength (RS) for different Substrates. The error bars represent standard deviation. For every level of comparison, groups with different letters shows a statistically significant difference according to Tukey’s test results.
Figure 57: Summary Bar chart of the mean values of Retention strength (RS) for different CAD/CAM materials. The error bars represent standard deviation. For every level of comparison, groups with different letters shows a statistically significant difference according to Tukey’s test results.
5. Discussion

The retention strength test has been employed to evaluate adhesion of dental crowns to the underlying tooth substrate. It offers several advantages over other adhesion tests. The retention test considers the complex configuration of the substrate to which the crown is cemented, not like the bond strength tests that consider the substrate as a one flat surface. This configuration factor is much higher in crowns cemented to their underlying teeth, compared to simple composite or ceramic cylinders cemented onto flat substrate surfaces as in bond strength tests assembly. The higher is the value of the configuration factor, the more the stresses are induced at the bonded interfaces as a result of polymerization shrinkage. Several studies have shown the significant effect of the configuration factor on adhesion, which might explain the low values obtained from retention testing in the range of 1–10 MPa compared to that obtained from microtensile bond strength testing in the range of 20–40 MPa.

Chewing simulation was used as a clinically relevant aging methodology, where crowns were subjected to chewing motion at a rate of 2 Hz, in a water bath of 37 C°, with load of 80 N and 1 mm vertical and horizontal movement of the blunt indenter, for 500,000 cycles. For an average person, the number of the daily chewing cycles can be in the range of 800 to 1400, and may reach up to 2700. However not all of these cycles are active cycles and the number of active cycles would be less, therefore, half a million cycles is considered to correspond to approximately 10 years of normal function in the oral cavity. The rate of chewing cycles was set at 2 Hz to simulate the natural condition. This is in agreement with Bates et al. (1975), who found that the frequency of normal chewing cycles can range from 1-2 Hz. The load used in the chewing simulator
was 80 N, which lies within the range of normal masticatory forces that was estimated to be in the range of 50 to 250 N, and may reach 500 to 800 N in parafunctional situations such as clenching and bruxism.\textsuperscript{225,226} The stainless steel antagonist cone used with 30° total taper and its end machined to a curvature equivalent to 3 mm diameter to reproduce clinical contact pressure, is in agreement with Kelly et al. (1999) who stated that in order to have clinically relevant contacts, the steel antagonists should have contact areas ranging from 0.5 to 3 mm.\textsuperscript{227}

Comparing different cements, etch-and-rinse adhesive-based resin cement (RelyX Ultimate) prevailed in all groups with the highest mean retention strength while self-adhesive resin cement (Rely X Unicem 2) showed the lowest statistically significant retention strength in all groups. This can be explained by the better demineralization ability of the etch-and-rinse system with the maximum smear layer removal, exposing a greater quantity of dentinal tubules, intertubular dentin and collagen fibers to resin infiltration, yielding the well-documented hybrid layer.\textsuperscript{80} The low bonding performance of self-adhesive resin cements could be explained by the weak tangible infiltration of these cements into dentin.\textsuperscript{110} Monticelli et al (2008) reported no demineralization of dentin and no hybrid layer formation with self-adhesive resin cements.\textsuperscript{137} Furthermore, interference with the polymerization reaction might occur as a result of incomplete neutralization of the acidic group, which may in turn result in lower mechanical properties of the set cement and lower bond strength values. It is well known that acidic monomers are capable of reacting with the free radical polymerization initiators, resulting in a decrease in the degree of polymerization of the resin cement.\textsuperscript{5,204,205} In addition, these acidic monomers are hydrophilic and can mediate hydrolytic reactions through increased
water sorption that can negatively affect the bond strength. Kumbuloglu et al. (2004),
found that RelyX Unicem had the lowest degree of conversion compared to three other
adhesive-based cements: 56% when light-cured and only 26% when self-cured. Lower
degree of conversion and higher residual monomer could have strong impact in lowering
the mechanical properties of the self-adhesive resin cement in relation to the etch-and-
rinse adhesive-based resin cement. Piwowarczyk et al. (2003) found that RelyX Unicem
had lower flexural and compressive strength compared with three other adhesive-based
resin cements. Results of the present study are in agreement with De Angelis et al
(2004) who found that multi-step adhesive-based resin cements are capable of achieving
higher bond strength compared with RelyX Unicem self-adhesive resin cements when
bonding lithium disilicate glass ceramic disks and composite resin disks to dentin.
Furthermore, Frankenberger et al (2008), found that etch-and-rinse adhesive-based resin
cements have better marginal sealability than RelyX Unicem self-adhesive resin cement,
and thus can provide better adhesive luting of glass ceramic esthetic restorations.

Comparing different substrates, non-restored natural teeth prevailed in all groups
with the highest statistically significant retention strength, while natural teeth restored
with composite resin ranked second, and natural teeth restored with amalgam
restorations showed the lowest mean retention strength. Non-restored natural teeth are
one-component substrate (dentin) with uniform mechanical properties such as the
modulus of elasticity and the degree of resilience, compared to other substrates when two
components exist, dentin and restoration, where each component had its own mechanical
properties and responded differently to applied stresses. When restorations were aged in
the chewing simulator, every component responded to the applied stress with different
degrees of elastic deformation based on different moduli of elasticity, which could lead to stress concentration and development of cracks within the cement layer, thus weakening the overall retention strength. Van Noort et al (1989) reported that stress distribution within the adhesive interface is affected by the modulus of elasticity of the materials forming the interface. Natural teeth with composite restorations presented higher mean retention strength compared to natural teeth with amalgam restoration. This could be explained by the copolymerization between unreacted monomers of the resin cement and that of the composite substrate, thus increasing the retention strength. Kallio et al (2001) demonstrated that the unreacted monomer groups in a composite substrate could allow formation of covalent bonds through copolymerization between the substrate and repair resin or intermediary resin.

Comparing different crown materials, there was no statistically significant difference between lithium disilicate glass ceramic (IPS e.max CAD) and composite resin (Lava Ultimate) crowns in all groups, except when self-adhesive resin cement (RelyX Unicem 2) was used to bond crowns to natural teeth, where composite resin (Lava ultimate) crowns showed the highest mean retention strength. This could be explained by the weak mechanical properties of the self-adhesive resin cement (RelyX Unicem 2) in relation to the etch-and-rinse adhesive based resin cement, which resulted in weaker micromechanical retention to the lithium disilicate ceramic in comparison with the composite substrate. However, this was not the case for other groups cemented with self-adhesive resin cement (RelyX Unicem 2) where both the ceramic and the composite resin crown materials showed similar mean retention strength values. This could be due to the two component nature of the substrate in these groups, where every component responds
to stresses in a different way and could lead to stress concentration with the adhesive interface and weakening of the overall retention strength, which could result in similar retention strength for both crown materials. Van Noort et al (1989) mentioned that the stress distribution within the adhesive interface is dependent on the modulus of elasticity of the materials forming this adhesive interface. Tam et al (2000) demonstrated that fracture toughness of the adhesive interface is affected by the elastic moduli of the materials constituting the interface as well as the degree of interaction whether mechanical or chemical between these materials.

The distribution of the modes of failure in this study was mainly adhesive failure at the crown/cement interface 42.71%, followed by adhesive failure at the substrate/cement interface 36.46%, followed by cohesive failures of substrate 15.63% and finally mixed failures 5.21%. This indicates that the retention strength test was capable of concentrating the stresses at the adhesive interface. Cohesive failures of the substrate could indicate that the retention strength at the adhesive interface was high enough to withstand the pulling forces compared to the substrate.
6. Limitations of the study

This study has the following limitations:

1. It is an in-vitro study that helps in preliminary assessment of dental biomaterials bonding properties, but cannot be an alternative to in-vivo studies.

2. Using natural teeth with different sizes and shapes creates a problem in standardization, however, even distribution of different sizes of the teeth among the study groups was achieved and the surface area of the prepared teeth was measured and considered in bond strength calculations.

3. This study did not include control group of specimens that were not subjected to fatigue cyclic loading and hence the effect of cyclic loading was not tested.

7. Conclusions

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

- Etch-and-rinse adhesive-based resin cement was capable of achieving the highest retention strength irrespective of the crown material or the bonding substrate.

- Self-adhesive resin cement showed the lowest retention strength irrespective of the crown material or the bonding substrate.

- Non-restored natural teeth were capable of achieving better retention strength compared to restored teeth irrespective of the crown material or the cement.

- Amalgam restorations negatively affect the retention strength to crown materials irrespective of the cement.

- Lithium disilicate glass ceramic crowns and composite resin crowns can achieve similar retention strength when luted with etch-and-rinse adhesive-based resin cement irrespective of the bonding substrate.
Chapter 6 Microleakage Of CAD-CAM Crowns Using Self-adhesive Resin Cement
1. Abstract

Objectives: To determine the microleakage of etch-and-rinse adhesive-based resin cement and self-adhesive resin cement systems, when used to bond lithium disilicate glass ceramic (IPS e.max CAD) and composite resin (Lava Ultimate) crowns to sound natural teeth. Methods: Thirty-two freshly extracted molars were prepared to receive CAD/CAM crowns using a special paralleling device and were divided into four equal groups. IPS- e.max-CAD (EMX) crowns were made for teeth in two groups while composite crowns (Lava Ultimate) (LU) were made for the other two groups. Crowns were milled using CEREC-3D system. Crowns of one EMX group and one LU group were cemented to their respective teeth using etch-and-rinse adhesive-based resin cement (RelyX-Ultimate) (RXL). Crowns of other two groups were cemented with self-adhesive resin cement (Rely-Unicem-2) (RXU). Crowns were then subjected to compressive cyclic-loading (500,000 cycle, 80N, 1mm vertical, 1mm horizontal, in water at 37°C, 2Hz) in chewing-simulator. Crown specimens were immersed in 2% aqueous solution of red basic Procion dye for 24 hours. The specimens were sectioned mesio-distally and the dye penetration was evaluated under stereomicroscope. Data were then statistically analyzed with Mann Whitney U test. Results: Mann Whitney U test revealed significant difference among the cement groups (P<0.05). RelyX Unicem showed statistically significantly higher microleakage scores compared to RelyX Ultimate. There was no significant difference between IPS e.max CAD and Lava Ultimate regarding microleakage scores. Conclusions: this in-vitro study showed that etch-and-rinse adhesive-based resin cement (RelyX Ultimate) resulted in lower microleakage scores.
compared to self-adhesive resin cement (RelyX Unicem 2). Crown material had no effect on the microleakage scores.

2. Introduction

Microleakage is defined as the movement of fluids carrying bacteria, molecules and ions at the boundary between a restoration and a tooth. Although it is difficult to detect clinically, microleakage is considered to be a major factor influencing the longevity of dental restorations since it may lead to staining at the margins of the restoration, recurrent caries at the tooth/restoration interface, hypersensitivity of restored teeth, or development of pulpal pathology.

Several methods have been used to evaluate microleakage around dental restorations whether in-vitro or in-vivo. In-vitro microleakage testing of dental materials is a commonly accepted evaluation technique of margin integrity. In-vitro studies use different techniques including the use of tracers such as dyes, radioactive isotopes, chemical tracers, and bacteria, as well as marginal percolation of water and exposure to air pressure, neutron activation analysis (NAA), scanning electron microscopy (SEM), and electrical conductivity.

Several factors have been described to influence the microleakage of bonded crown restorations, which include lack of adequate adhesion of the cement to the tooth structure, shrinkage of the cement on setting, mechanical failure of the cement and type of the crown material. Different types of cement systems vary considerably in solubility, strength, and ability to adhere to tooth structure. Several studies have found significant differences among cement systems in their ability to prevent interfacial leakage between the cement and the tooth structure. A recent in-vitro study
found that composite crowns (Paradigm MZ 100, 3M ESPE) showed higher microleakage scores compared with feldspathic ceramic crowns (VITABLOCS Mark II, VIDENT). However, in another study, microleakage scores of both types of crowns were similar.

This study was designed to compare the microleakage of an etch-and-rinse and a self-adhesive resin cement systems when used to bond a lithium disilicate glass ceramic crowns (IPS e.max CAD, Ivoclar Vivadent Inc.) and composite-resin crowns (Lava Ultimate, 3M ESPE) to sound dentin.

3. Materials and Methods

3.1 Materials

All the material used in the study and their composition (as reported by manufacturers) are listed in Table 26.

3.2 Methods:

3.2.1 Pilot study:

The current study was preceded by an external pilot study to detect any problems with the proposed methods of testing. Where 2 specimens per group were prepared for the pilot study. Following the completion of pilot study, 32 specimens were used for the main study.

3.2.2 Main study

Four groups were formed (n=8)(Figure 58). The number of specimens per group was calculated using web-based software® for sample size analysis at α=0.05 and 80%

® Sample size Analysis For Independent Samples Design http://www.stat.ubc.ca/~rollin/stats/ssize/n2.html/
power. Based on values obtained from the pilot study, $x_1=0.5$, $x_2=1.25$, $\sigma= 0.34$, and according to Lehmann (1998), sample size for non-parametric tests = sample size for parametric test $\times 15\%$, which yields a sample size of 5 samples per group. We used 8 samples per group for this study to optimize power.

Table 26: Material used in the Microleakage Study and their composition (according to the manufacturer)

<table>
<thead>
<tr>
<th>Material</th>
<th>Classification</th>
<th>Brand / Manufacturer</th>
<th>Composition</th>
<th>Lot #</th>
</tr>
</thead>
</table>
| CAD/CAM Materials         | Lithium disilicate ceramic | IPS e.max CAD/ Ivoclar Vivadent, Liechtenstein | $\text{SiO}_2$ (57 – 80 wt.%), $\text{Li}_2\text{O}$ (11 – 19 wt.%)
$\text{K}_2\text{O}$ (0 – 13 wt.%), $\text{P}_2\text{O}_5$ (0 – 11 wt.%), $\text{ZrO}_2$ (0 – 8 wt.%),
$\text{ZnO}$ (0 – 8 wt.%), Other and coloring oxides (0-12 wt.% | P63946  |
| Indirect composite resin  | Lava™ Ultimate/3M ESPE, St Paul, MN, USA | Composite material contains: Zirconia-silica nanocluster: Synthesized via a proprietary process from 20 nm silica particles and 4 to 11 nm zirconia particles;
Nanomere particles: 20 nm silica nanomer particles and 4-11 nm zirconia nanomere particles
The average nanocluster particle size is 0.6 to 10 micrometers.
Resin matrix composition was not mentioned by the manufacturer
Total filler loading is 80% wt. | N481731 |
<table>
<thead>
<tr>
<th>Resin Cements</th>
<th>Self-adhesive resin cement system</th>
<th>Self-adhesive cement</th>
<th>Catalyst paste: methacrylate monomers, alkaline (basic) fillers, silanated fillers, initiator components, stabilizers, pigments and rheological additives</th>
</tr>
</thead>
<tbody>
<tr>
<td>RelyX&lt;sup&gt;TM&lt;/sup&gt; Unicem 2/3M ESPE, St Paul, MN, USA</td>
<td></td>
<td>Base paste: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers, rheological additives.</td>
<td></td>
</tr>
<tr>
<td>(Filler=50% vol., 72 wt.%; avg. &lt; 12.5 μm)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Resin Cements</td>
<td>Silane coupling agent</td>
<td>Stable solution of a prehydrolyzed silane-coupling agent, alcohol and water.</td>
<td>515337</td>
</tr>
<tr>
<td>RelyX Ceramic primer / 3M ESPE, St Paul, MN, USA</td>
<td></td>
<td></td>
<td>N470024</td>
</tr>
<tr>
<td>Resin Cement</td>
<td>Acid etch</td>
<td>Scotchbond™ Phosphoric Etchant / 3M ESPE, St Paul, MN, USA</td>
<td>The etchant is 35% phosphoric acid by weight. It has a pH of approximately 0.6</td>
</tr>
<tr>
<td>Adhesive-based resin cement system</td>
<td>Bonding agent</td>
<td>Scotchbond™ Universal Adhesive / 3M ESPE, St Paul, MN, USA</td>
<td>One-bottle adhesive containing MDP Phosphate Monomer, Dimethacrylate resins, HEMA, Vitrebond™ Copolymer, Filler, ethanol, water, initiators and silane</td>
</tr>
<tr>
<td></td>
<td>Adhesive-based resin cement</td>
<td>RelyX™ Ultimate / 3M ESPE, St Paul, MN, USA</td>
<td>Consists of two pastes: Base paste: methacrylate monomers, radiopaque silanated fillers, initiator components, stabilizers, and rheological additives. Catalyst paste: methacrylate monomers, radiopaque alkaline (basic) fillers, initiator components, stabilizers, pigments, rheological additives, Fluorescence dye and Dark cure activator for Scotchbond Universal (Filler=43 vol.%; avg. = 13 μm)</td>
</tr>
<tr>
<td></td>
<td>Silane coupling agent</td>
<td>RelyX Ceramic primer / 3M ESPE, St Paul, MN, USA</td>
<td>Stable solution of a prehydrolyzed silane-coupling agent, alcohol and water.</td>
</tr>
<tr>
<td>Material</td>
<td>Classification</td>
<td>Brand / Manufacturer</td>
<td>Composition</td>
</tr>
<tr>
<td>--------------------------------------------</td>
<td>-----------------</td>
<td>-----------------------------------------------------------</td>
<td>-----------------------------------------------------------------------------</td>
</tr>
<tr>
<td>Mechanical Surface treatment material</td>
<td>Grit-etching</td>
<td>Aluminum Oxide-50 Micron/ Danville Engineering Inc., California</td>
<td>50 microns aluminum oxide powder.</td>
</tr>
<tr>
<td></td>
<td>Acid etching</td>
<td>IPS Ceramic Etching Gel /Ivoclar Vivadent, Liechtenstein</td>
<td>The etchant is &lt;5% hydrofluoric acid. It has a pH of approximately 2</td>
</tr>
</tbody>
</table>
3.2.2.1 Specimen collection and storage

Intact caries-free human molars were collected and cleaned with periodontal scalers and curettes, then sterilized with gamma radiation (Gamma cell 220, Atomic Energy Ltd, Mississauga, Canada) at the Department of Chemical Engineering and Applied Chemistry, University of Toronto. Teeth were placed in a cobalt chamber 5.5x8 inches, and exposed for 4 hours. The radiation dose rate was 0.3 KGy/h. This method of sterilization has been proved to not alter the mechanical or the physical
properties of the tooth tissue. Teeth were kept wet, wherever possible, in distilled water in a refrigerator during and between all experimental procedures, in order to preserve their optimum mechanical and physical properties.

3.2.2.2 Specimen preparation:

The dimensions of the teeth were measured from both mesio-distal and bucco-lingual direction at the cemento-enamel junction using a digital caliper (Mastercraft, Toronto, Canada) at approximately the mid-point of every surface. An average of a dimension was calculated for every tooth based on the bucco-lingual and the mesio-distal readings. Teeth were then divided into three groups based on size: small (less than 8mm), medium (8-10 mm) and large (more than 10mm). The teeth were randomly distributed among the study groups to have even distribution of different sizes of the teeth. Every group included two small molars, five medium-sized molars and one large molar (n=8). Teeth were then mounted in acrylic resin (Ivolen, Ivoclar Vivadent, Liechtenstein, Germany) using specially prepared mold.

Crown preparations were made with a 1 mm shoulder margin positioned just occlusal to the cemento-enamel junction and 6± 2 degree axial wall convergence for every wall (12± 4 degree total axial convergence mesio-distally and 12± 4 degree total axial convergence bucco-lingually) using flat-end taper diamond bur (#847, Brassler, USA) in a high speed air turbine handpiece with copious water coolant mounted in a parallelogrameter to standardize the taper of the preparation (Figure 59). Bur was changed every eight preparations to ensure good cutting efficiency. The optical impressions subsequently taken by the CAD/CAM camera was used to verify angle of convergence using measuring software (Onde rulers v1.13.1, Ondeosoft Computing Inc.) (Figure 61).
Occlusal surface was finished to create a non-anatomical occlusal table and maintain a vertical height of 4±0.25 mm using wheel diamond bur (#2909, Komet, USA) mounted in a high-speed air turbine handpiece with profuse water coolant.

Figure 59: Left: parallelogrameter adjusted for 6° axial wall convergence, Right: Tooth embedded in acrylic resin ready for crown preparation.

3.2.2.3 Impressions and restoration design

Optical reflective medium (IPS contrast spray, Ivoclar Vivadent, Liechtenstein) was used to cover the prepared teeth with a thin layer. An optical impression of the prepared tooth was captured using the CEREC 3D intraoral camera. CEREC software was used to design crowns (Figure 60). Each crown was made with anatomical features of lower second molar readily available in computer program. Minimum occlusal thickness (at central fossa) was 1.5 mm. Thickness of crown at various points was verified with Boley gauge (miltex, Germany) following milling.
3.2.2.4 Crown fabrication

Sixteen crowns were fabricated from each crown material: IPS. e.max CAD and the Lava Ultimate blocks, using the CEREC 3D milling unit (Figure 62). Cutting diamonds were changed after milling eight crowns. IPS. E.max CAD crowns were
crystallized in a ceramic furnace (EP 600 Combi, Ivoclar Vivadent, Liechtenstein) (Figure 65) according to the manufacturer instructions.

![Cerec 3D milling unit](image1)

**Figure 62: Cerec 3D milling unit.**

![Inside the milling chamber](image2)

**Figure 63: Inside the milling chamber, burs ready to mill IPS e.max CAD.**
Figure 64: Milled IPS e.max CAD crown.

Figure 65: IPS e.max Cad crowns in the EB600 Combi furnace.
3.2.2.5 Crowns surface treatment

The intaglio surfaces of the IPS. e.max CAD crowns were etched with 5% hydrofluoric acid etching gel (IPS ceramic etching gel, Ivoclar Vivadent, Liechtenstein) for 20 seconds then washed using water spray for 60 seconds, air-dried then silanated (RelyX ceramic primer, 3M ESPE). The intaglio surfaces of Lava Ultimate crowns were grit-etched with 50 µm aluminum oxide powder under 2 bar pressure at the nozzle, then cleaned with compressed air-water spray for 30 seconds and further cleaned in distilled water in an ultrasonic cleaner for 5 minutes, air-dried then silanated.

3.2.2.6 Cementation

All crowns were divided into two main groups: one group was cemented with etch-and-rinse adhesive cement (Rely X Ultimate, 3M ESPE) and the other group was cemented with self-adhesive resin cement (Rely X Unicem 2, 3M ESPE). The cements were mixed and applied to the intaglio surfaces of the crowns according to the manufacturer’s instructions (Table 20). The crowns were initially seated on their respective teeth using finger pressure, and then the crowns were placed under a static load of 22 N for 5 minutes (Figure 66). The excess cement was briefly light-cured to gel point then removed. After load removal, each crown was light cured for 20 seconds on each surface.
Figure 66: Crowns were placed under static load of 22N for 5 minutes.

3.2.2.7 Cyclic loading

Specimens with their acrylic base were then transferred to the plastic chamber of a chewing simulator (chewing simulator CS-4, SD Mechatronik Co., Germany)(Figure 67) and secured in their place using putty rubber base impression material (Provil novo putty, Heraeus Kulzer, Germany) and custom made acrylic ring (Ivolen, Ivoclar Vivadent, Liechtenstein, Germany). Crowns were then subjected to compressive cyclic loading for 500,000 cycles at speed 2 Hz in a water bath of 37 C°. The chewing
simulator stainless steel antagonist cone, with 30° total taper and its end machined to a curvature equivalent to 3 mm diameter, was adjusted to contact the central fossa of the crowns and preload the crowns with 80 N force by 0.2 mm vertical displacement. It was adjusted to move in two directions: vertical movement of 1 mm and horizontal movement 1 mm, to induce both vertical and horizontal forces during the simulated chewing cycles.

![Figure 67: Crowns being aged in the chewing simulator.](image)

### 3.2.2.8 Microleakage testing

The root surfaces and acrylic base of every specimen were sealed with nail polish except for 1 mm around the crown margins (Figure 68). Crowns were subjected to microleakage testing by immersion in 2% aqueous solution of red Procion dye (Pararosanilin, Imperial Chemical Industries) (Figure 69) for 24 hours at 37°C. Then the specimens were rinsed thoroughly in water for five minutes and then sectioned mesiodistally using a low-speed saw with a diamond blade (Isomet 1000, Buehler Ltd.,
Lake Bluff, IL)(Figure 70). Images of tooth sections were examined under stereomicroscope at 10x magnification, and a five-point scale was used to score microleakage at both mesial and distal aspects of each section as follows:

0 = no leakage.
1 = microleakage up to one third of axial wall.
2 = microleakage up to two-thirds of axial wall.
3 = microleakage along full length of axial wall.
4 = microleakage extending onto occlusal surface.

The section with the deepest dye penetration was selected as a representative of every specimen.

3.2.2.9  Data analysis

The microleakage data are qualitative (ordinal) data. Data were statistically analyzed with non-parametric Mann-Whitney U-test (P<0.05) at 95% confidence interval level and with analysis of ranks to detect the significant differences between different types of cement and also between crown materials.
Figure 68: Root surfaces and acrylic base sealed with nail polish.

Figure 69: Procion Dye.

Figure 70: low-speed saw with a diamond blade (Isomet 1000, Buehler Ltd., Lake Bluff, IL) used for sectioning specimens mesiodistally.
4. Results

4.1 Comparison between resin cements

Mann-Whitney U test was conducted to explore the impact of the type of cement on the microleakage scores within the levels of the other independent variable (CAD/CAM material). CAD/CAM material was divided into two groups: Group 1: IPS e.max CAD; Group 2: Lava Ultimate. Types of cement was divided into two groups: Group 1: Rely X Ultimate; Group 2: RelyX Unicem. The mean ranks are presented in Table 27 and the results of the Mann-Whitney U test are presented in Table 28.

<table>
<thead>
<tr>
<th>CAD/CAM material</th>
<th>Dependent Variable</th>
<th>Cement</th>
<th>Mean Rank</th>
<th>Sum of Ranks</th>
</tr>
</thead>
<tbody>
<tr>
<td>IPS Emax CAD</td>
<td>Microleakage</td>
<td>RelyX Ultimate</td>
<td>12.91</td>
<td>206.5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>RelyX Unicem</td>
<td>20.09</td>
<td>321.5</td>
</tr>
<tr>
<td>Lava Ultimate</td>
<td>Microleakage</td>
<td>RelyX Ultimate</td>
<td>12.06</td>
<td>193</td>
</tr>
<tr>
<td></td>
<td></td>
<td>RelyX Unicem</td>
<td>20.94</td>
<td>335</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>CAD/CAM Blocks</th>
<th>Microleakage</th>
</tr>
</thead>
<tbody>
<tr>
<td>IPS Emax CAD</td>
<td>Mann-Whitney U</td>
</tr>
<tr>
<td></td>
<td>Wilcoxon W</td>
</tr>
<tr>
<td></td>
<td>Z</td>
</tr>
<tr>
<td></td>
<td>Asymp. Sig. (2-tailed)</td>
</tr>
<tr>
<td></td>
<td>Exact Sig. [2*(1-tailed Sig.)]</td>
</tr>
<tr>
<td>Lava Ultimate</td>
<td>Mann-Whitney U</td>
</tr>
<tr>
<td></td>
<td>Wilcoxon W</td>
</tr>
<tr>
<td></td>
<td>Z</td>
</tr>
<tr>
<td></td>
<td>Asymp. Sig. (2-tailed)</td>
</tr>
<tr>
<td></td>
<td>Exact Sig. [2*(1-tailed Sig.)]</td>
</tr>
</tbody>
</table>

a. Grouping Variable: Cement
b. Not corrected for ties.
4.1.1 Lithium disilicate glass ceramic (IPS e.max CAD)

For lithium disilicate glass ceramic crowns (IPS e.max CAD), Mann-Whitney U test revealed that self-adhesive resin cement (RelyX Unicem 2) showed higher microleakage scores compared to etch-and-rinse adhesive-based resin cements (RelyX Ultimate) (U=70.5, z=-2.396, p=0.029). The Mean Ranks are presented in Table 27. The groups modes are presented in Figure 71 and the distribution of microleakage scores are shown in Figure 72.

![Figure 71: Modes of microleakage scores for cements used to bond IPS e.max CAD crowns](image)
Figure 72: Microleakage scores distribution for cements used to bond IPS e.max CAD crowns.
4.1.2 Composite resin (Lava Ultimate)

For Lava Ultimate crowns, Mann-Whitney U test revealed that self-adhesive resin cement (RelyX Unicem 2) showed higher microleakage scores compared to etch-and-rinse adhesive-based resin cement (RelyX Ultimate) (U=57, z=-3.141, p=0.007). The Mean Ranks are presented in Table 27. The groups modes are presented in Figure 73 and the distribution of microleakage scores are shown in Figure 74.

Figure 73: Groups Modes of microleakage scores for cements used to bond Lava Ultimate crowns.
Figure 74: Microleakage scores distribution for cements used to bond Lava Ultimate crowns.
4.2 Comparison between CAD/CAM materials

Mann-Whitney U test was conducted to explore the impact of the type of the CAD/CAM material on the microleakage scores within the levels of the other independent variable (Type of cements). CAD/CAM material was divided into two groups (Group 1: IPS e.max CAD; Group 2: Lava Ultimate). Type of cement was divided into two groups (Group 1: Rely X Ultimate; Group 2: RelyX Unicem). The mean ranks are presented in Table 29 and the results of the Mann-Whitney U test are presented in Table 30.

<table>
<thead>
<tr>
<th>Type of cement</th>
<th>Dependant Variable</th>
<th>CAD/CAM material</th>
<th>Mean Rank</th>
<th>Sum of Ranks</th>
</tr>
</thead>
<tbody>
<tr>
<td>RelyX Ultimate</td>
<td>Microleakage</td>
<td>IPS e.max CAD</td>
<td>17.22</td>
<td>275.5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Lava Ultimate</td>
<td>15.78</td>
<td>252.5</td>
</tr>
<tr>
<td>RelyX Unicem 2</td>
<td>Microleakage</td>
<td>IPS e.max CAD</td>
<td>17.06</td>
<td>273</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Lava Ultimate</td>
<td>15.94</td>
<td>255</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Type of cement</th>
<th>Microleakage</th>
<th>Mann-Whitney U</th>
<th>116.5</th>
</tr>
</thead>
<tbody>
<tr>
<td>RelyX Ultimate</td>
<td></td>
<td>Wilcoxon W</td>
<td>252.5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Z</td>
<td>-0.495</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Asymp. Sig. (2-tailed)</td>
<td>0.621</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Exact Sig. [2*(1-tailed Sig.)]</td>
<td>.669b</td>
</tr>
<tr>
<td>RelyX Unicem 2</td>
<td></td>
<td>Mann-Whitney U</td>
<td>119</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Wilcoxon W</td>
<td>255</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Z</td>
<td>-0.429</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Asymp. Sig. (2-tailed)</td>
<td>0.668</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Exact Sig. [2*(1-tailed Sig.)]</td>
<td>.752b</td>
</tr>
</tbody>
</table>

a. Grouping Variable: Cement
b. Not corrected for ties.
4.2.1 Etch-and-rinse resin cement (RelyX Ultimate)

For etch-and-rinse adhesive-based resin cement (RelyX Ultimate), Mann-Whitney U test revealed no significant difference between lithium disilicate glass ceramic crowns (IPS e.max CAD) and composite resin crowns (Lava Ultimate) \( (U=116.5, z=-0.495, p=0.669) \). The Mean Ranks are presented in Table 29. The groups modes are presented in Figure 75 and the distribution of microleakage scores are shown in Figure 76.

Figure 75: Groups Modes of microleakage scores for CAD/CAM materials that was bonded with RelyX Ultimate cement
Figure 76: Microleakage scores distribution for CAD/CAM materials that was bonded with RelyX Ultimate cement.
4.2.2 Self-adhesive resin cement (RelyX Unicem 2)

For self-adhesive resin cement (RelyX Unicem 2), Mann-Whitney U test revealed no significant difference between lithium disilicate glass ceramic crowns (IPS e.max CAD) and composite resin crowns (Lava Ultimate) \((U=119, z=-0.429, p=0.752)\). The Mean Ranks are presented in Table 29. The group modes are presented in Figure 77 and the distribution of microleakage scores are shown in Figure 78.

![Graph showing microleakage scores for CAD/CAM materials bonded with RelyX Unicem 2 cement.](image)

**Figure 77:** Groups Modes of microleakage scores for CAD/CAM materials that were bonded with RelyX Unicem 2 cement.
Figure 78: Microleakage scores distribution for CAD/CAM materials that were bonded with RelyX Unicem 2 cement
Figure 79: Microleakage in IPS e.max CAD & RelyX ultimate showing scores of zero on mesial and distal sides.

Figure 80: Microleakage in IPS e.max CAD & RelyX Unicem 2 showing scores of one on mesial and distal sides.
Figure 81: Microleakage in Lava Ultimate & RelyX ultimate showing scores of zero on mesial and one on distal side.

Figure 82: Microleakage in Lava Ultimate & RelyX Unicem showing scores of one on mesial and two on distal side.
5. Discussion

Microleakage testing investigates the sealability of adhesive interfaces. Clinically, if the adhesive interface or the adhesion process is compromised, this results in microleakage, which in turn could result in further adhesive failure.\textsuperscript{193}

In this study, crowns were subjected to chewing forces at a rate of 2 Hz, in a water bath of 37 C\textdegree, with load of 80 N and 1 mm vertical and horizontal movement of the blunt indenter, for 500,000 cycles which is considered to correspond to 10 years of normal function in the oral cavity.\textsuperscript{223} Bates et al. (1975) reported that the normal chewing cycles frequency can range from 1-2 Hz.\textsuperscript{224} The applied load 80 N lies within the range of the normal masticatory forces which was estimated to be in the range of 50 to 250 N, and may reach 500 to 800 N in parafunctional conditions such as clenching and bruxism.\textsuperscript{225,226} Clinical contact pressure was reproduced by the stainless steel load applicator with 30\textdegree total taper and its end machined to a curvature equivalent to 3 mm diameter. Kelly et al. (1999) reported that contact areas ranging from 0.5 to 3 mm are considered clinically relevant contacts.\textsuperscript{227}

In the current study our aim was to investigate the effect of an etch-and-rinse adhesive-based resin cement (RelyX Ultimate) and self-adhesive resin cement (RelyX Unicem 2) on the sealability of the adhesive interface when used to bond lithium disilicate glass ceramic crowns (IPS e.max CAD) and composite resin crowns (Lava Ultimate) to tooth structure. Also, this study aimed to investigate the effect of the crown material on this sealability.

When comparing different types of cements, self-adhesive resin cement (RelyX Unicem 2) presented the highest microleakage score irrespective of the crown material,
while the etch-and-rinse adhesive-based resin cement (RelyX Ultimate) showed the lowest microleakage scores irrespective of the crown material. This could be partly explained by the strong etching and resin infiltration of the etch-and-rinse adhesive based resin cement with subsequent hybrid layer formation,¹⁵⁰,²¹⁸,²³⁶,²³⁷ which can result in stronger micromechanical bonding to tooth structure and low microleakage scores.¹⁹⁹ On the other hand, the weaker adhesion of the self-adhesive resin as a consequence of their weak demineralization of tooth structure and tangible resin infiltration with no hybrid layer formation could be the reason of high microleakage scores.¹¹⁰,¹³⁷

Furthermore, the chemical composition of the cement plays an important role, where etch-and-rinse adhesive-based resin cement is more hydrolytically stable as opposed to the self-adhesive resin cement that incorporates acidic monomers in its chemical formulation to be capable of etching the tooth structure.¹⁰⁶,²⁰⁶ These acidic monomers are hydrophilic and results in increased water sorption which in turn mediate hydrolytic reactions at the adhesive interface thereby compromising adhesion to tooth structure.²²⁸ Also, self-adhesive resin cement is characterized by low degree of conversion as opposed to etch-and-rinse adhesive based resin cement, a condition that is associated with increased microleakage as a consequence to leaching out of the residual monomer and ingress of the oral fluids through the created microgaps.²²⁹ Results of the present study are in agreement with Kassem et al (2011) who found that self-adhesive resin cement (RelyX Unicem) showed the highest microleakage scores when compared to the adhesive-based resin cement Panavia F, when both cements were used to cement feldspatic ceramic crowns.²³⁸
When comparing different crown materials, there was no significant difference in microleakage scores between IPS e.max CAD ceramic crowns and Lava Ultimate composite resin crowns. This shows that the main factor in sealing the adhesive interface is the cement rather than the crown material. Our results are in agreement with Kassem et al (2011) who found that there was no significant difference in microleakage scores between ceramic and composite crowns.194

6. Limitations of the study

This study has the following limitations:

1. This is an in-vitro study that helps in primary examination of dental biomaterials bonding properties, but cannot be an alternative to in-vivo studies.

2. Implementing natural teeth in a study creates standardization problem due to different sizes and shapes of these teeth. However, even distribution of different sizes of the teeth among the study groups was achieved which help improving standardization.

3. There was no control group of specimens that were not subjected to fatigue cyclic loading in this study and hence the effect of cyclic loading can’t be demonstrated in this study.

7. Conclusions:

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

- Etch-and-rinse adhesive-based resin cement was capable of achieving better sealability irrespective of the crown material bonded to dentin.
Self-adhesive resin cement showed the highest microleakage scores irrespective of the crown material bonded to dentin.

Crown material had no effect on the microleakage scores.
Chapter 7 Summary and conclusions
This study investigated adhesion and microleakage of adhesive-based and self-adhesive resin cements when used to cement lithium disilicate glass ceramic and composite resin CAD/CAM crown materials to different substrates. Several testing approaches were utilized as follows:

1. Microtensile bond strength testing (chapter 3): the results of this part favored adhesive-based resin cements over self-adhesive resin cements, composite resin crown material (Paradigm MZ100) over lithium disilicate glass ceramic crown material (IPS e.max CAD) and composite resin substrate over dentin substrate. Also, based on the findings, hydrofluoric acid etching surface treatment resulted in superior bonding of the lithium disilicate glass ceramic to the substrate compared to grit-etching.

2. Microshear bond strength testing (chapter 4): the results of this part favored adhesive-based resin cements over self-adhesive resin cements and composite substrate over dentin substrate. However, composite resin crown material (Paradigm MZ100) and lithium disilicate glass ceramic crown material (IPS e.max CAD) behaved similarly regarding the bond strength when IPS e.max CAD was treated with hydrofluoric acid etching.

3. Retention strength testing (chapter 5): the results of this part favored adhesive-based resin cement over self-adhesive resin cement and non-restored natural teeth substrate over amalgam and composite resin restored natural teeth. Amalgam restorations were found to result in decreased retention strength. No difference in retention strength was detected for composite resin crown material (Lava
Ultimate) and lithium disilicate glass ceramic crown material (IPS e.max CAD) when luted with etch-and-rinse adhesive-based resin cements.

4. Microleakage testing (chapter 6): the results of this part favored etch-and-rinse adhesive-based resin cement over the self-adhesive resin cement. However, composite resin crown material (Lava Ultimate) and lithium disilicate glass ceramic crown material (IPS e.max CAD) had no effect on the microleakage scores.
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