PALACKY UNIVERSITY – FACULTY OF MEDICINE AND DENTISTRY

INSTITUTE OF DENTISTRY AND ORAL SCIENCES

DOCTORAL DISSERTATION

EFFECTIVENESS OF DENTIN PRE-TREATMENT ON BOND STRENGTH OF TWO SELF-ADHESIVE RESIN CEMENTS COMPARED TO AN ETCH-AND-RINSE SYSTEM; AN *IN VITRO* STUDY

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by

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Date____________________

DECLARATION

I declare that I performed this dissertation independently under the supervision of my supervisor and that I stated all used literature and other sources.

Signature:

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Table of Contents

1 ABSTRACT

1.1 Objectives

This *In Vitro* study aimed to evaluate the tensile bond strength of two self-adhesive resin cements after two dentin surface pre-treatments, and also to analyze the cement/dentin interface. Determining the failure type was also a part of this study.

1.2 Materials & methods

One hundred and twelve caries-free permanent third molars recently extracted from patients aged 20–30 years because of pericoronitis were collected. Ethics approval from the Ethics Committee at Palacky University and verbal consent of the donors were obtained. The teeth were then cleaned with an ultrasonic scaler and stored in a 10% formalin solution for one week after extraction, and then the teeth were kept in distilled water until use. The teeth were tested within a maximum of one month after extraction.

The roots of the teeth were embedded in auto-polymerized acrylic resin to facilitate handling during the cutting and testing procedures.

The teeth were distributed into seven groups (n=16). Maxcem Elite Chroma (MAX) and Relyx U200 (RLX) were used without pre-treatment or with two dentin pre-treatments (polyacrylic acid or phosphoric acid). A conventional etch-and-rinse

(EAR) luting cement, NX3 Nexus (NX3), was used as an external control group, since the EAR system is still considered as the gold standard for dental adhesion. Before testing, all specimens were stored in distilled water for 24 hours. Three specimens from each group were prepared for scanning electron microscopy observation (SEM). A tensile bond strength test (TBS) was performed for the remaining samples. The data were statistically analyzed using the Kruskal-Wallis test and Pairwise comparisons using the Wilcoxon rank sum test.

1.3 Results

MAX without pre-treatment and with phosphoric acid etching attained statistically similar bond strengths to NX3 (P>0.05). There was a statistical difference (P=0.00488) between RLX without pre-treatment (5.62 MPa) and NX3 (10.88 MPa). Phosphoric acid pre-treatment increases the bond strength values of RLX to a strength that is comparable to NX3 (P>0.05). The lowest tensile bond strength (TBS) was attained after the application of polyacrylic acid with MAX (1.98 MPa). No statistical differences were found between the RLX bond strength values after polyacrylic acid treatment and RLX without pre-treatment or NX3 (P>0.05). SEM observations disclosed an enhanced potential of the self-adhesive cements to infiltrate into dentin tubules and form resin tags when applied after phosphoric acid pre-treatment. The failure mode was dominantly adhesive.

1.4 Significance

On dentin, the self-adhesive resin cement MAX might be an effective alternative to conventional resin cement. Etching the dentin with phosphoric acid does not have a negative effect on the bond strength of MAX to dentin. On the other hand, phosphoric acid improved the bond strength of RLX when compared to EAR cement.

1.5 Keywords

Adhesion; Bond Strength; Dentin surface pre-treatment; Lithium disilicate; Self-adhesive resin cements

2 INTRODUCTION

Adhesive dentistry is at this time considered as one of the foremost important fields in restorative dentistry. The physical and mechanical properties of adhesive materials, as well as the evaluation of adhesion mechanisms and the quality of adhesive bonding to dental tissues and other dental materials, are the focus of scientific research. Adhesive materials come in a wide range of properties. Particularly in recent decades, as the desire for simpler adhesive materials and techniques has remained consistent, examining these materials and demonstrating their dependability and suitability for adhesive operations is still critical.

2.1 Adhesion principles in dentistry

Since Buonocore discovered selective enamel etching for improving the adhesion of acrylic filling material to enamel in 1955, the science of adhesion in dentistry has moved quickly forward (1). Without adhesive approaches, restorative dentistry is nowadays no longer conceivable. Adhesion is a process in which the "adherent" (or substrate) develops a "interface" with the "adhesive" that has been applied. Enamel, dentin, composite, ceramic, metal, and many other materials are examples for adherents used in dentistry (Figure 1)(2). The adhesives can contain one or multiple interfaces to mediate the bonding of, for example, ceramic to metal or ceramic restorations to dental tissues (3).

Figure 1: Shows the main process of adhesion in dentistry (Xiao-zhuang Jin *et al*, 2016)

Several types of adhesion can be differentiated in restorative dentistry:

- Chemical adhesion happens when chemical bonds between materials have been formed, like with Glass-ionomer cements, in which the carboxyl groups of polyalkenoic acid have true chemically interaction with calcium at the hydroxyapatite (HAp).
- Macro-mechanical adhesion is a type of bonding that utilizes the macroscopic surface irregularities of macroretentive preparations. A typical example is the cementation of fixed bridges and crowns with zinc phosphate cements.
- Comparably, micro-mechanical adhesion utilizes the microscopic surface irregularities. Good examples for this are composite resins and the accompanied adhesive systems (4)(5)(6).

Since 1878 and for more than 100 years , the water-based zinc phosphate cement with its macroretentive approach was the most commonly used luting agent. However, many efforts were placed to develop a more efficient cementing material because of its lack of adhesion to tooth structure, high dissolution in the mouth, potential for chemical and thermal injury to the pulp, and minimal anti-cariogenic effect (7). Different types of cements then have been introduced to the market. That included many types as zinc polycarboxylate and glass ionomer-based cements. Glass ionomers possess the ability to chemically bond to the tooth tissues, but they are susceptible to early moisture contamination, resulting in microcracks, in addition to the potential of elastic deformation in areas of high masticatory stress because of its low modulus of elasticity (8).

Cements as zinc phosphate, polycarboxylate, and conventional glass-ionomer cements set through an acid-base reaction. The lifetime success of these traditional luting agents is well recognised, but only if the concept of a proper geometric form of tooth preparation that limits the paths of displacement of the restoration is respected. However, an ideal axial wall convergence of 2 to 5 degree taper is rarely achieved. Instead a more realistic 10 to 22 degrees was found amongst practitioners over four decades, according to a systematic review (9).

In general, these cements can be used for the cementation of metal, metal-ceramic and non-glass based ceramic restorations, but are not recommended for luting resins and glass-based ceramic restorations (10)(11).

As an alternative to acid–base reaction cements, and in an attempt to improve the properties of the luting agents, resin cements were introduced in the mid-1980s, these materials have a setting reaction based on polymerization (12).

The basic mechanism of composite resin adhesion to dental tissues is micromechanical and based on an exchange process in which inorganic tooth component (HAp) is replaced with synthetic resin which infiltrates the collagen fibres to form a mixed layer (hybridization) that ensures adhesion. This bonding mechanism is clearer for dentin because of the higher proportion of collagen (13).

2.2 Human dentin as a bonding substrate

In order to better understand how the resin infiltrates the collagen fibres during hybrid layer formation and degradation over time, it is essential to understand the basic structure and composition of dentin, with special focus on the organic matrix, and the structural changes that occur during the adhesive procedures (14).

Dentin is a complex biocomposite structure, defined as a mineralized collagen matrix that contains c. 30–50 vol% organic material, c. 40 vol% inorganic materal (hydroxyapatite) and c. 20 vol% of water (15). As opposed to enamel, dentin is a humid and more organic substrate (Figure 2).

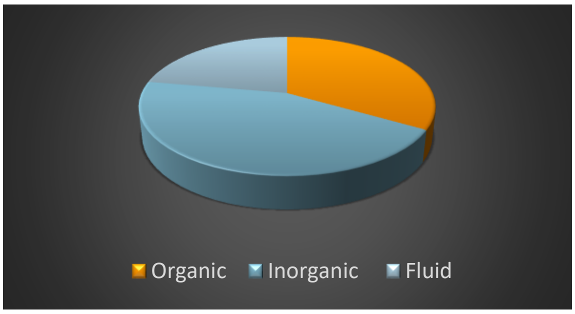

Figure 2: Shows the composition of dentin

The dentin composition varies in different regions of the tooth, depending on its proximity to the pulp, patient’s age, as well as whether the matrix is demineralized or caries affected/infected. These differences have a significant impact on the mechanical properties of dentin (16)(17). Due to these differences and complex histology, adhesion to dentin has been considered one of the most challenging and less predictable missions in adhesive dentistry.

Type I collagen makes up 90% wt of the protein content in the dentinal collagen structure. The collagen has a quaternary structure which has a triple helix with an arrangement that makes it very stable and not easily degraded (Figure 3) (18), but once it is, it cannot be replaced due to their poor capacity to regenerate, unlike insoluble collagen in other systems in the body (15).

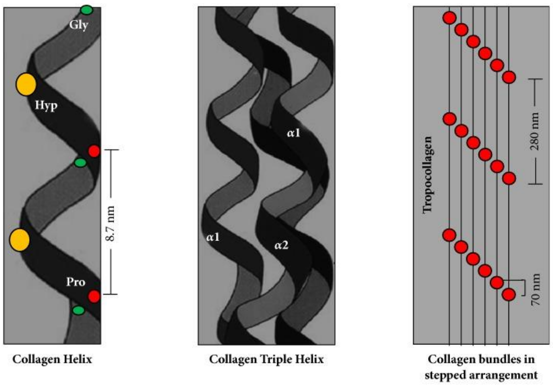

Figure 3: Shows the triple helix structure of the collagen type 1 in dentin (Betancourt DE et al, 2019)

Because dentin collagen does not metabolically turn over, the numbers of cross-links within collagen molecules accumulate with age and can influence the mechanical properties of collagen fibrils (Figure 4) (19) . These cross-links are responsible for the ability of dentin collagen to be acid-etched during bonding procedures without denaturing its collagen when normal 15 s etch has been applied (20)(21)(22)(23)(24)

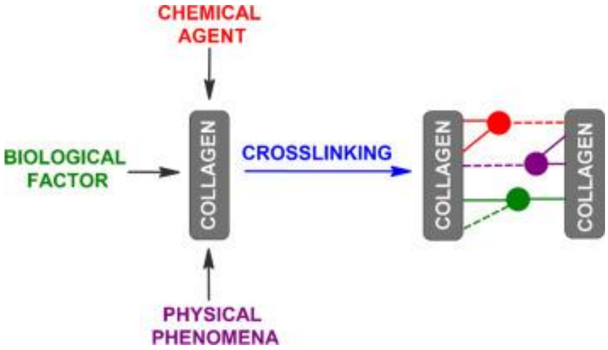

Figure 4: Shows the cross-links within collagen molecules in dentin (Adamiak et al, 2020)

The dentin has a group of inverted-cone shaped tubules that run in a radial arrangement from the pulp to the dentinoenamel junction (DEJ) surrounded by intertubular dentin. The larger diameter of these tubules face the pulp (25). Garberoglio and Brännström in 1976 (26) measured the area occupied by the tubules and the tubular diameter in 30 extracted teeth. Close to the pulp, the number of tubules was 45,000/mm^2 and their diameter 2.5 µm. In middle dentin, the number of tubules was 29,500/mm^2 and the average diameter was 1.2 µm. In superficial dentin, the area occupied by tubules was 20,000/mm^2 and the average tubule diameter was 0.9 µm (Figure 5) (15)(27). The contents of water decrease 20-fold from deep to superficial dentin. The mean tubule volume in coronal dentin is 10% of the entire dentin volume, while near the DEJ it is 4% and increases to 28% near the pulp.

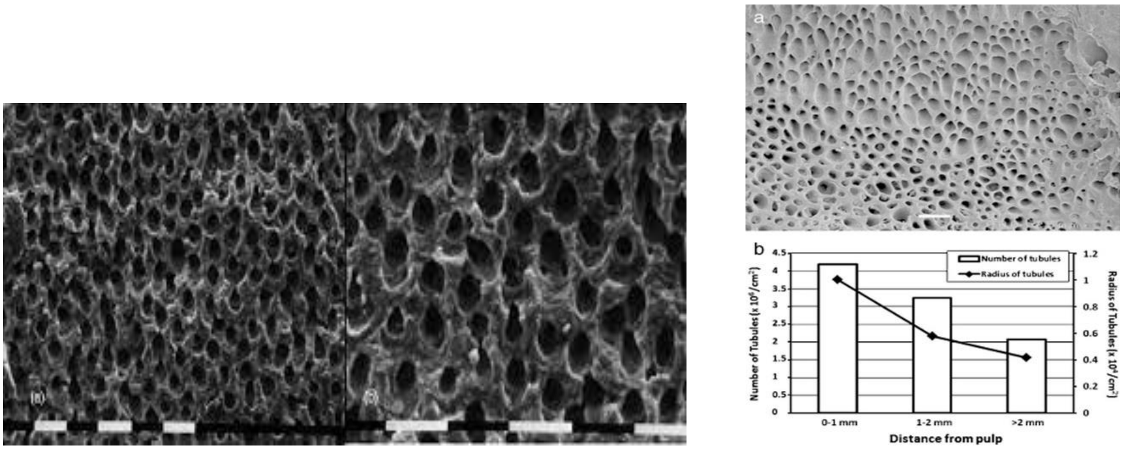

Figure 5: Shows the dentin structure and the relationship between the number and the diameter of tubules and the distance from pulp (Kontakiotis et al, 2015, Tjäderhane et al, 2012)

In the vital dentin the flow of fluid from the pulp to the dentinoenamel junction is the result of a slight but constant pulpal pressure, which is estimated to be 25–30 mm of Hg (28). In addition, dentin contains extensions of the odontoblast (odontoblastic processes) (Figure 6)(29) and intra-tubular collagen fibers in deeper areas, less frequently in middle and superficial dentin. These characteristics, which we sometimes as clinicians fail to notice, certify the greater challenge when an adhesive procedure is applied on deep dentin compared to one applied on more superficial dentin.

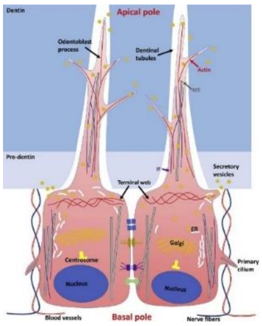

Figure 6: Shows odontoblasts extensions in dentin (Chang et al, 2019)

Furthermore, there are compositional differences between the crystalls of hydroxyapatite in enamel and dentin. Enamel crystallites are larger and placed in a more regular and parallel oriented pattern, whereas dentin hydroxyapatite crystals are smaller and placed in a crisscross arrangment inside the organic matrix, making micro-mechanical interlocking with dentin more challenging. These smaller size and crisscross orientation of the hydroxyapatite crystallites, on the other hand, enhance the chemical bonding to dentin (30).

Dentin degree of mineralization is increased as a result of physiologic changes caused by dentin aging or in reaction to carious lesions and other aggressive stimuli, resulting in increased dentin thickness and decreased dentin permeability. (31)(32). Dentin permeability affects the adhesion process, hence a decrease in permeability with age may have a direct impact on dentin bond strengths. (33).

- **The role of the dentinal smear layer in the adhesion process**

The smear layer on dentin is an adherent layer of organic and inorganic matter when tooth surfaces are cut with rotary or hand instruments (34). The smear layer found in deep dentin contains more organic material than those found on the superficial dentin (35).

When discussing the adherence process to tooth substances, the smear layer must be addressed as a significant component of adhesion. Furthermore, because existing adhesives are classified based on their interaction mode with the smear layer, it is essential to consider the changes that occur inside the smear layer during the bonding process. Usually after dental tissues preparation, a 0.5 - 5 μm thick smear layer is formed on the prepared surface, filling the orifices of the dentinal tubules. This layer contains bacteria, saliva, blood cells and denaturated collagen (36)(37)(38). The smear layer generates smear plugs by filling the orifices in the dentinal tubules, however it is not always securely adhered to the dentin samples (Figure 7) (39)(36)(40).

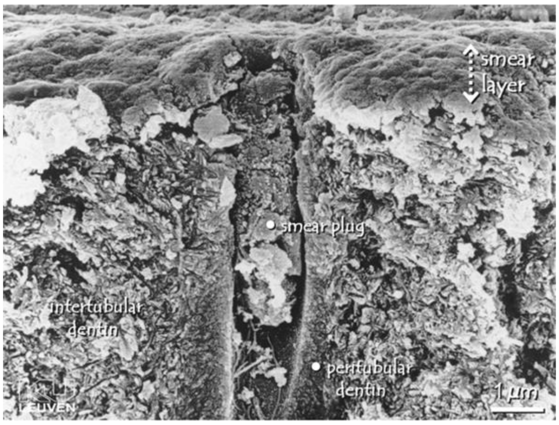

Figure 7: Shows the smear layer on dentin (Van Landuyt et al, 2005)

Dentin permeability is reduced by up to 86% when the smear layer is formed (31). The cohesive strength of the smear layer is about 5 – 10 MPa (41)(31). Although the smear layer reduces water perfusion on the surface of cut dentin, the submicron porosity within it permits dentinal fluid to diffuse (31). This dentinal fluid can be critical for adhesion (42).

The bond strength is also influenced by the thickness of a smear layer. A thin smear layer is preferable for self-etching adhesives (31). The early bonding systems such as Scotchbond Dual Cure were applied directly to smear-layer-covered dentine because it was thought to protect the pulp. The resin was unable to penetrate through the smear layer and hence gave very low bond strengths (*c.* 5–10 MPa), which reperesents the cohesive strength of the smear layer. When both sides of the failed resin–dentine bonds were checked, smear-layer material on both sides was found. As a result, the 'bond strength' was actually a measure of the cohesive strength of the smear layer, which was split into upper and lower parts (43). Dentin must be appropriately conditioned to eliminate or modify the smear layer and allow monomer diffusion into the surrounding, partially demineralized collagen matrix in order to achieve high bonding strength and seal (37).

2.3 Resin cements in dentistry

Resin luting agents nowadays are used for luting most of the indirect restorations. These resin luting agents have the capacity to bond both to the tooth and to the restoration. This integration has been known to reduce microleakage at the restoration tooth interface, and also to lessen post-operative sensitivity, marginal staining, and recurrent caries (44).

Usually resin-based composite cements consist of polyurethane matrix or a bis-GMA/TEGDMA (2,2- bis[4-(2-hydroxy-3-methacryloyloxypropoxy) phenylpropane / triethyleneglycol dimethacrylate) in which micro-filler particles (0.04-0.2μm) of quartz are immersed. To achieve radio-opacity, heavy metals such as zinc, barium, strontium, or yttrium are added into the glass. A good bond between the inorganic filler and the organic matrix is required for a composite to have successful properties. For this reason, during manufacture, a coupling agent compound is applied to the filler particles to form this bond. This agent, which known as silane, is an organic silicon compound that allows a chemical bond to develop between the resin matrix and the fillers. A chemically initiated process, photo-polymerization, or a combination of both can be used to polymerize resin composite cement. (45)(46)(47).

Resin composite cements have a high compressive strength, are tensile fatigue resistant, and are nearly insoluble in the mouth. Chemically, they can attach to resin composite restorative materials as well as to silanated porcelain. Resin composite cements have become the adhesive of choice for esthetic type restorations, such as resin composite inlays and onlays, all-ceramic inlays and onlays, veneers, crowns, FPDs, and fiber-reinforced composite restorations, due to their ability to adhere to multiple substrates, high strength, insolubility in the oral environment, and shade-matching potential (46)(48)(49).

2.4 Classification of dental resin cements and their clinical application

Resin cements may be classified by their adhesive scheme: etch-and-rinse (total-etch), self-etching, and self-adhesive (50). The self-adhesive resin cements may be referred to as "all-in-one" resin cements or universal cement (51).

They can also be classified according to their polymerization mechanisms into light-cured, chemical-cured, and dual-cured (50) (Figure 8).

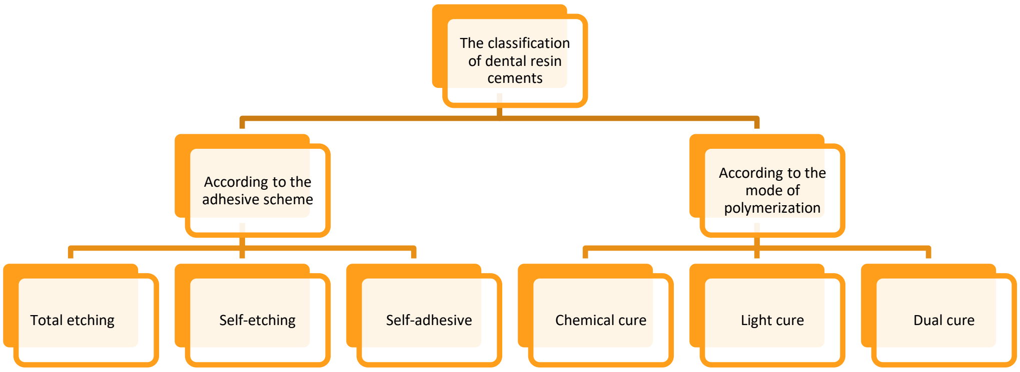

Figure 8: Shows a classification of resin cements in dentistry

Additionally, they can be classified according to the number of clinical steps and the way they interact with the smear layer (31).

2.4.1 **Etch-and-rinse system**

Total Etch is a multi-step approach that includes separate etch-and-rinse phases. The term "total-etch" refers to a procedure in which the dentin and enamel are both conditioned at the same time. In the “selective-etch” technique enamel is selectively etched before the dentin pretreatment. An acid (usually 30-40% phosphoric acid) is applied for 15 seconds on dentin to remove the smear layer and superficial hydroxyapatite and to expose the microporous collagen network. After that, the etchant is rinsed away, then a primer is applied, followed by the application of an adhesive resin (Figure 9) (52)(53)(54).

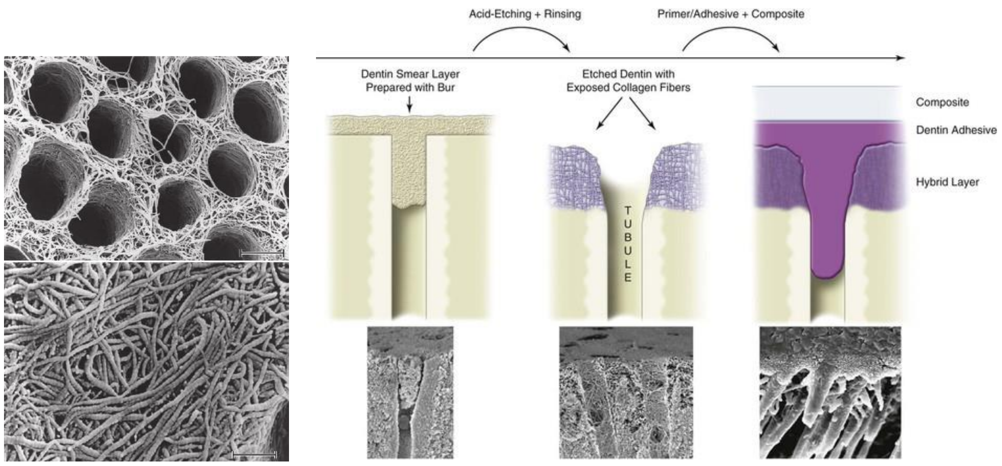

Figure 9: Shows the dentin after acid etching (exposed collagen fibrils) and after the application of primer/adhesive (Perdigão et al, 2018)

The adhesive system is defined as three step etch-and-rinse adhesive if etching, priming, and adhesive application are performed in separate clinical phases. There are also two-step etch-and-rinse adhesives that have the primer and adhesive combined in one application step. The primer solvent within etch-and-rinse adhesives is an important factor affecting the handling and performance properties of etch-and-rinse adhesives (55)(56)(57). There are ethanol-based, acetone-based and water-based primers. The highly technique-sensitive “wet-bonding“ technique should be used when using an acetone-based primer. The less technique-sensitive “dry-bonding“ technique, by applying a gentle stream of air-drying following the rinsing step of the acid, should be used when a water/ethanol-based primer is used,. The adhesive is then applied to the preparation to adhere the cement to the tooth (53)(58)(13).

This group has the highest cement-to-tooth bond strength but also requires the most steps to bond ceramic, composite resin, or metal to tooth structures. Because each step represents a potential contamination point, this multi-step procedure is considered complex and may decrease the bonding performance (50).

These cements and the adhesives used with them can be light- or dual-cured (59).

Examples include: RelyX ARC (3M ESPE); Variolink II (Ivoclar Vivadent Inc.); and Choice 2 (BISCO, Inc.) (Figure 10).

Figure 10: Shows some resin cements which use EAR system

2.4.2 Self-etching system (Etch-and-dry)

This system has been introduced to the market in order to simplify the adhesive systems avoiding the technique sensitivity of the etch-and-rinse bonding technique (Figure 11) (60). Self-etching adhesives do not include a separate etching step; they consist of a complex mixture of functional monomers that were grafted with one or more carboxylic or phosphate acid groups, to be able to simultaneously condition and prime dentin. Also they include solvents and water (61)(53).

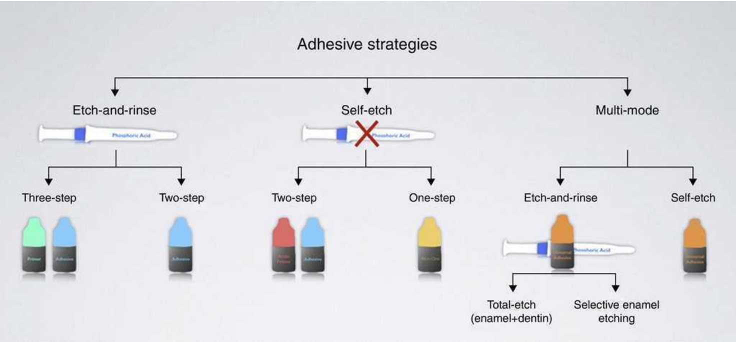

Figure 11: Shows the number of clinical steps for EAR and self-etching systems (Sezinando 2014)

The infiltration of monomers occurs 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 (13)(62). Self-etching adhesives affect the smear layer without removal of the dissolved calcium phosphates since the rinsing step is eliminated (Figure 12) (58)(63).

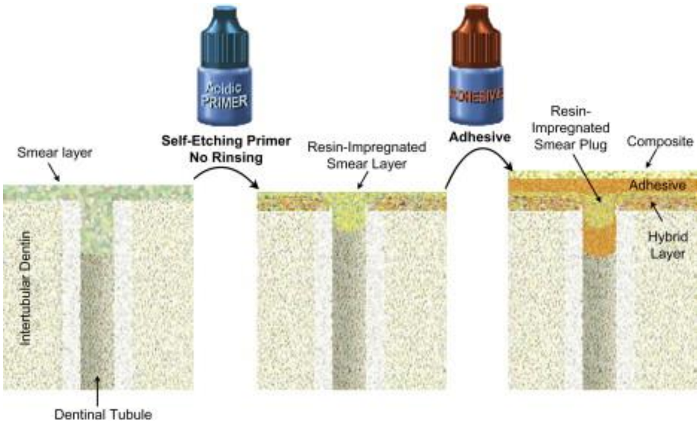

Figure 12: Shows the clinical process of self-etching adhesive system (Perdigão 2007)

Self-etching adhesives are currently available in two-step or single-step systems, the latter also known as all-in-one adhesive systems (64). Self-etching adhesives are classified depending on the pH of the system as strong (pH≤1), intermediate strong (pH≈1.5), or moderate (pH≥2). The pH of strong self-etching adhesives usually is 1 or below 1. This high acidity results in rather a deep demineralization effect (65). The bonding process and interfacial ultra-morphology of strong self-etching adhesives are comparable to those of etch-and-rinse adhesives. These strong self-etch adhesives have been linked to poor dentinal bonding performance in the lab and in clinical practice, particularly at the dentin substrate (66)(67)(68). This could be due to the presence of soluble calcium phosphates in the interfacial zone (66). Furthermore, the high concentrations of acidic resin monomers cause these adhesives to function like hydrophilic permeable membranes, allowing water to flow from dentin to the surface (61)(69)(25). In addition, the residual solvent that remains within the adhesive interface can impair the bonding process (13)(70).

Intermediate strong or moderate self-etching adhesives have a pH of around 1.5. The two-fold build-up of the dentinal hybrid layer is the most common. Despite the fact that the hybrid layer's superficial layer has been totally demineralized, the base still retains undissolved HAp (13). Mild self-etching systems have a pH of around 2 and are usually two-step systems. They dissolve fewer calcium phosphates and demineralize dentin up to the depth of 1 μm (Figure 13) (39). The presence of HAp within the submicron hybrid layer could act as a receptor for further chemical bonding, particularly for functional monomers with the capacity to bond to calcium (71).

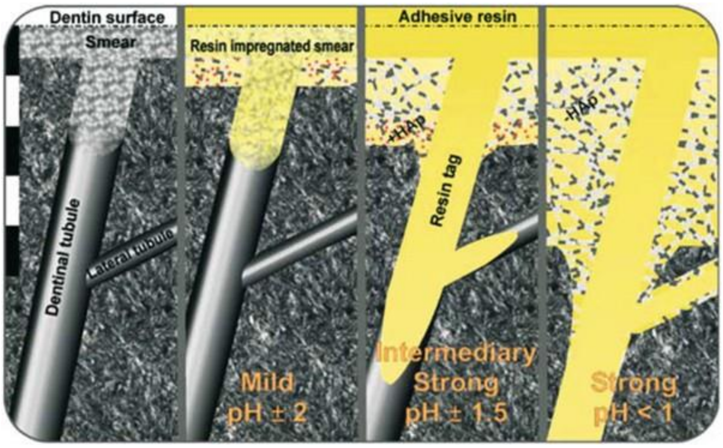

Figure 13: Shows the smear layer and the process of hybridization using self-etching system with different pH (Van Landuyt et al, 2005)

Functional monomers with specific phosphate groups, such as phenyl-P (2-methacryloxyethyl phenyl hydrogen phosphate), and 10-MDP (10-methacryloxydecyl dihydrogen phosphate) or carboxylic groups such as 4-META (4- methacryloxyethyl trimellitic acid), are capable to bond chemically to calcium of the residual HAp crystals through ionic binding (Figure 14) (72)(73)(71)(74). 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)(Figure 14)(75)(76).

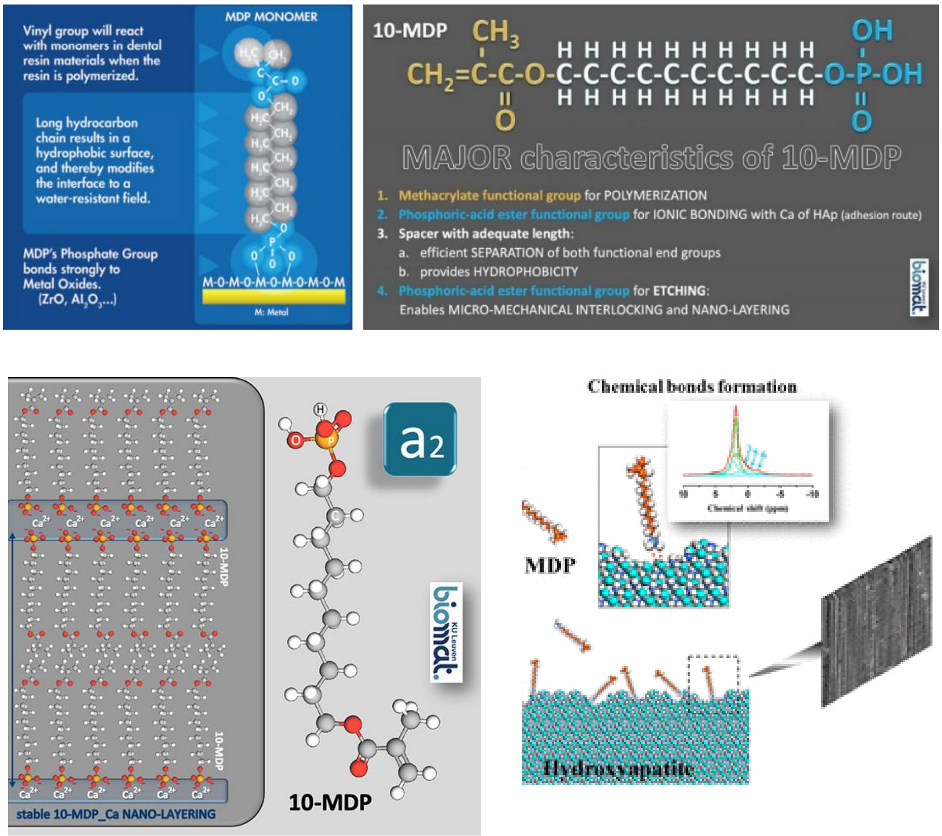

Figure 14: Shows the structure of MDP and the forming of the chemical bond with the calcium in the HAp

(Mehta et al, 2010)(van Meerbeek et al, 2020) (Han et al, 2020)

Intermediately strong adhesives have a pH of around 1.5 and are neither mild nor strong self-etching adhesives. At the enamel and dentin levels, this results in better micromechanical interlocking than moderate self-etching adhesives. The resulting demineralized layer is 2.5 μm thick and has a completely demineralized surface, while the base has residual HAp crystals that allow for chemical interactions, providing a more gradual transition of the exposed collagen network to the underlying undamaged dentin (13). Self-etch adhesives have a stronger connection to collagen than etch-and-rinse adhesives, which increases the probability of chemical bonding between remaining hydroxyapatite crystals and monomeric groups, which improves bonding (63). In terms of restoration durability, the resulting two-fold micro-mechanical and chemical bonding mechanism is thought to be favorable. The chemical bonding component gives resistance to hydrolytic breakdown, while the micro-mechanical bonding component gives resistance to sudden de-bonding stress (53)(77).

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 creates a more hydrophobic interface and allows better bond durability (78). One-step self-etch adhesives are easier to use and less time consuming than two-step self-etch adhesives, but they have a lower bonding performance (79). This could be due to their poor mechanical characteristics, decreased degree of conversion, and greater water sorption from dentin via osmosis (79)(66).

In case of cementation using self-etch systems, a self-etching primer is applied to prepare the dental substrate, then the mixed cement is applied over the primer. Bonds to dentin structure using this category of cements are almost as high as those of the total-etch cements (59).

2.4.3 **Self-adhesive system**

Simplifications to reduce clinical steps were made and resulted in the development of so called self-adhesive resin luting agents. Adhesives and composite resin were combined into one material that can adhere to tooth structure without the need for adhesive or etching process. Self-etching primers in resin cements reduce steps during application, minimizing operator errors and technique sensitivity while also promoting simplicity of use. They were introduced to dentistry as a subgroup of resin cements and have gained popularity (80). However, it is yet uncertain how the simplifications affected bonding efficacy, the nature of adhesion, and the clinical durability of the simplified resin luting agents. The morphology of the adhesive interface of these materials, in particular, is not entirely clear. Nevertheless, it is important to follow the manufacturer's instructions during adhesive cementation, including use of the manufacturer's adhesive and resin cement combination, because investigators have observed incompatibilities between some dual-cure resin cements and simplified adhesive systems (81).

RelyX Unicem, the first self-adhesive resin cement on the market, has been manufactured since 2002 (Figure 15).

Figure 15: Shows the first self-adhesive resin cement in the dental market, RelyX Unicem

RelyX Unicem is still a strong reference for in vitro and in vivo investigation data of other self-adhesive resin cements due to its clinical effectiveness. The numerous self-adhesive cements available in the dental market differ in their composition, working/setting time, the delivery system and number of shades available (50)(59). The main composition of these cement consist of two pastes that require hand-mixing, auto-mixing or capsule trituration. This composition is similar to the resin composite filling, where there are resin and inorganic filler part. Once the cement is mixed, it can be applied in a single clinical step (82)(80). The cement has a high initial hydrophilicity after mixing, which aids in wetting and adaption to the tooth structure. Acidic groups react with tooth calcium and metal oxides generated by ion-leachable fillers. As the acidic groups are consumed throughout the reaction, the cement becomes more hydrophobic. The adhesion obtained is due to micromechanical interlocking with tooth structure and chemical bonding between the acidic monomers and HAp crystals (59).

Because the smear layer is not removed, no post-operative sensitivity is expected, according to the manufacturer's literature. These cements are shown to be moisture resistant, and some of them can release 20 fluoride ions in the same way that glass ionomer cement can (83). The resin part of the self-adhesive resin cements is mostly a mix of conventional monomers: mono-, di-, or multimethacrylates such as Bis-GMA, UDMA, HEMA, TEGDMA etc. The particular acidic functional monomers 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) (59). The phosphate groups in these acidic monomers serve as demineralizers of enamel and dentin, while also acting as mediators for the chemical bond to calcium. With calcium, the MDP monomer generates the most insoluble salts, whereas 4- META and phenyl-P have a lower bonding potential to HAp and hence produce fewer hydrolytically stable salts (76). The pH ranges from 1.5 to 3 in newly mixed self-adhesive cements, depending on acidity and functional monomer concentration. This corresponds to the acidity of the mild self-etching primer. Then the pH rises quickly in the first hour after setting and reaches pH 7 in 24–48 hours (59)(84). Combinations of fluoroaluminoborosilicate glass, barium glass, quartz, fumed silica, ytterbium fluoride, and other fluoride-releasing fillers represent the inorganic filler part in self-adhesive luting agents. Such cements have a lower filler content than compomers and is similar to flowable resin composites, with filler percentage ranging from 60-75 % by weight (59).

Examples include: RelyX™ Unicem (3M ESPE), BisCem® (BISCO, Inc.), Maxcem Elite™ (Kerr Corporation), SpeedCEM™ (Ivoclar Vivadent Inc.).

- **Regarding the other classification of resin cements according to their polymerization mode, they can be classified into chemical-cured, light-cured, and dual-cured.**

Photo-initiators, which are activated by light, are used in light-cure resin cements (Figure 16). With this type of cement, the capacity of light to enter all areas and activate the photo-initiators is critical. When compared to other types of cements, light-curing cements have the advantage of having a longer working time. As a result, the physician can remove any extra cement before curing (85)(86). In comparison to chemical-cure or dual-cure resin cements, another advantage of light-cure cements is their color stability (85). When cementing thin, translucent ceramic restorations, light-polymerized resins are advised because they permit light to pass through to the resin cement (87).

Figure 16: Shows some light-cure resin cements (eCement®- Bisco), (G-CEM Veneer®- GC)

Dual-cure resin cements (Figure 17) contain both self-cure initiators and photo-initiators as well. Therefore, such cements are capable of being cured by both chemicals and light (49). When the ceramic material is too thick or opaque to allow light to pass through, dual-polymerized resin cements are recommended. Light-curing is still required for dual-cure resin cements to achieve a high degree of polymerization, according to studies (85)(88).

Figure 17: Shows some dual-cure resin cements (Panavia®- Kuraray), (Duo-Link®- Bisco)

Chemical-cure Resin cements are also known as "self-curing" since they polymerize through a chemical process. This indicates that for the setting reaction to begin, two components must be mixed together (89). These cements are especially beneficial in situations when light curing isn't an option. The cases with ceramic restorations that do not allow enough polymerization of the resin cement from the curing unit might be a good indication for such cements (90).

2.5 Dental ceramics as a restorative biomaterial

Gold noble alloys, due to their excellent physical and mechanical properties, were considered the gold standard for indirect restorations in prosthodontics (Figure 18) (91)(92). After that the metal-ceramic restorations which replaced the use of the gold restoration were also considered as the gold standered (Figure 18)(93).

Figure 18: Shows gold and metal-ceramic restorations (Donovan 2004)(Spear et al, 2008)

However, the need for esthetic dentistry, as well as the growing concern about the biocompatibility of dental alloys, encourage the development of new products. Because of their aesthetics, inertness, and biocompatibility, ceramics have become increasingly popular as restorative materials.

This demand for more esthetic and metal-free restorations, as well as rising metal prices, are expected to boost the number of all-ceramic prostheses even further. However, ceramics are brittle and susceptible to fracture, which is a major clinical issue (94). The feldspathic porcelain crown was the only choice for full coverage aesthetic restorations when dental ceramics were invented in the late 1800s

(Figure 19) (94). Because of the feldspathic porcelain's low flexural strength, it was almost applied only on the frontal teeth (95).

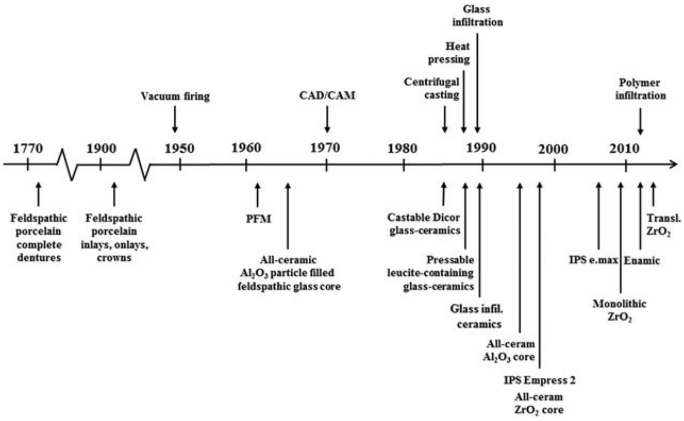

Figure 19: Shows the history of dental ceramic development (Zhang et al, 2017)

A few remarkable reinforced 4 glass ceramics have been produced throughout the years to overcome some of the drawbacks of feldspathic porcelain. The leucite reinforced ceramic was the first known reinforced feldspathic porcelain (96). Lithium disilicate (eg, IPS Empress 2) was produced as a stronger alternative to leucite reinforced ceramics (95). Lithium disilicate's physical and mechanical characteristics allowed for the fabrication of three unit fixed-partial dentures in the anterior and premolar areas (97) (Figure 20) (98).

Figure 20: Shows full ceramic bridge made form lithium disilicate replacing the first premolar (Schneider, 2016)

The insertion of metal oxides as the main component of the ceramic glassy matrix, such as the glass-infiltrated alumina ceramic, was another advancement in dental ceramics. The use of alumina as a feldspathic porcelain reinforcement was first presented in 1965, just a few years after the introduction of leucite reinforcement (99). Based on the results of several long-term retrospective studies, glass-infiltrated zirconia toughened alumina (In-Ceram Zirconia, VITA Zahnfabrik) with a marginally higher flexural strength than glass-infiltrated alumina has also been produced and indicated for similar applications as glass-infiltrated alumina (100).

2.6 Classification of dental ceramics

There are many classifications of dental ceramics, only the most important classifications, from the clinical point of view, will be discussed in this part. Modern all-ceramic systems can be categorized based on their glass and crystalline content (Figure 21) (101).

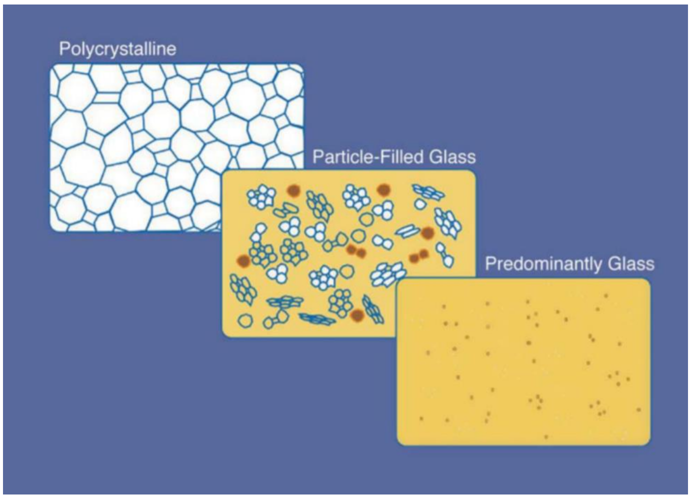

Figure 21: Shows schematic representation of three basic classes of dental ceramics according to Kelly (Kelly, 2008)

In general, high strength ceramics contain higher amounts of reinforcing crystalline content, resulting in less aesthetic restorations when compared to ceramics with higher silica content such as feldspathic porcelain or leucite ceramic (102) (Figure 22) (103). The optical and mechanical properties of the ceramic are determined by the crystalline phase's nature, crystal size, pattern of particle distribution, and other properties (104).

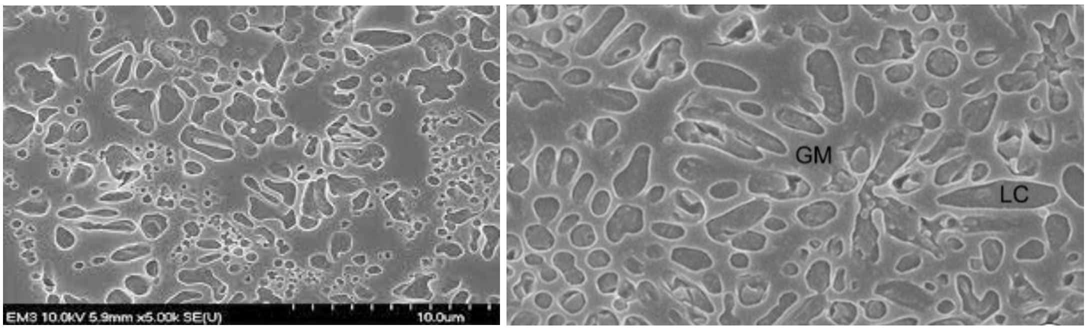

Figure 22: Shows the microstructure of leucite reinforced ceramic and the growth of crystals (LC) within the glassy matrix (GM) (Byeon et al, 2018)

Ceramics can be divided into two basic groups: silica-based ceramics and oxide-based ceramics. In general, ceramics that contain less than 15% silica are not regarded as silica-based ceramics (95). However, without a thorough understanding of ceramic compositions and properties, such a simple grouping can cause considerable misunderstanding. Therefore, Kelly made another classification, where he defined dental ceramic as a composite, or a mixture of two or more components, and he divided dental ceramics into three categories as: (1) predominantly glass, (2) particle-filled glass and (3) polycrystalline (101).

2.6.1 Predominantly glass ceramics

Glass ceramics have an amorphous glass matrix with a random network of cross-linked silica as their microstructure. This type of ceramic, as the name implies, includes a few or no filler particles, which are primarily utilized to control optical effects (101). Feldspathic porcelain and glaze that belong to the aluminosilicate glass family are examples of predominantly glass ceramics. This type of ceramic is the best at mimicking the aesthetic characteristics of enamel, making it ideal for bonded porcelain veneer fabrication and use as veneering materials over high-strength core substructures (Figure 23) (105)(106)(107)(95). On the other hand, they are considered the weakest among the other types of dental ceramics, with flexure strengths range from 70 to 90 MPa (108)(109).

Figure 23: Shows veneers made from feldspathic ceramic (McLaren et al, 2011)

2.6.2 Partially-filled glass

To improve the physical and mechanical properties of glass, filler particles are added into the mixture. Currently, crystalline or high-melting glasses that are stable at firing temperatures are employed as filler particles in ceramics. To increase their physical and mechanical properties, stronger glass ceramics like IPS Empress, IPS Empress 2/IPS e.max, In-Ceram Alumina, and In-Ceram Zirconia are reinforced with leucite crystals, lithium disilicate, alumina, and zirconia toughened alumina respectively (110)(111)(112). As a result of their improved characteristics, they can be used to make fixed partial dentures and full coverage crowns for posterior teeth (109). In-Ceram Alumina (VITA Zahnfabrik) was first introduced in 1990 and had been one of the most popular glass-infiltrated aluminum-oxide ceramics (94). InCeram Vita (IN-CERAM ALUMINA, ZIRCONIA) was made through a process called slip-casting, which involves the condensation of an aqueous porcelain slip on a refractory die (Figure 24). After firing, the porous core is glass infiltrated, which is a process in which molten glass is drawn into the pores through capillary action at high temperatures.

Figure 24: Shows the slip-casting process (Nautiyal et al, 2015)

Compared to traditional feldspathic porcelains, these materials have less porosity, fewer processing defects, better strength, and toughness. For final aesthetics, the glass-infiltrated core is afterwards veneered with feldspathic ceramic. These have excellent translucency and esthetic qualities, but have poor physical properties (95).

Given these physical weaknesses of this system and in order to be able to extend the use of ceramic restorations, a lithium disilicate-based glass ceramic (SiO_2-Li_2O) using the hot pressing procedure has been developed (Empress II, Ivoclar-Vivadent) in 1998 (Figure 25) (113).

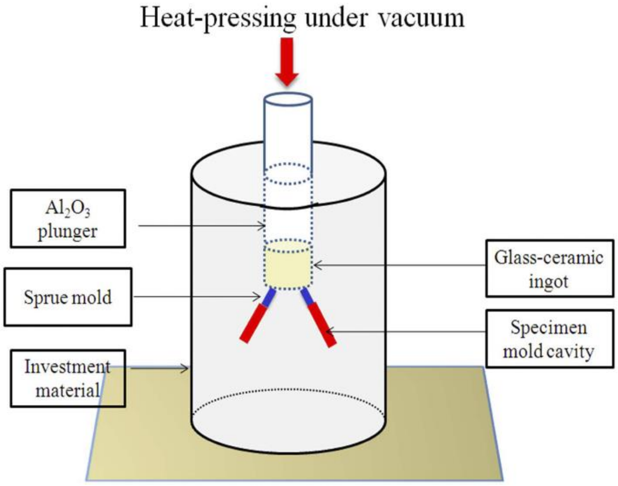

Figure 25: Shows the hot pressing technique (Wang et al, 2015)

This company also launched IPS e.max Press in 2001, which is a lithium disilicate glass ceramic with a higher lithium disilicate composition than Empress II. As a result, mechanical properties have improved (111). The ingots of lithium disilicate (IPS e.max Press) are heat-pressed into a special furnace after burning out the wax pattern (114) (Figure 26).

Figure 26: Shows the ingots of lithium disilicate (IPS e.max Press)

Four years later, IPS e.max CAD was introduced for CAD/CAM restoration in the dental clinic. IPS e.max CAD is available as blocks for chairside CAD/CAM systems (115) (Figure 27).

Figure 27: Shows blocks of IPS e.max CAD

The machineable lithium disilicate blocks have a “intermediate” crystallization that allows them to be milled efficiently in a crystalline intermediate phase (blue, translucent state). The intermediate crystallization process leads to the formation of lithium metasilicate crystals, which are responsible for the material’s processing characteristics, machineability, and good edge stability. It is after the milling procedure and the restorations are fired that they reach their final crystallized state and their high strength. The microstructure of intermediate crystallized IPS e. max CAD lithium disilicate comprises of a glassy phase with 40% platelet-shaped lithium metasilicate crystals. The length of these crystals ranges from 0.2 to 1.0 m. The microstructure of the IPS e. max CAD lithium disilicate material after post-crystallization is composed of 70% fine-grain lithium disilicate crystals embedded in a glassy matrix (114)(116) (Figure 28)(94). Clinicians with IPS e. max CAD are able to fabricate restorations during a single visit by using intraoral digital impressions and in-office milling machine (117).

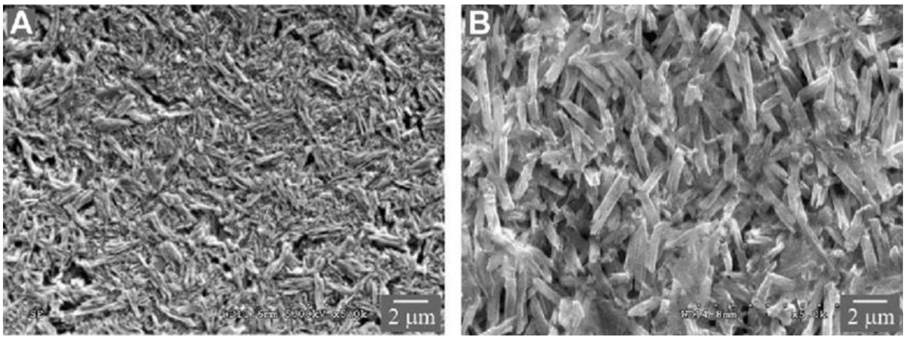

Figure 28: Shows the microstructures of lithium disilicate glass-ceramics. (A) CAD and (B) Press (Zhang et al, 2017)

After the production of lithium disilicate, and because of the fact that the elastic properties of these ceramics are not compatible with enamel or dentin substrate, and instead of increasing the crystalline phase to increase flexural strength of ceramic, some developers have taken a different approach with a recent development of a novel polymer-infiltrated ceramic (Enamic, 7 VITA Zahnfabrik), which is less brittle than other types of ceramic (Figure 29). According to the manufacturer, 75% of its volume is feldspathic ceramic and 25% polymer (an organic phase of dimethacrylate resin containing UDMA and TEGDMA). The infiltrated polymer provides a flexural strength, elastic modulus, hardness, fracture toughness, and similar properties to those of natural tooth structure.

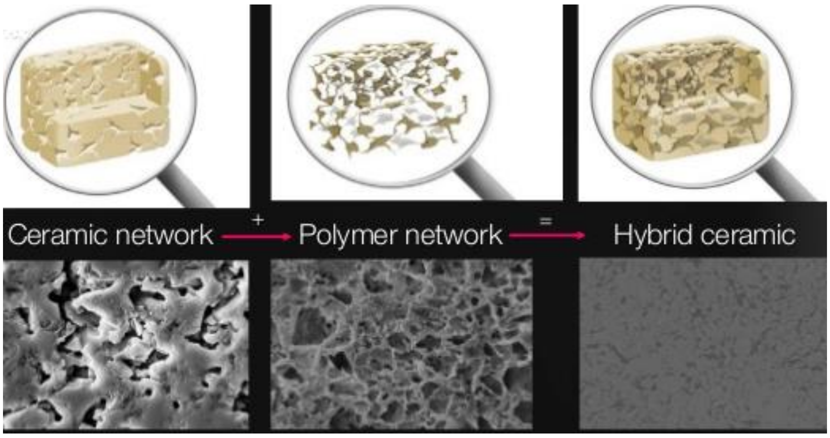

Figure 29: Shows the polymer-infiltrated ceramic (Enamic)

Also another type of resin-matrix ceramics (nano-ceramic resin) was produced. Ceramics in this group contain organic matrix highly filled with 80% ceramic nanoparticles as (Lava Ultimate®, 3M ESPE) (Figure 30).

Figure 30: Shows Lava™ Ultimate Restorative for CEREC®, 3M™ ESPE™

Although the filler loading (80 – 90 wt% or 65 – 77 vol%) in the millable resin-matrix ceramics is similar to that of polymer-infiltrated ceramic, their elastic properties and fracture behavior are quite different. In the case of polymer-infiltrated ceramic materials, the interconnectivity of the ceramic phase provides stiffness and hardness that are necessary for the resistance to plastic deformation and wear. The ductile polymer network, on the other hand, is able to effectively distribute stresses in all directions. As a result, the 3-D interpenetrating dual network of polymer-infiltrated ceramic materials possess enhanced resistances to a variety of breakdown phenomena, including contact and flexural damage as well as fatigue crack growth and wear (118)(94).

2.6.3 Polycrystalline

This type of ceramic is known as densely sintered high-purity oxide-based or polycrystalline ceramics as it does not contain silica (Figure 31) (119).

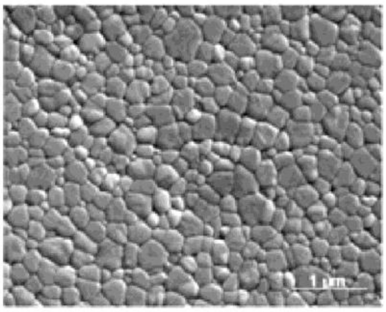

Figure 31: Shows the microstructure of polycrystalline ceramic (no silica) (De Aza et al, 2006)

Aluminum oxide and zirconium oxide are two examples of polycrystalline ceramics. The introduction of polycrystalline ceramics in which aluminum oxide or 3% yttrium-stabilised zirconia (3Y-TZP) can be used as a core material have widened the field of application in indirect all-ceramic restorations such as the fabrication of fixed partial dentures and single crowns in the molar region (120)(104)(110) (Figure 32)(121).

Figure 32: Shows a FPD in molar region made from zirkonia (Partiyan et al, 2017)

However, due to its limited translucency, Y-TZP has significant clinical shortcomings. When putting an anterior crown or shortspan FDPs in the presence of natural teeth, zirconia's opacity becomes a concern. In that case, the reflectance and light scattering do not appear natural. In order to create space for a porcelain veneer thick enough to cover an opaque zirconia core and to match the optical properties of the adjacent natural dentition, a substantial reduction of existing tooth structure is required. Furthermore, the fractures rate between the core and the veneering ceramic was higher than PFM’s restorations. In an effort to avoid veneer chipping and delamination, monolithic zirconia is often used in full arch restorations, posterior crowns and FPDs. In all these cases, the opacity of Y-TZP zirconia remains a serious issue, although the white, opaque monolithic Y-TZP restorations may be suitable for bleached teeth (122)(123)(94). The reported flexural strengths of densely sintered alumina and zirconia range from 487 to 699

MPa and 900 to 1200 MPa respectively, which easily take the oxide-based ceramics away as the strongest materials in the non-metal class (124)(125). The construction approach for high-strength alumina ceramic frameworks was described by Andersson and Odén in 1993. Despite the fact that densely sintered alumina has been utilized as a biocompatible material in the medical profession since 1964, volumetric shrinkage of 15-20% during the sintering stage made precise veneering framework fabrication problematic. Once the shrinkage could be controlled, the introduction of this high strength framework material to dentistry has made restoration of posterior teeth with ceramic crowns and fixed-partial dentures a feasible option (126). The same issue has to be considered with zirconia-based ceramic (127)(128) (Figure 33) (129).

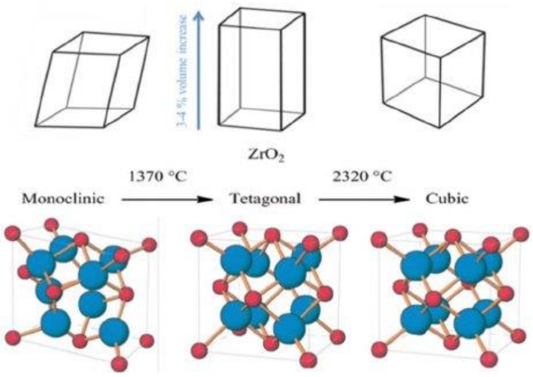

Figure 33: Shows the phases of zirconia crystalls (Brog et al, 2013)

2.7 Fixation of all-ceramic crowns

Besides the resin cements that were discussed above (self-etch and self-adhesive), also flowable composite and preheated composite can be used to lute the all-ceramic restoration when totall etch system is used (130). In comparison to restorative composites, flowable composites have a lower filler content. Depending on the kind and shape of the filler material, most flowable composites are filled between (41-53% by volume and 56-70% by weight) (131). When the filler component is raised, the flow is reduced, minimizing polymerization shrinkage but increasing the difficulty of adequately seating a restoration when used as a luting cement (132). If the filler content of a flowable composite was similar to that of a restorative composite (70 - 80 %) and the physical properties were the same, it may be a feasible alternative to resin cement. (131)(132).

Figure 34: Shows some products of flow composite

All flowable composites are not created equal. Flowable composites come in a wide variety of physical characteristics, such as flow, modulus of elasticity, flexural strength, and radiopacity, as well as filler content (132). The flexural strengths have been reported to range from 66 MPa to 145 MPa (132)(133).

Preheating resin composites simplifies restorative placement and increases monomer conversion without causing changes in optical characteristics (130)(134)(135) as is the case with light-polymerizing resin-based cements (136).

Preheating composite resins may also be an option for improving the microshear bond strength of composites on dentin (137). Furthermore, preheating hybrid composite reduces its viscosity and film thickness, allowing for easier handling, improved composite adaptability to cavity walls, and increased polymerization and depth-of-cure (138)(139). However, preheating composite to relatively high temperatures (54°C or 68°C) to promote flow and adaptability increases the volumetric shrinkage (139). Moreover, Kahnamouei *et al* found that repeated preheating of composite resin samples, up to 55–60°C for 40 rounds, resulted in more color changes compared with unheated composite resin samples (140).

Figure 35: Shows different types of composite heaters

3 AIMS OF STUDY

It has been shown that selective-etch on enamel improves bond strength of some self-adhesive resin cements to enamel. However, this carries the risk of contaminating neighboring dentin surfaces with the phosphoric acid.

It has been also suggested to apply a weak acid like polyacrylic acid (PAA), to improve the bond strength of self-adhesive cements to dentin. However, some worries exist regarding its application times and concentrations.

Therefore, the aims of this study were to evaluate the effectiveness of dentin pretreatment with PAA and/or H3PO4 using two self-adhesive resin cements compared to an etch-and-rinse system; on the tensile bond strength between Lithium Disilicate restorations and dentin; and to evaluate the cement/dentin interfaces using a scanning electron microscopy analysis (SEM). Determining the failure type was also a part of our investigations.

4 MATERIALS AND METHODS

One hundred and twelve caries-free third molars recently extracted from patients aged 20–30 years because of pericoronitis were collected. Ethics approval number 80/21 from the Ethics Committee at Palacky University and verbal consent of the donors were obtained. The teeth were then cleaned with an ultrasonic scaler and stored in a 10% formalin solution (HistoFOR BFS-L1; Pro-charitus.r.o, CZ) (Figure 36), for one week after extraction (141), and then the teeth were kept in distilled water until use. The teeth were tested within a maximum of one month after extraction.

Figure 36: Shows an extracted tooth in formalin solution

The roots of the teeth were embedded in auto-polymerized acrylic resin (Spofacryl™; SpofaDentala.s, Jičín, CZ) to facilitate handling during the cutting and testing procedures (Figure 37).

Figure 37: Shows an extracted tooth in the acrylic base before preparation

Two self-adhesive dual-cure cements, Maxcem Elite Chroma (Kerr, Scafati, Italy) and Relyx U200 (3M ESPE, Neuss, Germany), were used. A conventional resin dual-cure cement, NX3 Nexus (Kerr, Scafati, Italy) (Table 1), which requires the application of an adhesive, was also used as an external control group, since the EAR system is still considered as the gold standard for dental adhesion (142) (Figure 38).

Figure 38: Shows the cements used in this study

4.1 Preparation of dentin specimens

The crowns of the teeth were cut perpendicularly to the long axis of the tooth with a low-speed diamond saw (IsoMet, Buehler, Lake Bluff, IL) under copious water to expose a flat, middle third dentin surface. The ground dentin surfaces were observed under an optic microscope to verify complete enamel removal. When needed, the surface was further ground with the same saw until complete removal of enamel was achieved.

A standardized smear layer was achieved by grinding the flat dentin surfaces using 320-grit silicon carbide paper with a single-wheel grinder and polisher (Saphir 550, Metalco Testing s.r.o, Roztoky u Prahy, CZ) for one minute under continuous water irrigation to simulate the creation of a smear layer that would be created clinically by a red diamond bur (143) (Figure 39), the one used in crown preparation, to induce clinically relevant standardized surface roughness.

Figure 39: Shows the machine used for the standardization process of the smear layer (Saphir 550)

Teeth were kept, in distilled water during and between all experimental procedures. Teeth were randomly divided into seven groups consisting of 16 teeth each.

Group 1 (NX3) (the external control group), the dentin etched for 10 seconds with Kerr Gel etchant 37.5% phosphoric acid (Kerr, Scafati, Italy), then was thoroughly washed using a water spray for at least 30 seconds, then gently air-dried for 5 seconds. Primer (OptiBond FL, Kerr, Scafati, Italy) was applied twice, followed by air-drying for 15 seconds, then adhesive (OptiBond FL, Kerr, Scafati, Italy), then air-drying for 15 seconds (Figure 40), and after that NX3 Nexus dual-cure resin cement was applied.

Figure 40: Shows samples preparation in the Group 1 and the materials used

Group 2 (MAX-no): no dentin pre-treatment, Maxcem was applied according to the manufacturer’s instructions.

Group 3 (MAX-PAA): after the application of 25% polyacrylic acid (Ketac Conditioner; 3M ESPE, Seefeld, Germany) for 15 s, the acid was thoroughly washed using a water spray for at least 30 s, then Maxcem was applied (Figure 41).

Figure 41: Shows samples preparation in the Group 3&6 and the acid used

Group 4 (MAX-HPO): after the application of 37.5% phosphoric acid for 10 s, the acid was thoroughly washed using a water spray for at least 30 s, then Maxcem was applied.

Group 5 (RLX-no): no dentin pre-treatment, Relyx was applied according to the manufacturer’s instructions.

Group 6 (RLX-PAA): after the application of 25% polyacrylic acid (Ketac Conditioner; 3M ESPE, Seefeld, Germany) for 15 s, the acid was thoroughly washed using a water spray for at least 30 s, then Relyx was applied (Figure 41).

Group 7 (RLX-HPO): after the application of 37.5% phosphoric acid for 10 s, the acid was thoroughly washed using a water spray for at least 30 s, then Relyx was applied.

Before the bonding procedure in all groups, the moisture in the dentin was moderately removed with short, moderate blasts of air, leaving a bright surface without any fluid movement.

4.2 Preparation of Lithium disilicate-based ceramic samples

The sample design (5 mm in diameter and 10 mm in height), as a digital STL file (Figure 42).

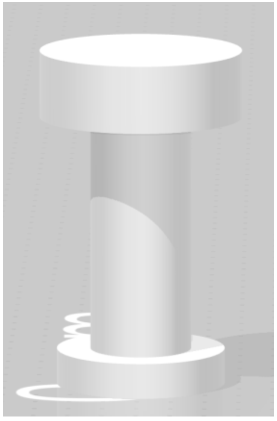

Figure 42: Shows the ceramic sample design as STL file

was prepared to be printed out by a Straumann P series Rapidshape 3D printer using Detax Freeprint resin for the digital production of the cast pattern (100% residue-free burning) (Figure 43).

Figure 43: Shows the 3D printer Straumann P series Rapidshape and Detax Freeprint resin for the digital production of the cast pattern (100% residue-free burning)

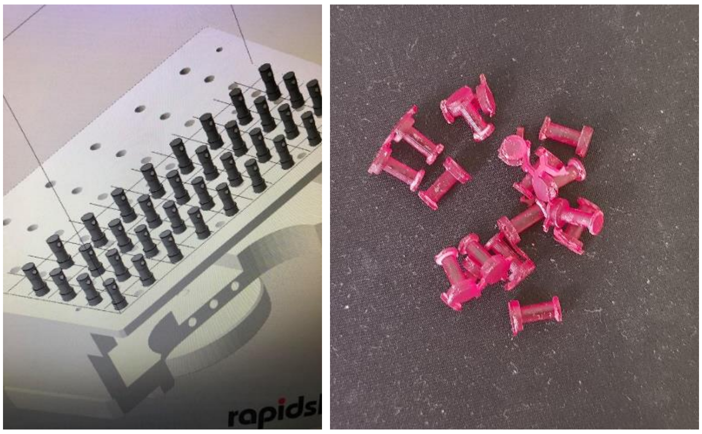

Figure 44: Shows the digital sample design and the resin sample printed out by 3D printer

then lithium disilicate-based ceramic cylinders (IPS e.max Press; IvoclarVivadent, Schaan, Liechtenstein) were made with the hot-pressing technique. The resin samples were attached to wax sprues and invested in flasks using an investment material (Pressvest speed, Ivoclar vivadent, Liechtenstein), before burning the resin out in a furnace (EP 600; IvoclarVivadent, Schaan, Liechtenstein). Afterwards, e.max Press ingots were heat-pressed into the space created by the burned resin. The ceramic samples, when cooled, were removed from the flasks (Figure 45), sprues were cut.

Figure 45: Shows the ceramic sample after deflasking

Then the samples were removed also from the investment material (IPS® Press Vest InvestmentMaterial; IvoclarVivadent, Schaan, Liechtenstein), smoothed and polished according to the manufacturer's instructions.

The teeth and the ceramic blocks were examined under digital optical microscope (Keyence VHX-5000, Belgium) and only intact samples were included in this study. Before the cementation process, the ceramic samples were etched with 9% hydrofluoric acid (Porcelain etch; Ultradent Products, Inc, Cologne, Germany) (Figure 46) for 20 seconds, then rinsed with air/water for 30 seconds. The samples were then immersed in 96% alcohol (Ethanolum 96%; Fagron. CZ) and then subsequently in an ultrasonic bath (ZZlinker,LK-D32, China) for 5 minutes (141) (Figure 47), for better removal of the product residues after acidic conditioning.

Figure 46: Shows the etching hydrofluoric acid gel

Figure 47: Shows the ultrasonic bath

After that, a silane (Ceramic silane; Ultradent Products, Inc, Cologne, Germany) (Figure 48) was applied to the etched ceramic surfaces two times with a microbrush.

Figure 48: Shows the silane used to prepare the ceramic surface

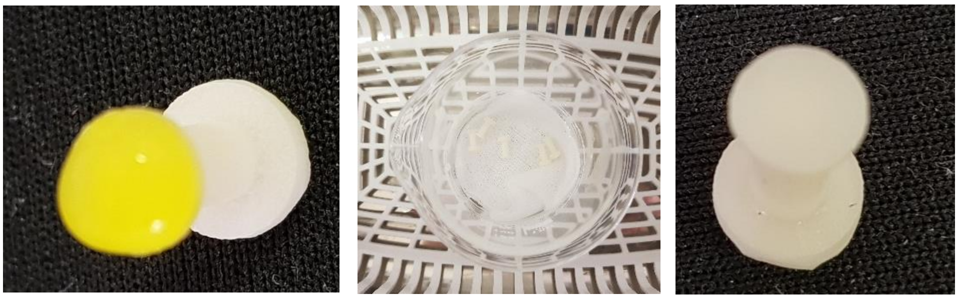

Figure 49: Shows the application of hydrofluoric acid, ultrasonic cleaning with alcohol, and silane application to prepare the ceramic surface

4.3 Cementation procedures

The ceramic samples were cemented to the dentin surfaces using the different cements, according to the mentioned application in the table 1 (Figure 50).

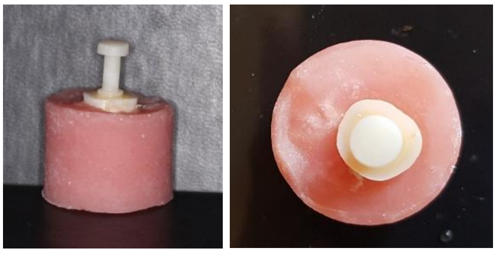

Figure 50: Shows a study sample after the fixation process

The bonding of the ceramic samples was achieved under a static load (250 g) until complete setting to simulate and standardize finger pressure (144). This was done by means of a metallic tool to create a uniform resin thickness (Figure 51).

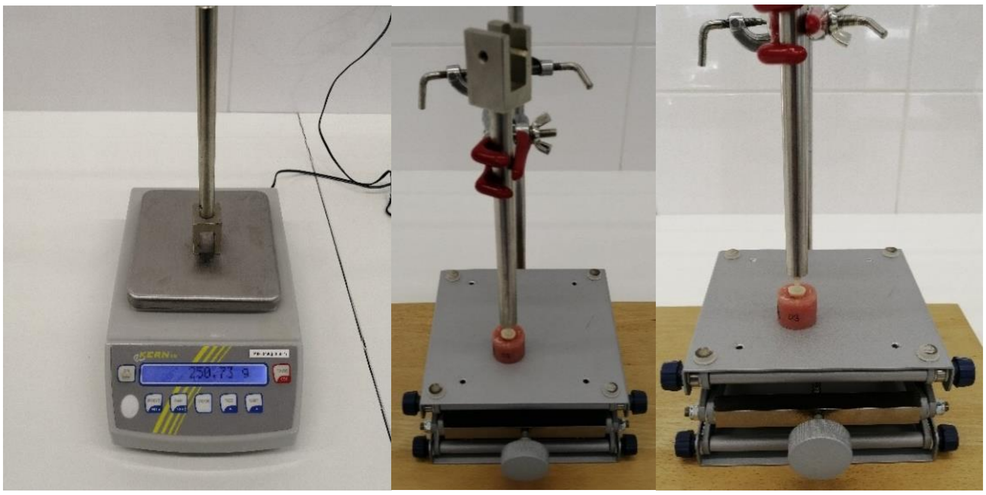

Figure 51: Shows a metallic tool used to create a uniform resin thickness

The seating force was applied for the first 6 min until complete setting of the cement leaving the material to set in the self-curing mode.

During that period, the excess cement was removed with microbrush and scalpel blade. Then photoactivation was performed with a polymerization light unit (VALO; Ultradent Products, Inc, Cologne, Germany) (Figure 52) for 20 s (60 s in total) from each side with a power density of 1000 mW/ cm^2 which was verified before use with radiometer.

Figure 52: Shows the lamp used for light curing the cement after fixation

After cementation, all the samples were stored in distilled water for 24 h at 37 °C before the tensile bond strength tests were performed.

4.4 Tensile bond strength test and fracture analysis

Tensile bond strength tests were performed for 13 specimens in each group using a load cell of 1 KN at a cross-head speed of 0.5 mm/min until failure using a Zwick/Roell Universal Testing Machine (Zwick/Roell, Ulm, Germany) (Figure 53) (Figure 54).

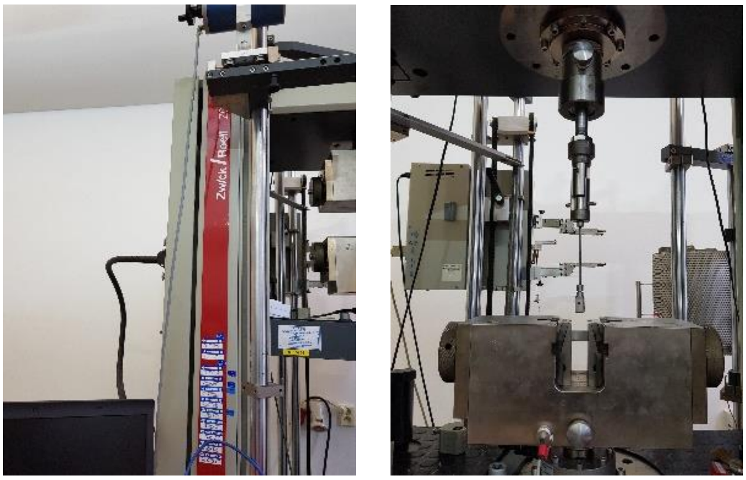

Figure 53: Shows the machine used for the tensile test

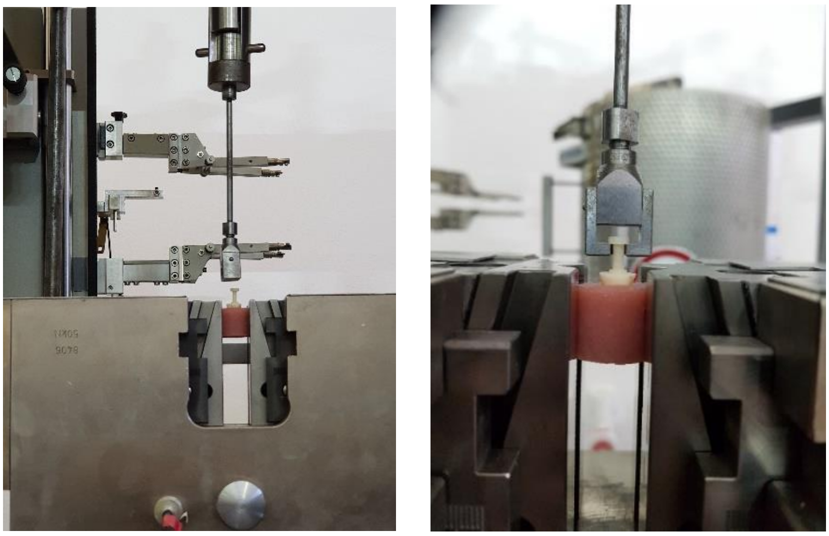

Figure 54: Shows the test performed by the Universal Testing Machine (Zwick/Roell, Ulm, Germany)

The TBS was expressed in MPa, and derived by dividing the force that was imposed (N) at the time of fracture by the bond area (mm^2) (145). After debonding, the dentin and ceramic surfaces were examined under an optical microscope at 20X to analyze the failure types. The failure types were classified as follows: adhesive (failure at the resin/dentin or resin/ceramic interface), cohesive (failure in the dentin or ceramic, or within the luting cement itself), or mixed (146) (Figure 55).

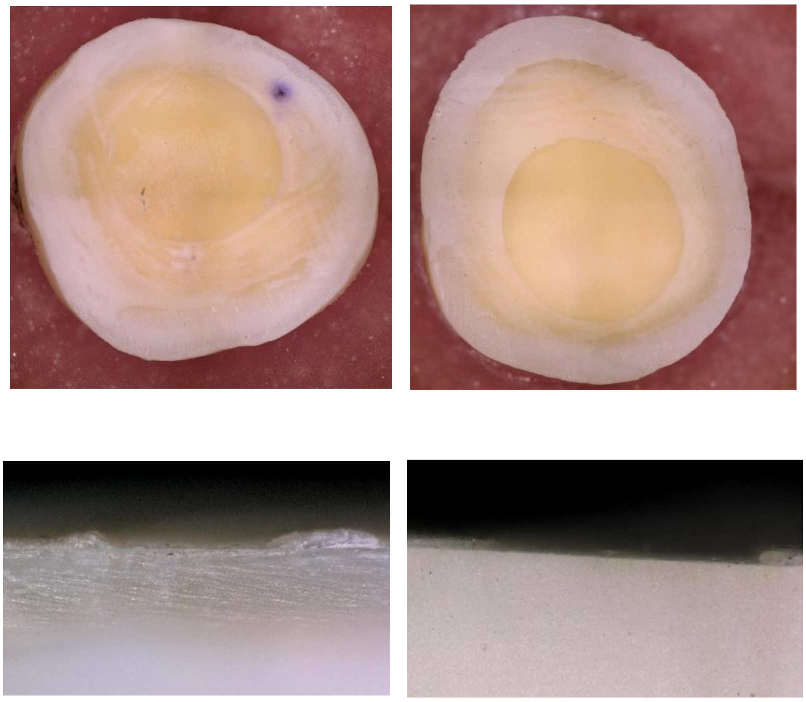

Figure 55: Shows the mixed failure A, and the adhesive failure B

4.5 Sectioning and interface evaluation under SEM

For examination under SEM (Tescan VEGA3 LMU, Brno, CZ) (Figure 56), the bonded teeth (n=3) were cut mesiodistally by a low-speed diamond saw (IsoMet, Buehler, Lake Bluff, IL) under copious water. Then the dentin sides of all the samples were etched with H3PO4 for 5 seconds and washed with distilled water for 30 seconds. After that, the samples were immersed in sodium hypochlorite 2.5% for 3 minutes, then washed with distilled water for 30 seconds. The samples were then dehydrated in a graded series of ethanol solutions (147). The samples were attached on the SEM holders by conductive carbon tape. All the specimens were analyzed using the SEM at an electron-accelerating voltage of 5 kV to evaluate the dentin/cement interface.

Figure 56: Shows the SEM microscope used in this study Tescan VEGA3 LMU

SEM observations for the dentin-resin interfaces were performed at different magnifications (800x, 3000x, 6000x). However, only one magnification (6000x) is demonstrated in the results of this study.

4.6 Statistical analysis

Descriptive statistical methods were used for the statistical analysis, especially sample mean, standard deviation, coefficient of variation, and median. The normality of the data samples was tested using the Shapiro-Wilk test and homoscedasticity was tested using the Bartlett test. The Shapiro-Wilk test detected that the data of some groups were not normally distributed. Therefore, the

Kruskal-Wallis test followed by a pairwise comparison using the Wilcoxon rank sum test were used to test the influence of dentin conditionings and different types of cements on bond strength and, in addition, to compare the median values among the groups (a=.05) (Figure 57). The statistical analysis of the data was performed in R 3.6.2 (Vienna, Austria).

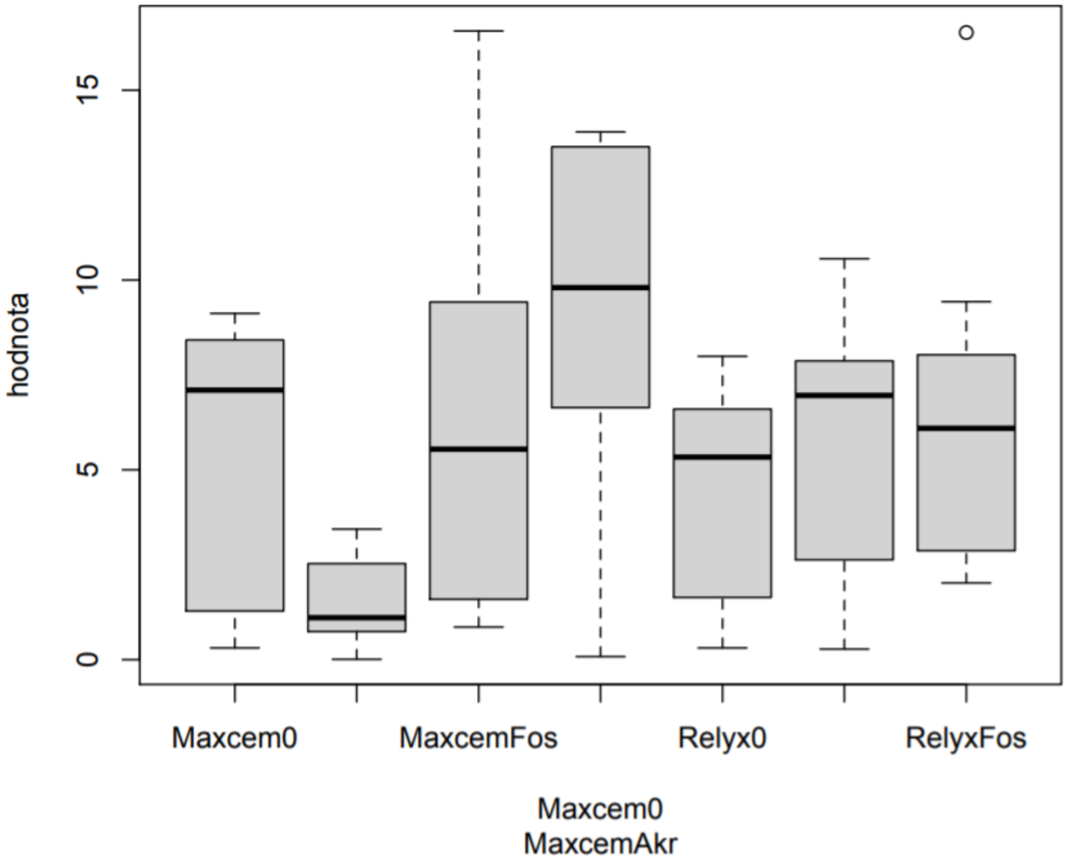

Figure 57: Shows distribution of data in a box chart. The horizontal line in the box represents the median value

5 RESULTS

The tensile bond strength results among all the groups are summarized in Table 2. The highest mean value was observed for NX3. The bond strength obtained for MAX after the pre-treatment with PAA was the lowest mean value, and showed statistical differences from all the other groups ($P < 0.05$).

No statistical difference was found when MAX-no and RLX-no were compared.

The mean value of NX3 (the control group) was higher than that of RLX-no ($P = 0.0488$). The dentin pre-treatment using phosphoric acid ameliorates the TBS of the RLX to be compared to NX3.

No statistical differences were found between RLX-HPO, MAX-HPO, and NX3 ($P = 1.000$).

Concerning SEM observations, the cement/dentin interface for each pre-treatment with the cements that were used is illustrated in Figures 58, 59, and 60. Only the etch-and-rinse groups (NX3, MAX-HPO, RLX-HPO) demonstrated the infiltration of resin tags into the dentinal tubules. In contrast, the groups with untreated dentin or with PAA pre-treatment showed no resin infiltration, except a few shorter tags with RLX-PAA.

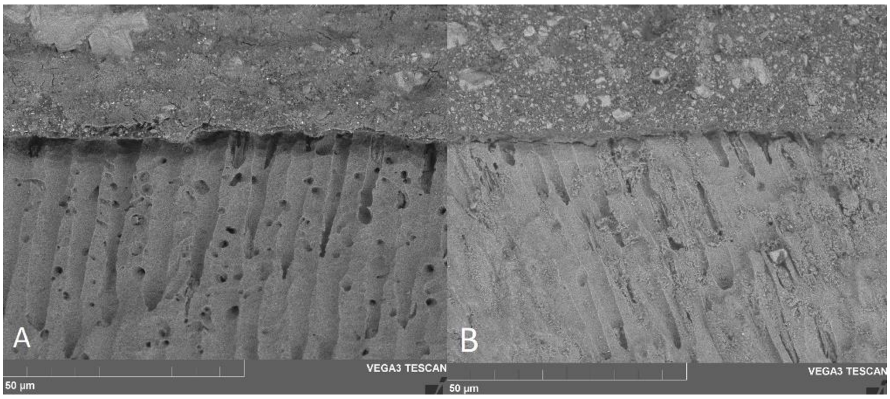

Figure 58: Shows the cement/dentin interface under SEM (no-treatment groups) A: Maxcem, B: Relyx

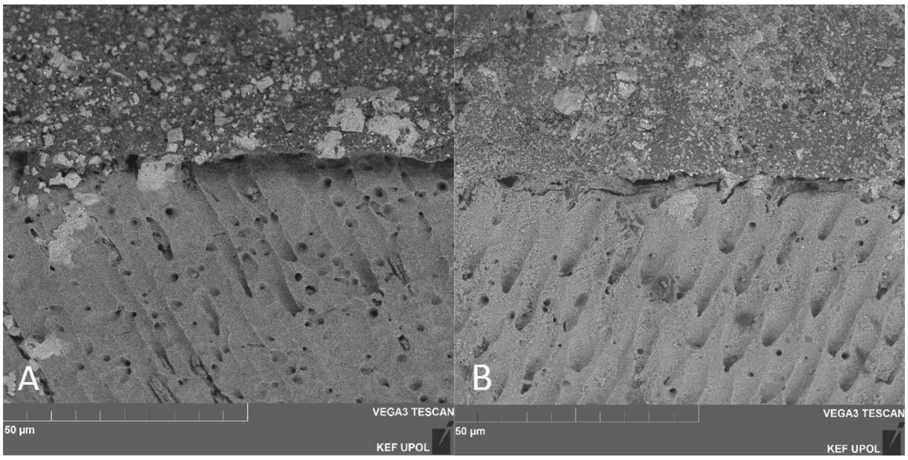

Figure 59: Shows the cement/dentin interface under SEM (PAA pre-treatment groups) A: Maxcem, B: Relyx

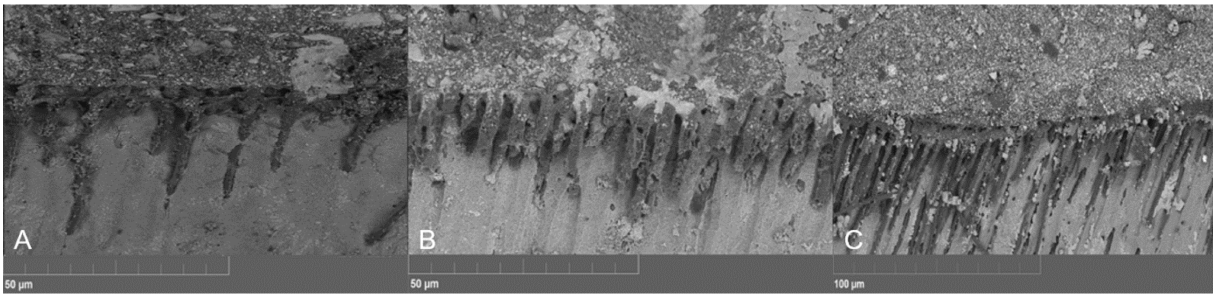

Figure 60: Shows the cement/dentin interface under SEM (H3PO4 pre-treatment groups) A: Maxcem, B: Relyx, C: NX3

Concerning the type of failure that was obtained after the TBS test, there were no cohesive or mixed failures among the no-pre-treatment groups. An adhesive failure was also dominant with the MAX-PAA group. Mixed and adhesive failures were observed in the MAX-HPO, RLX-HPO, RLX-PAA, and NX3 groups. The percentages of failure types are described in Table 3.

	Manufacturer	Composition	Application
Material	Lot number		
Relyx U200	(3M ESPE, Neuss, Germany) 4957491	Base paste: glass powder treated with silane, 2-propenoic acid, 2-methyl 1,10-(1-[hydroxymetil]-1,2-ethanodlyl) ester dimethacrylate, triethylene glycol dimethacrylate (TEGDMA), silica treated silane, glass fiber, sodium persulfate and per-3,5,5-trimethyl hexanoate t-butyl; catalyst paste: glass powder treated with silane, substitute dimethacrylate, silica-treated silane, sodium p-toluenesulfonate, 1-benzyl-5-phenyl-acid barium, calcium, 1,12-dodecane dimethacrylate, calcium hydroxide, and titanium dioxide	Apply the cement after mixing on the ceramic surface; seat the restoration gently onto the preparation allowing the cement to flow from all sides, then press. All samples were put under a static load, waiting for 60 s; during this time we clean the excess cement, then light cure for 20 s from each side (60 s in total)
Maxcem Elite Chroma	(Kerr, Scafati, Italy) 7205841	HEMA, GDM, UDMA, 1,1,3,3-tetramethylbutyl hydroperoxide TEGDMA, fluoroaluminosilicate glass, GPDM, barium glass filler, fumed silica (69 wt %)	Apply the cement after mixing on the ceramic surface; seat the restoration gently onto the preparation allowing the cement to flow from all sides, then press. All samples were put under a static load, waiting for 60 s; during this time we clean the excess cement, then light cure for 20 s from each side (60 s in total)
Nexus NX3 dual-cure	(Kerr, Scafati, Italy) 7233567	Uncured methacrylate ester monomers, HEMA, PTU, CHPO, free tertiary amines and benzoyl peroxide, inert mineral fillers, titanium dioxide, radiopaque agent, and pigments	Apply the cement after mixing on the ceramic surface; seat the restoration gently onto the preparation allowing the cement to flow from all sides, then press. All samples were put under a static load, waiting for 60 s; during this time we clean the excess cement, then light cure for 20 s from each side (60 s in total)

Table 1: Shows the composition, batch number, and manufacturer of materials used in this study

Group	**N**	**Mean (MPa)+ SD**	**Median(MPa)**
NX3	13	9.66±4.53^{a,b}	10.88
RLX-no	13	5.40±2.83^{a,c}	5.62
RLX-PAA	13	5.73±3.01^{e}	6.47
RLX-HPO	13	7.87±4.48^{f}	7.20
MAX-no	13	5.36±3.25^{d}	6.28
MAX-PAA	13	1.94±1.35$^{b\text{-}g}$	1.98
MAX-HPO	13	7.58±4.81^{g}	8.45

Table 2: Shows the Mean, Median TBS with (SD) for each luting agent after different dentin surface treatments. Upper letters indicate significant differences between the different groups ($p < 0.05$).

	Relyx U200	**Maxcem Elite Chroma**	**Nexus NX3 dual-cure**
No pre-treatment	100 % A	100 % A	53.84% A, 46.15% M
H3PO4	69.24 % A, 30.76% M	61.53% A, 38.46%M	
PAA	84.61% A, 15.38% M	100 % A	

Table 3: Shows the percentage of failure mode: A- adhesive, M- mixed, C- cohesive

6 DISCUSSION

Bonding to dentin is demanding, because dentin is a compound material with about 50 vol% of inorganic minerals, 30 vol% of collagen and 20 vol% of water (148). Bonding strategy to dentin concentrates on both inorganic and organic phase (mainly collagen) under the humid environment. Moreover, most of the dental substrates after preparation are covered with smear layer, which behaves as a barrier against the penetration of cement molecules into dental tissues (149)(150).

The existence of the smear layer has been recognized as the weak link in bonding of GIC's to dentin (151), and this might be the same with self-adhesive cements also (152). The smear layer contains dentin-buffering components that may participate into the neutralization effect during setting of the self-adhesive resin cements (153). Clinically, modification or removal of the smear layer is therefore necessary to form the hybrid layer to ensure a potent bond between the resin and dentin (154)(155).

In the present study, when Maxcem was applied according to the manufacturer's instructions (no pre-treatment), its bond strength was comparable to EAR system ($P>0.05$), while with Relyx had a lower statistically value ($P=0.0488$).

The self-adhesive resin cements, in general, have a limited ability to demineralize the hard dental tissues (156)(157). However, the different chemical compositions of the self-adhesive cements could influence their mechanical properties and bonding performances (158). This might explain the differences in behavior between the no-treatment groups (RLX and MAX) when compared to EAR. Similarly, Monticelli *et al.* (157) showed that RLX had lower bond strength to dentin than EAR systems.

The application of polyacrylic acid as pretreatment with Relyx didn't affect the bond strength values when compared to RLX-no. These results contradict the findings of Pavan et al (159), who verified a notable enhancement in the bond strength of this self-adhesive cement when 25% polyacrylic acid pretreatment was performed for 10s.

On the other hand, Maxcem with PAA treatment had the lowest bond strength value. We agree with the results obtained in the study of Mazzitelli et al (160). The authors found that the bond strength of the 2-hydroxyethyl methacrylate (HEMA)-based cement as Maxcem in our study, significantly decreased after dentin treatment with PAA. Therefore, PAA pretreatment didn't improve the bond strength but in contrast, this treatment good decreases the bond strength value.

Additionally, after the application of 25% polyacrylic acid (pH 1.53), the smear layer was partially removed, but all the tubules in the dentin stayed unplugged (161). This type of demineralization could damage the interaction between the resin and the collagen in dentin (162). It is also suggested that monomers such as

UDMA in Maxcem have hydrophobicity and thus might infiltrate less into dentin, even though UDMA has low molecular weight, which contributes to the lower viscosity of the cement (163).

It is also hypothesized, that high concentration of carboxylic groups in PAA acid conditioner were implicated in a reaction with residual calcium, which may cause the formation a PAA-based polymeric gel layer within the bonding interface, which might deteriorate the bond strength (164).

Furthermore, the HEMA monomer in Maxcem, which is one of the highest hydrophilicities among dental resins (165). Without unplugging the tubules in dentin, HEMA might not have the potential to infiltrate into dentin tubules.

The differences in behavior between RLX and MAX after the application of PAA might also be because of the different chemical compositions (158).

According to the dentin pre-treatment with H3PO4, the bond strength values of both self-adhesive cements (RLX-HPO and MAX-HPO) showed no differences from the EAR group, while there was a statistical difference between RLX-no and EAR group. Our findings do not confirm the results of De Munck et al (166) and Hikita et al (167), who found that the former conditioning of dentin deteriorates the bond strength of the self-adhesive resin cement (RelyX Unicem; 3M ESPE) because of its high viscosity, which hinders penetration on the collagen matrix and leads to the formation of a weak link between the cement and the dentin. On the contrary, Barcellos et al (168) claimed that dentin pre-treatment with phosphoric acid and adhesive increased the bond strength of (RelyX Unicem).

Similar results also were reported by Duarte et al (169), and other investigators (167) observed bond strength in self-adhesive cements similar to that of conventional resin cements after the use of H3PO4.

This enhancement of bond strength with Relyx after etching the dentin with H3PO4, may be due to increasing the hydration state of dentin, because phosphoric acid esters may require water to become ionized and interact with dentin (170)(171). This suggests that the increased water in the environment after opening the dentin tubules by H3PO4 may have optimized the acid/base reaction and improved bond strength.

According to Maxcem, it has in its composition HEMA and GDM, which have one of the highest hydrophilicities among dental resins (165). The authors speculate that after the etching with H3PO4; and the tubules in the dentin have been unplugged; such monomers might have the potential to infiltrate into the dentin tubules.

The findings with SEM observations on the surface of untreated dentin emphasize that the bonding mechanism of these simplified luting agents to dentin needs further improvements (such as chemical bonding and improved infiltration into dentin surface). No notable demineralization and real hybridization could be observed, when compared to H3PO4-treated groups. The existence of resin tags in dentinal tubules after using of H3PO4 means that the tubule orifices were evident enough for resin to penetrate into the tubule and to hybridize with the nearby collagen fibrils, allowing better sealing (172). This analysis indicates improved micromechanical retention of dentin substrates. However, it is still uncertain whether the mechanical properties of the resin cements are the outcome of a chemical interaction and/or of micro-mechanical inter-locking type with the dental substrates (173). The findings obtained here and elsewhere can be explained by the increased micro-irregularities produced by H3PO4 and the pressure used (166). However, according to Goracci et al (156), the pressure participates in the thickness and porosity reduction of the cement, and develops adaptation to the cavity walls (166).

In the present study, PAA pre-treatment was not able to open the tubules, and thus, no resin tags were formed except a few shorter tags with RLX-PAA (Figure 59).

Concerning the type of failure that was obtained after the TBS test, with no-treatment groups and MAX-PAA there were no cohesive nor mixed failures among the no-pretreatment groups. An adhesive failure was dominant. Mixed and adhesive failures were observed in MAX-HPO, RLX-HPO, RLX-PAA and NX3 groups. The presence of mixed failures obtained for RLX-HPO and MAX-HPO corroborates with resin tags formation, suggesting a greater interaction of the resin cement with dentin but did not increase the interfacial adhesion more than the cohesive strength of dentin or the ceramic, nor within the cement itself.

The high variability of the values of bond strength reported in the published articles may reflect the lack of a standard testing protocol and to the heterogeneity in tooth structure and composition. The relative low TBS values obtained in this study, might be because macro-bond strength test was used, which its values is about half as low as the weighted mean of the micro-bond strength test (66).

Other possible reasons for such high differences between studies might be due to different sample sizes, variability in cementation procedure or storage time variations (i.e. 6 months vs. 24 hours) and type of media used for samples storage. These disparities also could be due to the samples diameter of 5mm that have been used in this study. It was covering the central and peripheral dentine which have different design, while in other studies with micro test, they focused on a specific area from dentin surface.

Indeed, micro-tensile bond-strength testing (µTBS), which was developed in 1994 by Sano et al. with samples about 1mm^2 or less has some advantages as the better economic use of teeth (with multiple microspecimens originating from one tooth), the better stress distribution at the true interface (avoiding cohesive failure in tooth substrate or composite), the better control of regional differences (e.g. peripheral versus central dentin), etc., but in our study we chose the Macro TBS because we wanted to test the specimens in more clinically relevant circumstances, since practically there are no dental restorations within 1mm^2 area. Beside that the micro-tensile bond-strength test is more laborious and technique-sensitive (66).

Another issue includes the big amount of pre-testing failures often recorded with micro-tensile bond strength testing can’t be neglected (66).

Regarding the limitations of this in-vitro study, we can mention that usually when the bond strength test is employed, the bonding performance is tested in a flat dentin surface, but this doesn’t mimic the oral environmental conditions. Other factors that might affect the resin-dentin bonds such as occlusal loading, pH changes, and enzymatic challenges were not taken into consideration in this study. The literature has not provided the minimum bond strength that the luting agents must have to the dental tissues in order to guarantee the longevity and success of the cemented restorations (174). Therefore, long-term clinical trials and further investigations are advisable to improve adhesive dentistry, not only by developing less time-consuming cements, but also by enhancing the quality and longevity of the bonds created with such cements.

7 CONCLUSION

Within the limitations of this study, we conclude that,

- Maxcem Elite Chroma as a self-adhesive resin cement might be an effective alternative to conventional resin cement (EAR system). Etching the dentin with phosphoric acid doesn't negatively affect the bond strength of MAX to dentin. On the other hand, phosphoric acid improved the bond strength of Relyx U200 when compared to EAR cement.
- Polyacrylic acid didn't affect the bond strength of Relyx U200 to dentin, whereas it decreased significantly the bond strength of Maxcem Elite. Therefore, we can't conclude that all self-adhesive cement could be classified as one homogeneous group.

- Also in this study, it can be concluded that hydrophobic/hydrophilic properties of the cements and their functional monomers significantly affected the bond strength after smear layer treatment.

- Clinicians should understand the properties of each cement before using it as a luting agent.

8 REFERENCES

1. Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. J Dent Res. 1955;
2. Jin X zhuang, Homaei E, Matinlinna JP, Tsoi JKH. A new concept and finite-element study on dental bond strength tests. Dent Mater. 2016;
3. Marshall SJ, Bayne SC, Baier R, Tomsia AP, Marshall GW. A review of adhesion science. Dent Mater. 2010;
4. Jones RM, Moore BK, Goodacre CJ, Munoz-Viveros CA. Microleakage and shear bond strength of resin and porcelain veneers bonded to cast alloys. J Prosthet Dent. 1991;
5. Fujishima A, Fujishima Y, Ferracane JL. Shear bond strength of four commercial bonding systems to cpTi. Dent Mater. 1995;
6. Ishijima T, Caputo AA, Mito R. Adhesion of resin to casting alloys. J Prosthet Dent. 1992;
7. Ladha K, Verma M. Conventional and contemporary luting cements: An overview. Journal of Indian Prosthodontist Society. 2010.
8. Anusavice KJ. Phillips' Science of Dental Materials (Anusavice Phillip's Science of Dental Materials). Elsevier Saunders. 2013.
9. Tiu J, Al-Amleh B, Waddell JN, Duncan WJ. Clinical tooth preparations and associated measuring methods: A systematic review. Journal of Prosthetic Dentistry. 2015.
10. Diaz-Arnold AM, Vargas MA, Haselton DR. Current status of luting agents for fixed prosthodontics. J Prosthet Dent. 1999;
11. Christensen GJ. Cementing porcelain-fused-to-metal crowns. J Am Dent Assoc. 1997;
12. Pameijer CH. A review of luting agents. International Journal of Dentistry. 2012.
13. Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, et al. Adhesion to enamel and dentin: Current status and future challenges. In: Operative Dentistry. 2003.
14. Betancourt DE, Baldion PA, Castellanos JE. Resin-dentin bonding interface: Mechanisms of degradation and strategies for stabilization of the hybrid layer. International Journal of Biomaterials. 2019.
15. Tjäderhane L, Carrilho MR, Breschi L, Tay FR, Pashley DH. Dentin basic structure and composition-an overview. Endod Top. 2009;
16. Carvalho RM, Tjäderhane L, Manso AP, Carrilho MR, Carvalho CAR. Dentin as a bonding substrate. Endod Top. 2009;
17. Perdigão J, Sezinando A, Monteiro PC. Effect of substrate age and adhesive composition on dentin bonding. Oper Dent. 2013;
18. Dos Santos PH, Karol S, Bedran-Russo AK. Long-term nano-mechanical properties of

biomodified dentin-resin interface components. J Biomech. 2011;

19. Adamiak K, Sionkowska A. Current methods of collagen cross-linking: Review. International Journal of Biological Macromolecules. 2020.

20. Miura J, Nishikawa K, Kubo M, Fukushima S, Hashimoto M, Takeshige F, et al. Accumulation of advanced glycation end-products in human dentine. Arch Oral Biol. 2014;

21. Miguez PA, Pereira PNR, Atsawasuwan P, Yamauchi M. Collagen cross-linking and ultimate tensile strength in dentin. J Dent Res. 2004;

22. Shinno Y, Ishimoto T, Saito M, Uemura R, Arino M, Marumo K, et al. Comprehensive analyses of how tubule occlusion and advanced glycation end-products diminish strength of aged dentin. Sci Rep. 2016;

23. Veis A, Schlueter RJ. The Macromolecular Organization of Dentine Matrix Collagen. I. Characterization of Dentine Collagen. Biochemistry. 1964;

24. Ritter A V., Walter R, Boushell LW. Sturdevant's art and science of operative dentistry. Sturdevant's Art and Science of Operative Dentistry. 2018.

25. Perdigão J. Current perspectives on dental adhesion: (1) Dentin adhesion – not there yet. Japanese Dental Science Review. 2020.

26. Garberoglio R, Brännström M. Scanning electron microscopic investigation of human dentinal tubules. Arch Oral Biol. 1976;

27. Kontakiotis EG, Tsatsoulis IN, Filippatos CG, Agrafioti A. A quantitative and diametral analysis of human dentinal tubules at pulp chamber ceiling and floor under scanning electron microscopy. Aust Endod J. 2015;

28. Swift EJ, Perdigão J, Heymann HO. Bonding to enamel and dentin: a brief history and state of the art, 1995. Quintessence Int. 1995;

29. Chang B, Svoboda KKH, Liu X. Cell polarization: From epithelial cells to odontoblasts. European Journal of Cell Biology. 2019.

30. Yoshihara K, Yoshida Y, Hayakawa S, Nagaoka N, Irie M, Ogawa T, et al. Nanolayering of phosphoric acid ester monomer on enamel and dentin. Acta Biomater. 2011;

31. Perdigão J. Dentin bonding-Variables related to the clinical situation and the substrate treatment. Dent Mater. 2010;

32. Marshall GW, Marshall SJ, Kinney JH, Balooch M. The dentin substrate: Structure and properties related to bonding. J Dent. 1997;

33. Burrow MF, Takakura H, Nakajima M, Inai N, Tagami J, Takatsu T. The influence of age and depth of dentin on bonding. Dent Mater. 1994;

34. Pashley DH. Smear layer: overview of structure and function. Proceedings of the Finnish Dental Society. Suomen Hammaslääkäriseuran toimituksia. 1992.

35. Bhagwat S, Heredia A, Mandke L. The Smear Layer Revisited. Indian J Med Res Pharm Sci. 2016;

36. Gwinnett AJ. Smear layer: morphological considerations. Oper Dent Suppl. 1984;

37. Germain HA. Advances in Operative Dentistry, Volume 2: Challenges of the Future. J Prosthodont. 2004;

38. Pashley DH, Tao L, Boyd L, King GE, Horner JA. Scanning electron microscopy of the substructure of smear layers in human dentine. Arch Oral Biol. 1988;

39. Van Landuyt K, De Munck J, Coutinho E, Peumans M, Lambrechts P, Van Meerbeek B. Bonding to dentin: Smear layer and the process of hybridization. In: Dental Hard Tissues and Bonding: Interfacial Phenomena and Related Properties. 2005.

40. Sugizaki J, Morigami M, Uno S, Yamada T. Clinical evaluation and interfacial morphology observation of Xeno III self-etching resin bonding and restorative system. Dent Mater J. 2007;

41. Pashley DH. The evolution of dentin bonding from no-etch to total-etch to self-etch. Adhes Techol Sol Jan 2003 1. 2002;

42. Van Landuyt KL, Yoshida Y, Hirata I, Snauwaert J, De Munck J, Okazaki M, et al. Influence of the chemical structure of functional monomers on their adhesive performance. J Dent Res. 2008;

43. Pashley D, Tay F. Mechanical stability of resin–dentine bonds. In: Dental biomaterials. 2008.

44. Carvalho AA, Moreira FCL, Cunha LM, de Moura SM, Souza JB de, Estrela C, et al. Marginal microleakage of class II composite resin restorations due to restorative techniques. Rev Odonto Ciência. 2010;

45. Pratap B, Gupta RK, Bhardwaj B, Nag M. Resin based restorative dental materials: characteristics and future perspectives. Japanese Dental Science Review. 2019.

46. Parameswari BD, Rajakumar M, Lambodaran G, Shyam S. Comparative study on the tensile bond strength and marginal fit of complete veneer cast metal crowns using various luting agents: An in vitro study. J Pharm Bioallied Sci. 2016;

47. Kowalska A, Sokolowski J, Bociong K. The photoinitiators used in resin based dental composite—a review and future perspectives. Polymers. 2021.

48. Zakir M, Ashraf U, Tian T, Han A, Qiao W, Jin X, et al. The Role of Silane Coupling Agents and Universal Primers in Durable Adhesion to Dental Restorative Materials - a Review. Current Oral Health Reports. 2016.

49. De Souza GM, Braga RR, Cesar PF, Lopes GC. Correlation between clinical performance and degree of conversion of resin cements: A literature review. J Appl Oral Sci. 2015;

50. Burgess JO, Ghuman T, Cakir D. Self-adhesive resin cements. J Esthet Restor Dent. 2010;

51. Simon JF, Darnell LA. Considerations for proper selection of dental cements. Compendium of continuing education in dentistry (Jamesburg, N.J. : 1995). 2012.

52. Perdigão J, Walter R, Miguez PA, Swift EJ. Fundamental concepts of enamel and dentin adhesion. In: Sturdevant's Art and Science of Operative Dentistry. 2018.

53. De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Braem M, et al. A critical review of the durability of adhesion to tooth tissue: Methods and results. Journal of Dental Research. 2005.

54. Li F, Zhang L, Weir MD, Cheng L, Zhang K, Xu HHK. Understanding the chemistry and improving the durability of dental resin-dentin bonded interface. In: Material-Tissue Interfacial Phenomena: Contributions from Dental and Craniofacial Reconstructions. 2017.

55. Carvalho RM, Mendonça JS, Santiago SL, Silveira RR, Garcia FCP, Tay FR, et al. Effects of HEMA/solvent combinations on bond strength to dentin. J Dent Res. 2003;

56. Tay FR, Gwinnett JA, Wei SHY. Relation between water content in acetone/alcohol-based primer and interfacial ultrastructure. J Dent. 1998;

57. Curtis R V., Watson TF. Dental biomaterials: Imaging, testing and modelling. Dental Biomaterials: Imaging, Testing and Modelling. 2008.

58. Perdigão J. New Developments in Dental Adhesion. Dental Clinics of North America. 2007.

59. Ferracane JL, Stansbury JW, Burke FJT. Self-adhesive resin cements - chemistry, properties and clinical considerations. Journal of Oral Rehabilitation. 2011.

60. Sezinando A. Looking for the ideal adhesive - A review. Revista Portuguesa de Estomatologia, Medicina Dentaria e Cirurgia Maxilofacial. 2014.

61. Tay FR, Pashley DH, Suh BI, Carvalho RM, Itthagarun A. Single-step adhesives are permeable membranes. J Dent. 2002;

62. Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, et al. Adhesion to enamel and dentin. Current state and future tasks - Part 1. Asthetische Zahnmedizin. 2004;

63. Giannini M, Makishi P, Ayres APA, Vermelho PM, Fronza BM, Nikaido T, et al. Self-Etch adhesive systems: A literature review. Brazilian Dental Journal. 2015.

64. Hashimoto M. A review - Micromorphological evidence of degradation in resin-dentin bonds and potential preventional solutions. Journal of Biomedical Materials Research - Part B Applied Biomaterials. 2010.

65. Lazarescu F. Comprehensive Esthetic Dentistry. Stomatol EDU J. 2017;

66. Van Meerbeek B, Peumans M, Poitevin A, Mine A, Van Ende A, Neves A, et al. Relationship between bond-strength tests and clinical outcomes. Dental Materials. 2010.

67. De Munck J, Van Meerbeek B, Satoshi I, Vargas M, Yoshida Y, Armstrong S, et al. Microtensile bond strengths of one- and two-step self-etch adhesives to bur-cut enamel and dentin. Am J Dent. 2003;

68. Migliau G. Classification review of dental adhesive systems: from the IV generation to the universal type. Ann Stomatol (Roma). 2017;

69. Tay FR, Pashley DH. Water treeing - A potential mechanism for degradation of dentin adhesives. Am J Dent. 2003;

70. Hardan L, Bourgi R, Kharouf N, Mancino D, Zarow M, Jakubowicz N, et al. Bond strength of universal adhesives to dentin: A systematic review and meta-analysis. Polymers. 2021.

71. Yoshida Y, Nagakane K, Fukuda R, Nakayama Y, Okazaki M, Shintani H, et al. Comparative study on adhesive performance of functional monomers. J Dent Res. 2004;

72. Mehta D, Shetty R. Bonding to Zirconia : elucidating the confusion. Int Dent SA. 2010;

73. van Meerbeek B, Yoshihara K, van Landuyt K, Yoshida Y, Peumans M. From buonocore's pioneering acid-etch technique to self-adhering restoratlves. A status perspective of rapidly advancing dentai adheslve technology. J Adhes Dent. 2020;

74. Inoue S, Koshiro K, Yoshida Y, De Munck J, Nagakane K, Suzuki K, et al. Hydrolytic stability of self-etch adhesives bonded to dentin. J Dent Res. 2005;

75. Han F, Dai S, Yang J, Shen J, Liao M, Xie H, et al. Glycerol Phosphate Dimethacrylate: An Alternative Functional Phosphate Ester Monomer to 10-Methacryloyloxydecyl Dihydrogen Phosphate for Enamel Bonding. ACS Omega. 2020;

76. Carrilho E, Cardoso M, Ferreira MM, Marto CM, Paula A, Coelho AS. 10-MDP based dental adhesives: Adhesive interface characterization and adhesive stability-A systematic review. Materials. 2019.

77. Coutinho E, Van Landuyt K, De Munck J, Poitevin A, Yoshida Y, Inoue S, et al. Development of a self-etch adhesive for resin-modified glass lonomers. J Dent Res. 2006;

78. Tay FR, Pashley DH. Have dentin adhesives become too hydrophilic? Journal (Canadian Dental Association). 2003.

79. Van Landuyt KL, Mine A, De Munck J, Jaecques S, Peumans M, Lambrechts P, et al. Are one-step adhesives easier to use and better performing? Multifactorial assessment of contemporary one-step self-etching adhesives. J Adhes Dent. 2009;

80. Carvalho Silva JM, Alves Feitosa R, Lima Araújo Ferreira D, Oliveira Santos Viana M. Use of Self-Adhesive Resin Cements in Dentistry: a Literature Review. J Heal Sci. 2021;

81. Kanehira M, Finger WJ, Hoffmann M, Komatsu M. Compatibility between an all-in-one self-etching adhesive and a dual-cured resin luting cement. J Adhes Dent. 2006;

82. Pegoraro TA, da Silva NRFA, Carvalho RM. Cements for Use in Esthetic Dentistry. Dental Clinics of North America. 2007.

83. Weiser F, Behr M. Self-Adhesive Resin Cements: A Clinical Review. J Prosthodont. 2015;

84. Han L, Okamoto A, Fukushima M, Okiji T. Evaluation of physical properties and surface degradation of self-adhesive resin cements. Dent Mater J. 2007;

85. Stamatacos C, Simon JF. Cementation of indirect restorations: an overview of resin cements. Compendium of continuing education in dentistry (Jamesburg, N.J. : 1995). 2013.

86. Segreto DR, Naufel FS, Brandt WC, Guiraldo RD, Correr-Sobrinho L, Sinhoreti MAC. Influence of photoinitiator and light-curing source on bond strength of experimental resin cements to dentin. Braz Dent J. 2016;

87. Vargas MA, Bergeron C, Diaz-Arnold A. Cementing all-ceramic restorations: Recommendations for success. J Am Dent Assoc. 2011;

88. Kilinc E, Antonson SA, Hardigan PC, Kesercioglu A. The effect of ceramic restoration shade and thickness on the polymerization of light- and dual-cure resin cements. Oper Dent. 2011;

89. Arrais CAG, Rueggeberg FA, Waller JL, de Goes MF, Giannini M. Effect of curing mode on the polymerization characteristics of dual-cured resin cement systems. J Dent. 2008;

90. Rueggeberg FA, Giannini M, Arrais CAG, Price RBT. Light curing in dentistry and clinical

implications: A literature review. Brazilian Oral Research. 2017.

91. Donovan T, Simonsen RJ, Guertin G, Tucker R V. Retrospective clinical evaluation of 1,314 cast gold restorations in service from 1 to 52 years. J Esthet Restor Dent. 2004;

92. Givan DA. Precious metal alloys for dental applications. In: Precious Metals for Biomedical Applications. 2014.

93. Spear F, Holloway J. Which all-ceramic system is optimal for anterior esthetics? Journal of the American Dental Association. 2008.

94. Zhang Y, Kelly JR. Dental Ceramics for Restoration and Metal Veneering. Dental Clinics of North America. 2017.

95. Pollington S, van Noort R. An update of ceramics in dentistry. In: Perspectives on Clinical Dentistry. 2011.

96. Kelly JR, Benetti P. Ceramic materials in dentistry: Historical evolution and current practice. Aust Dent J. 2011;

97. Willard A, Gabriel Chu TM. The science and application of IPS e.Max dental ceramic. Kaohsiung Journal of Medical Sciences. 2018.

98. Schneider O. Long-term Cerec 3D chairside bridges milled from IPS e.max CAD LT blocks. Int J Comput Dent. 2016;

99. Pathrabe A, Lahoti K, Gade JR. Metal Free Ceramics in Dentistry: A Review. Int J Oral Heal Med Res. 2016;

100. Chen YM, Smales RJ, Yip KHK, Sung WJ. Translucency and biaxial flexural strength of four ceramic core materials. Dent Mater. 2008;

101. Kelly JR. Dental ceramics: What is this stuff anyway? Journal of the American Dental Association. 2008.

102. Bunek SS, Swift EJ. Contemporary ceramics and cements. J Esthet Restor Dent. 2014;

103. Byeon S-M, Song J-J. Mechanical Properties and Microstructure of the Leucite-Reinforced Glass-Ceramics for Dental CAD/CAM. J Dent Hyg Sci. 2018;

104. Bajraktarova-Valjakova E, Korunoska-Stevkovska V, Kapusevska B, Gigovski N, Bajraktarova-Misevska C, Grozdanov A. Contemporary dental ceramic materials, a review: Chemical composition, physical and mechanical properties, indications for use. Open Access Macedonian Journal of Medical Sciences. 2018.

105. McLaren EA, LeSage B. Feldspathic veneers: what are their indications? Compend Contin Educ Dent. 2011;

106. Mclaren E a, Cao PT. Ceramics in Dentistry—Part I: Classes of Materials. Insid Dent. 2009;

107. Barizon KTL, Bergeron C, Vargas MA, Qian F, Cobb DS, Gratton DG, et al. Ceramic materials for porcelain veneers: Part II. Effect of material, shade, and thickness on translucency. J Prosthet Dent. 2014;

108. Castelnuovo J, Tjan AHL, Phillips K, Nicholls JI, Kois JC. Fracture load and mode of failure of ceramic veneers with different preparations. J Prosthet Dent. 2000;

109. Warreth A, Elkareimi Y. All-ceramic restorations: A review of the literature. Saudi Dental Journal. 2020.

110. Santos MJMC, Costa MD, Rubo JH, Pegoraro LF, Santos GC. Current all-ceramic systems in dentistry: a review. Compendium of continuing education in dentistry (Jamesburg, N.J. : 1995). 2015.

111. Kang S-H, Chang J, Son H-H. Flexural strength and microstructure of two lithium disilicate glass ceramics for CAD/CAM restoration in the dental clinic. Restor Dent Endod. 2013;

112. El-Meliegy E, Noort R Van. Glasses and glass ceramics for medical applications. Glasses and Glass Ceramics for Medical Applications. 2012.

113. Wang F, Chai Z, Deng Z, Gao J, Wang H, Chen J. Effect of heat-pressing temperature and holding time on the microstructure and flexural strength of lithium disilicate glass-ceramics. PLoS One. 2015;

114. Tysowsky GW. The science behind lithium disilicate: A metal-free alternative. Dent Today. 2009;

115. Fasbinder DJ, Dennison JB, Heys D, Neiva G. A Clinical Evaluation of Chairside Lithium Disilicate CAD/CAM Crowns. J Am Dent Assoc. 2010;

116. Aschheim KW. Esthetic dentistry: A clinical approach to techniques and materials, third edition. Esthetic Dentistry: A Clinical Approach to Techniques and Materials, Third Edition. 2014.

117. Baroudi K, Ibraheem SN. Assessment of Chair-side Computer-Aided Design and Computer-Aided Manufacturing Restorations: A Review of the Literature. J Int oral Heal JIOH. 2015;

118. Albero A, Pascual A, Camps I, Grau-Benitez M. Comparative characterization of a novel cad-cam polymer-infiltrated-ceramic-network. J Clin Exp Dent. 2015;

119. De Aza PN, De Aza AH, De Aza S. Crystalline Bioceramic Materials. ChemInform. 2006;

120. Abd El-Ghany OS, Sherief AH. Zirconia based ceramics, some clinical and biological aspects: Review. Futur Dent J. 2016;

121. Partiyan A, Osman E, Rayyan MM, Aboushelib M, Ibrahim A, Jimbo R. Fracture resistance of three-unit zirconia fixed partial denture with modified framework. Odontology. 2017;

122. Beuer F, Stimmelmayr M, Gueth JF, Edelhoff D, Naumann M. In vitro performance of full-contour zirconia single crowns. Dent Mater. 2012;

123. Stober T, Bermejo JL, Rammelsberg P, Schmitter M. Enamel wear caused by monolithic zirconia crowns after 6 months of clinical use. J Oral Rehabil. 2014;

124. Dullabh H, Herbst D, Sykes L, Biseswar N. Biaxial flexural strength of three ceramic oxide core materials. South African Dent J. 2017;

125. Guazzato M, Albakry M, Ringer SP, Swain M V. Strength, fracture toughness and microstructure of a selection of all-ceramic materials. Part II. Zirconia-based dental ceramics. Dent Mater. 2004;

126. Andersson M, Odén A. A new all-ceramic crown: A dense-sintered, high-purity alumina coping with porcelain. Acta Odontol Scand. 1993;

127. Triwatana P, Nagaviroj N, Tulapornchai C. Clinical performance and failures of zirconia-based fixed partial dentures: A review literature. J Adv Prosthodont. 2012;

128. Grech J, Antunes E. Zirconia in dental prosthetics: A literature review. Journal of Materials Research and Technology. 2019.

129. Brog JP, Chanez CL, Crochet A, Fromm KM. Polymorphism, what it is and how to identify it: A systematic review. RSC Advances. 2013.

130. Tomaselli L de O, de Oliveira DCRS, Favarão J, da Silva AF, Pires-De-Souza F de CP, Geraldeli S, et al. Influence of pre-heating regular resin composites and flowable composites on luting ceramic veneers with different thicknesses. Braz Dent J. 2019;

131. Baroudi K, Rodrigues JC. Flowable resin composites: A systematic review and clinical considerations. Journal of Clinical and Diagnostic Research. 2015.

132. Helvey GA. Creating super dentin: using flowable composites as luting agents to help prevent secondary caries. Compend Contin Educ Dent. 2013;

133. Lassila L, Säilynoja E, Prinssi R, Vallittu P, Garoushi S. Characterization of a new fiber-reinforced flowable composite. Odontology. 2019;

134. Dionysopoulos D, Tolidis K, Gerasimou P, Koliniotou-Koumpia E. Effect of preheating on the film thickness of contemporary composite restorative materials. J Dent Sci. 2014;

135. Choudhary N, Kamat S, Mangala TM, Thomas M. Effect of pre-heating composite resin on gap formation at three different temperatures. J Conserv Dent. 2011;

136. Almeida JR, Schmitt GU, Kaizer MR, Boscato N, Moraes RR. Resin-based luting agents and color stability of bonded ceramic veneers. J Prosthet Dent. 2015;

137. Demirbuga S, Ucar FI, Cayabatmaz M, Zorba YO, Cantekin K, Topçuoğlu HS, et al. Microshear bond strength of preheated silorane- and methacrylate-based composite resins to dentin. Scanning. 2016;

138. Rickman LJ, Padipatvuthikul P, Chee B. Clinical applications of preheated hybrid resin composite. Br Dent J. 2011;

139. Baroudi K, Mahmoud S. Improving Composite Resin Performance Through Decreasing its Viscosity by Different Methods. Open Dent J. 2015;

140. Abed Kahnamouei M, Gholizadeh S, Rikhtegaran S, Daneshpooy M, Kimyai S, Alizadeh Oskoee P, et al. Effect of preheat repetition on color stability of methacrylate- and silorane-based composite resins. J Dent Res Dent Clin Dent Prospects. 2017;

141. Mounajjed R, Salinas TJ, Ingr T, Azar B. Effect of different resin luting cements on the marginal fit of lithium disilicate pressed crowns. J Prosthet Dent. 2018;

142. Self-etching adhesive systems, bond strength and nanofiltration: a review. Rev Fac Odontol Univ Antioquia. 2012;

143. Kharouf N, Mancino D, Naji-Amrani A, Eid A, Haikel Y, Hemmerle J. Effectiveness of etching by three acids on the morphological and chemical features of dentin tissue. J Contemp Dent Pract. 2019;

144. Ferro MC de L, Colucci V, Marques AG, Ribeiro RF, Silva-Sousa YTC, Gomes ÉA. Fracture

strength of weakened anterior teeth associated to different reconstructive techniques. Braz Dent J. 2016;

145. Tonial D, Ghiggi PC, Lise AA, Burnett LH, Oshima HMS, Spohr AM. Effect of conditioner on microtensile bond strength of self-adhesive resin cements to dentin. Stomatologija. 2010;

146. Lührs AK, Guhr S, Günay H, Geurtsen W. Shear bond strength of self-adhesive resins compared to resin cements with etch and rinse adhesives to enamel and dentin in vitro. Clin Oral Investig. 2010;

147. Kharouf N, Arntz Y, Eid A, Zghal J, Sauro S, Haikel Y, et al. Physicochemical and Antibacterial Properties of Novel, Premixed Calcium Silicate-Based Sealer Compared to Powder–Liquid Bioceramic Sealer. J Clin Med. 2020;

148. Nakabayashi N, Pashley D. Hybridization of Dental Hard Tissues. Chicago Quintessence. 1998;

149. Pashley DH, Michelich V, Kehl T. Dentin permeability: Effects of smear layer removal. J Prosthet Dent. 1981;

150. Saikaew P, Chowdhury AFMA, Fukuyama M, Kakuda S, Carvalho RM, Sano H. The effect of dentine surface preparation and reduced application time of adhesive on bonding strength. J Dent. 2016;

151. Tay FR, Smales RJ, Ngo H, Wei SH, Pashley DH. Effect of different conditioning protocols on adhesion of a GIC to dentin. J Adhes Dent. 2001;

152. Al-Assaf K, Chakmakchi M, Palaghias G, Karanika-Kouma A, Eliades G. Interfacial characteristics of adhesive luting resins and composites with dentine. Dent Mater. 2007;

153. Oliveira SSA, Pugach MK, Hilton JF, Watanabe LG, Marshall SJ, Marshall GW. The influence of the dentin smear layer on adhesion: A self-etching primer vs. a total-etch system. Dent Mater. 2003;

154. Tao L, Pashley DH. Shear bond strengths to dentin: effects of surface treatments, depth and position. Dent Mater. 1988;

155. Eliades G, Watts D, Eliades T. Dental hard tissues and bonding: Interfacial phenomena and related properties. Dental Hard Tissues and Bonding: Interfacial Phenomena and Related Properties. 2005.

156. Goracci C, Cury AH, Cantoro A, Papacchini F, Tay FR, Ferrari M. Microtensile bond strength and interfacial properties of self-etching and self-adhesive resin cements used to lute composite onlays under different seating forces. J Adhes Dent. 2006;

157. Monticelli F, Osorio R, Mazzitelli C, Ferrari M, Toledano M. Limited decalcification/diffusion of self-adhesive cements into dentin. J Dent Res. 2008;

158. Hattar S, Hatamleh MM, Sawair F, Al-Rabab'ah M. Bond strength of self-adhesive resin cements to tooth structure. Saudi Dent J. 2015;

159. Pavan S, Dos Santos PH, Berger S, Bedran-Russo AKB. The effect of dentin pretreatment on the microtensile bond strength of self-adhesive resin cements. J Prosthet Dent. 2010;

160. Mazzitelli C, Monticelli F, Toledano M, Ferrari M, Osorio R. Dentin treatment effects on the bonding performance of self-adhesive resin cements. Eur J Oral Sci. 2010;

161. Ayad MF. Effects of rotary instrumentation and different etchants on removal of smear layer on human dentin. J Prosthet Dent. 2001;

162. Mushashe AM, Gonzaga CC, da Cunha LF, Furuse AY, Moro A, Correr GM. Effect of enamel and dentin surface treatment on the self-adhesive resin cement bond strength. Braz Dent J. 2016;

163. Van Landuyt KL, Snauwaert J, De Munck J, Peumans M, Yoshida Y, Poitevin A, et al. Systematic review of the chemical composition of contemporary dental adhesives. Biomaterials. 2007.

164. Es-Souni M, Fischer-Brandies H, Zaporojshenko V, Es-Souni M. On the interaction of polyacrylic acid as a conditioning agent with bovine enamel. Biomaterials. 2002;

165. Sokolowski G, Szczesio A, Bociong K, Kaluzinska K, Lapinska B, Sokolowski J, et al. Dental resin cements-The influence of water sorption on contraction stress changes and hydroscopic expansion. Materials (Basel). 2018;

166. De Munck J, Vargas M, Van Landuyt K, Hikita K, Lambrechts P, Van Meerbeek B. Bonding of an auto-adhesive luting material to enamel and dentin. Dent Mater. 2004;

167. Hikita K, Van Meerbeek B, De Munck J, Ikeda T, Van Landuyt K, Maida T, et al. Bonding effectiveness of adhesive luting agents to enamel and dentin. Dent Mater. 2007;

168. Barcellos DC, Batista GR, Da Silva MA, Rangel PM, Torres CRG, Fava M. Evaluation of bond strength of self-adhesive cements to dentin with or without application of adhesive systems. J Adhes Dent. 2011;

169. Duarte S, Botta AC, Meire M, Sadan A. Microtensile bond strengths and scanning electron microscopic evaluation of self-adhesive and self-etch resin cements to intact and etched enamel. J Prosthet Dent. 2008;

170. Banomyong D, Palamara JEA, Burrow MF, Messer HH. Effect of dentin conditioning on dentin permeability and micro-shear bond strength. Eur J Oral Sci. 2007;

171. Moszner N, Salz U, Zimmermann J. Chemical aspects of self-etching enamel-dentin adhesives: A systematic review. Dent Mater. 2005;

172. Eshrak Sofan, PhD Afrah Sofan, PhD Gaspare Palaia, PhD Gianluca Tenore, MD, DDS Umberto Romeo, MD, DDS Guido Migliau, MD D. Classification review of dental adhesive systems: from the IV generation to the universal type. Ann Stomatol (Roma). 2017;

173. Gerth HUV, Dammaschke T, Züchner H, Schäfer E. Chemical analysis and bonding reaction of RelyX Unicem and Bifix composites-A comparative study. Dent Mater. 2006;

174. Stona P, Borges GA, Montes MAJR, Júnior LHB, Weber JBB, Spohr AM. Effect of polyacrylic acid on the interface and bond strength of self-adhesive resin cements to dentin. J Adhes Dent. 2013;

9 LIST OF FIGURES

10 LIST OF TABLES

11 ACKNOWLEDGMENT

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12 SOUHRN

Srovnání účinnosti přípravy dentinu na sílu vazby u dvou samoadhezivních pryskyřičných cementu ve srovnání s etch-and-rinse systémem, *In Vitro* studie

Úvod do problematiky: Je prokázané, že selektivní leptání skloviny zlepšuje pevnost spojení některých samoleptacích pryskyřičných cementů. Obdobné tvrzení dosud nebylo jednoznačně prokázáno v případě dentinu.

Cíl: Vyhodnotit pevnost v tahu dvou samo-adhesivních pryskyřičných cementů při použití různých přípravných schémat povrchu dentinu. Analýza spoje mezi dentinem a použitým cementem pod elektronovým mikroskopem.

Metodika a materiály. Bylo použito 120 extrahovaných třetích molárů. Zuby byly rozděleny do sedmi skupin (n = 16). Rozdělení bylo na základě testovaného cementu. Maxcem Elite Chroma (MAX) (Kerr, Scafati, Itálie) a RelyX U200 (RLX) (3M ESPE, Neuss, Německo). Další skupiny byly vytvořeny na základě způsobu přípravy povrchu dentinu a to: bez přípravy, příprava za pomoci kyseliny polyakrylátové nebo kyseliny fosforečné. Následovala kontrolní skupina, kde byl použit konvenční cement a standardní příprava povrchu dentinu (EAR) (NX3 Nexus (NX3) (Kerr, Scafati, Itálie). Před testováním byly všechny vzorky skladované v destilované vodě po dobu 24 hodin. Tři vzorky z každé skupiny byly podrobeny analýze za pomoci elektronového mikroskopu (SEM). U zbývajících vzorků byl proveden test pevnosti v tahu (TBS). Data byla statisticky analyzována pomocí Kruskal-Wallisova testu a srovnávací Pairwise za použití Wilcoxon rank sum testu.

Výsledky: MAX bez přípravy povrchu dentinu a při leptání kyselinou fosforečnou dosáhl statisticky obdobných hodnot vazby jako kontrolní skupina NX3 (P> 0,05). Mezi RLX bez předběžného ošetření dentinu (5,62 MPa) a NX3 (10,88 MPa) byl shledán statistický rozdíl (P = 0,00488). Při přípravě povrchu dentinu pomoci kyseliny fosforečné se zvyšuje hodnota pevnosti vazby RLX na úroveň srovnatelnou s NX3 (P> 0,05). Nejnižší pevnosti v tahu (TBS) byla shledána po aplikaci kyseliny polyakrylové s MAX (1,98 MPa). Nebyly nalezeny žádné statistické rozdíly mezi hodnotami vazební síly RLX po ošetření kyselinou polyakrylovou a RLX bez předběžného ošetření ve srovnání s NX3 (P> 0,05). Po předchozí přípravě povrchu dentinu pomoci kyseliny fosforečné, se ukázala za pomoci elektronového mikroskopu (SEM) větší míra infiltrace cementu do dentinových tubulů, a vytvoření takzvaných chapadel (resin tags).

Při selhání spoje, spíše převládalo adhesivní selhání na úrovni dentinu než kohezivní ve struktuře cementu.

Závěr: Při cementaci keramické náhrady na dentinový povrch, může být samo-adhesivní pryskyřičný cement MAX účinnou alternativou ke konvenčnímu pryskyřičnému cementu. Leptání dentinu kyselinou fosforečnou nemá negativní vliv na pevnost vazby MAX na dentin. Kyselina fosforečná zlepšila pevnost vazby RLX cementu a dentinu.

13 CURRICULUM VITAE

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January 01, 1987	Born in Syria
2004-2009	Doctor of Dental Surgery (DDS), Al-Baath University, Syria
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