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Dental adhesion (Bonding)

A Graduation Project Submitted to The College of Dentistry, University of Baghdad, Department of Restorative and Aesthetic Dentistry in Partial Fulfillment for the Bachelor of Dental Surgery

BY

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Certification of the Supervisor

I certify that this project entitled "Dental adhesion (Bonding) in Operative Dentistry" was prepared by the fifthyear student Fatima Mohammed Yass under my supervision at the College of Dentistry/University of Baghdad in partial fulfilment of the graduation requirements for the Bachelor Degree in Dentistry.

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Dedication

This study is wholeheartedly dedicated to my beloved parents, who have been my source of inspiration and gave me strength when I thought of giving up, who continually provide their moral, spiritual, emotional, and financial support.

To my brothers, sister, friends, and classmates who shared their words of advice and encouragement to finish this study

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Introduction

Since 1955, with Buonocore's introduction of the concept of treating enamel to chemically change its surface and hence facilitate the adhesion of filling materials to enamel surfaces, adhesion dentistry has rapidly changed and evolved. This is so because adhesion is necessary to oppose and withstand contraction forces during the polymerization of composite resin, and to promote better retention and marginal seal when the restored tooth is in operation (Poticny DJ. 2013).

Acid-etch adhesive systems can be used to achieve adhesion to dental structures. They can also be used to act as management and adhesive agents, as in the case of self-etch adhesives (Van Meerbeek B et al., 2011).

Acid-etching of the enamel surface gave rise to etch-and-rinse techniques, where both surfaces, enamel and dentin, are etched with acid. The acid is then removed so that the resin can adhere to the surfaces. Effective adhesion to dentin, at or above 17 MPa, has posed a much greater technological challenge than adhesion to enamel. (Spencer P, Ye Q et al., 2010)

State that current dentin adhesion systems focus on the formation of a hybrid layer on the dentin surface, which has polymerized monomers within a collagen network of the dentin, hence resulting in micromechanical interlocking. With traditional etch-and-rinse systems, this infiltration technique requires the dentin surface to be wet to provide support to collagen fibers, hence allowing for the necessary resin penetration to create a mineral/collagen/resin interface (Marshall SJ, Bayne SC et al., 2010). Total-etch or etch-and-rinse techniques have been used for decades, with excellent verified clinical results on the enamel. However, results on dentin are more variable (Qi CZ, Jiang Y et al., 2011).

The second group includes self-etch adhesive systems. These systems, characterized by acid monomers that do not require rinsing, have become more popular given their technical simplicity, the need to follow fewer steps and because the professional does not need to determine residual dentin moisture (Grégoire G et al., 2009). These systems etch, demineralize and infiltrate enamel and dentin simultaneously. The smear layer is impregnated but not eliminated, and rinsing is not indicated. Eliminating the etch-and-rinse step may reduce the risk of over-preparing the dentin, hence minimizing the problem of inadequate penetration of adhesive monomers and reducing the risk of postoperative sensitivity (Fernandes Pegado RE et al., 2010). These self-etch systems have presented adequate and stable dentin bonding forces, even stronger than those of older adhesive systems (SánchezAyala A et al., 20

1. Review of literature

Enamel and dentin are the dental substrates to which we bond our restorative materials. Cementum may also be involved when the cavo-surface margin is located apically to the cementum-enamel junction.

1.1 Enamel

Enamel is a dry substrate without vital structures containing 92 vol% of mineral phase (hydroxyapatite), which makes enamel almost the ideal substrate to form a tight adhesive joint. The acid-etch technique (M.G. Buonocore,1955) is still the gold standard for bonding resin-based materials to tooth structure. The micromechanical interaction of adhesives with enamel is a result of the diffusion and interlocking of resin monomers into the array of microporosities left by the acid chemical dissolution of enamel.

Bonding to enamel after etching with phosphoric acid is certainly the foundation for the durability of adhesive restorative procedures.

1.2 Dentin

The dentin is a mineralized connective tissue that makes the major portion of the tooth structure. Dentin is a vital tissue, continues to change through the life by physiological and pathological stimulus (Goldberg et al., 2011). Dentin provides support for enamel and act as shock absorber as dentin is tougher and can dissipate forces better than enamel which is hard but brittle. Dentin has also an important role in pulp protection from external stimuli (Eicket al., 1997).

Dentin is composed of 50% by volume of inorganic material, 30% organic matrix and the remaining is water. The organic matrix is formed almost

of type I collagen fibrils that constitutes 90% by volume and only 10% of noncollagenous protein and these collagen fibrils are arranged to form a mesh for the crystals of the inorganic material. Dentin is less mineralized than enamel but more mineralized than bone and cementum. Dentin minerals are mainly hydroxyapatite crystallites having a length of 20-100nm and a width of 3nm similar in dimensions to the hydroxyapatite crystallites found in cementum and bone but smaller than that in enamel (Teruel Jde et al., 2015).

Dentin is slightly yellow and becomes darker with age, harder than bone and cementum but softer than enamel. Dentin hardness near the pulp is one third of its hardness near the dentinoenamel junction (DEJ). Dentin becomes harder with aging due to mineral deposition, even though it is flexible with a modulus of elasticity about 18 gigapascals (MPa) (Goldberg et al., 2011).

1.2.1 Structure of dentin

A-Dentinal tubules

Dentinal tubules are small canals that run in a wavy pattern across the whole thickness of dentin from the pulp towards the DEJ in the crown and to the cementodentinal junction in the root (Eick et al., 1997).

Each tubule contains a cytoplasmic process of the odontoblastic cell. The diameter of the dentinal tubule is about 0.63μ m near the DEJ and increases towards the pulp to reach 2.37μ m near the pulp. The tubules are sparsely distributed near the DEJ and densely packed near the pulp and their numbers range from 15000-20000/mm2

near the DEJ to 45000-65000/mm2 near the pulp

(Trowbridge et al., 2002).

The increased number of tubules and the diameter of the tubules in approximation to the pulp increases the dentin permeability as we go deeper in dentin and the remaining dentin is not as an effective barrier as it is close to the DEJ (Kinney et al., 2003).

B-Peritubular and intertubular dentin

Peritubular dentin is the dentin that lines the tubule and forms its wall and surrounded by the intertubular dentin, the intertubular dentin forms the greatest portion of the dentin and lies between the tubules (Goldberg et al., 2011).

1.2.2 Types of dentin

According to Bhaskar (1997) there are three types of dentin; primary, secondary and tertiary that form in different developmental stages. 1997)

A-Primary dentin

Primary dentin forms the primary shape of the tooth and it is completed three years after tooth eruption. It starts from the point of the future cusp tip or the incisal edge and continue until complete root formation in the case of permanent teeth (Bhaskar, 1997).

B-Secondary dentin

After the formation of primary dentin, secondary dentin starts to form throughout the life in a very slow manner even without an external stimulus. Dentinal tubules are less regular in secondary dentin but usually continuous with those of the primary dentin. Secondary dentin deposits on the periphery of the pulp but in multi-rooted teeth they tend to deposit on the floor and roof rather than the walls (Bhaskar, 1997).

C-Tertiary dentin

When the tooth is subjected to a harmful stimulus like caries or abrasion, odontoblast forms tertiary dentin as a reparative process in response to this stimulus and it is characterized by fewer dentinal tubules with more twisted form (Bhaskar, 1997).

1.2.3 Smear layer

After tooth preparation, a layer of debris is formed on the dentin surface and termed "smear layer". Smear layer may vary in its thickness, roughness or adhesion to the dentin depending on the cutting procedure of the dentin. This layer plays an important role in adhesive dentistry because it must be removed or modified to achieve a successful bond between the dentin and the restoration (Oliveira et al., 2004).

Smear layer thickness is $1-10 \ \mu m$ and consists of two zones, the inner zone has a disordered crystalline arrangement while the outer zone contains destructed collagen fibrils and appetite crystallites formed during mechanical cutting and the heat of friction during the preparation (Pashley and Carvalho, 1997).

1.3 Adhesive systems

Adhesive systems can be considered revolutionary in many aspects of conservative dentistry, making possible previously inconceivable clinical maneuvers. Current adhesive systems allow clinicians to bond to tooth structure without the need of a retentive cavity since they provide immediate bond strength (Breschi et al., 2018).

1.3.1 The basic components of a dental adhesive system

A) Etchant, currently phosphoric acid in a concentration between 30% and 40%. Most phosphoric acid gels are thickened with sil- ica microparticles, although there are a few that contain other thickeners such as xanthan gum. A color dye is always included to improve the application accuracy and ensure that all gel is washed off. Glycol is often added to improve wettability and decrease viscosity. The etchant is always rinsed off from the tooth surface.

B) Primer which is a hydrophilic solution of resin monomers, organic solvent (alcohol or acetone), water, and stabilizers. The hydrophilic groups boost the wettability to the dentin surface, which is a humid environment. The role of primers in dentin adhesives is comparable to that of primers in paints. The primer adheres to surfaces and forms a binding layer that is better pre- pared to receive the paint, in this case, the bonding resin. Primers are not usually rinsed off nor cured once placed on the tooth surface; they are only air-dried.'

C) The bonding resin is a solvent-free (hydrophobic) low-viscosity resin that is applied over the primer and then light-cured. The hydrophobic groups interact and copolymerize with the restora- tive material and make dentin bonding more stable and more durable by sealing the bonded interface against nanoleakage (Sezinando A,2015)(Perdigão J,2013). The hydrophobic resin improves both the polymerization rate of the primer and the mechanical properties of the adhesive and hybrid layer(Perdigão J,2013). Adhesive systems that have this separate bonding step result in better in vitro and clinical out- comes(Peumans M,2014).

1.3.2 Classification of adhesive systems

Dental adhesives are currently categorized using two different classifications.

A-<u>By generation</u> – from first to eighth generations.

It is used mostly by the dental industry to highlight the latest trend. It is a confusing nomenclature, as the first dentin adhesives that used a phosphoric acid etchant on enamel and dentin are known as the fourth generation. This classification is not very informative either, especially when considering the missing components of the dental adhesive, i.e., etchant, primer and bonding resin.

First-Generation Adhesives (1960)

• Development of surface-active comonomer NPG-GMA

• Theoretically, this comonomer could chelate with calcium on the tooth surface to generate water-resistant chemical

bonds of resins to dentinal calcium • Bond strength 2 to 3 MPa.

Drawbacks

- It showed poor clinical results
- Example: Cervident (SS white), cosmic bond(Kenneth J,2003)(Roberson Theodore,2002).

Second-Generation Adhesives (Late 1970s)

Phosphate ester dentin bonding agents were introduced containing phenyl P and HEMA in ethanol

- Its mechanism of action was based on the polar interaction between negatively changed phosphate groups in resin and positively changed Ca++ in smear layer
- The bond strength was 5 to 6 MPa.

Drawbacks

• Loosely attached smear layer and hydrophobic nature

• Example: ScotchBond (3M dental), Clearfil bond system.

Third-Generation Adhesives (1980s)

• The third generation materials were designed not to remove the entire smear layer but rather to modify it and allow penetration of acidic monomers, such as phenyl-P and Penta

• These introduced acid-etching to heavily alter or to remove the smear layer and demineralizing dentin and a separate primer (bifunctional monomer in a volatile solvent) designed penetrate dentin by its own monomer and those of adhesive monomers

• Adhesive is an unfilled or partially filled resin that may contain some component of primer (e.g. HEMA) in an

attempt to promote increased bond strength

- Bond strength is 3 to 8 MPa
- Examples: ScotchBond 2, Tenure, Universal bond 2,

Coltene ART.

Fourth-Generation (Early 1990s)

• When primer and bonding resin are applied to etched dentin, they penetrate the intertubular dentin forming a resin dentin interdiffusion zone or hybrid layer

- They have the ability to bond as strongly to dentin as to enamel (total etch)
- Ability to bond to moist dentin (wet bonding)
- Multiple substrate bonding to metal, amalgam, porcelain and indirect composite
- Bond strength 13 to 30 MPa
- Examples: All-Bond 2, OptiBond FL and ScotchBond

multipurpose.

Fifth-Generation Adhesives

These are essentially distinguished by being 'one-step' or 'one- bottle' system. This is a bit of a misnomer because these products are applied in two steps (etchant + primer and adhesive) in one bottle.

• Bond strength is 3 to 25 MPa.

DRAWBACKS

- They lack many of the components necessary to perform multisubstrate bonding
- Multiple coats of these agents are required
- Examples: Prime and bond, single bond, OptiBond Solo

and OptiBond Solo Plus(Kenneth J,2003)(Roberson Theodore,2002).

Sixth-Generation Adhesives

It was introduced in late 1990 and early 2005:

1. They dissolve the smear layer when applied and do not require rinsing.

2. Minimize postoperative sensitivity as they do not expose dentinal tubules.

3. Bond strength to enamel and superficial dentin are typically greater than deep dentin.

The bond strength to dentin and enamel is lower than the fourth- and fifth- generation systems(Farah John W,2004).

Seventh-Generation

Introduced in Late 2002

- Self-etching adhesive
- Require no mixing
- Not compatible with self-cured composite cores or resin

cements

- Single bottle containing acidic adhesive
- Examples—iBond
- Bond strengths and marginal sealing to be equal to the sixth-

generation system.

Eighth-Generation

• Dual-cured self-etch adhesive for direct and indirect restora- tions with self-, lightand dual-cured resin materials(The Dental Advisor,2008)(Krithikadatta J,2010)(VOCO,2011).

B-By adhesion strategy – with or without etching enamel and dentin simultaneously with phosphoric acid

Three step etch and rinse adhesive

In three step etch and rinse a(Perdigão, 2001 #156)(Perdigao, 1999

#64)dhesive system, the smear layer is totally removed by acid etchant to permit resin bonding to the underlying dentin substrate (Van Meerbeek et al, 1992). Acids demineralize intertubular and peritubular dentin, open the dentin tubules, and expose a dense filigree of collagen fibrils, increasing the microporosity of the intertubular dentin. Dentin is demineralized by up to approximately 7.5 μ m, depending on the type of acid, application time, and concentration (Perdigao et al, 2001).

This bonding system consists of three essential components that are applied sequentially, the three essential components are (1) a phosphoric acid– etching gel that is rinsed off; (2) a primer containing reactive hydrophilic monomers in ethanol, acetone, or water; and (3) an unfilled or filled resin bonding agent. Some authors refer to this third step as adhesive. It contains hydrophobic monomers such as Bis-GMA, frequently combined with hydrophilic molecules such as HEMA (Sturdevant, 2014).

The acid-etching step not only alters the mineral content of the dentin substrate but also changes its surface free energy. The latter is an undesirable effect because for good interfacial contact, any adhesive must have a low surface tension, and the substrate must have a high surface free energy (Tsujimoto et al, 2017).

After etching, the dense web of exposed collagen is a low surface energy substrate. The primer in a three-step system is designed to increase the critical surface tension of dentin, and a direct correlation between surface energy of dentin and shear bond strengths has been shown (Erickson et al, 1992). When primer and bonding resin are applied to etched dentin, they penetrate the intertubular dentin, forming a resin–dentin interdiffusion zone, or hybrid layer. They also penetrate and polymerize in the open dentinal tubules, forming resin tags. For most etch-and-rinse adhesives, the ultramorphologic

characterization of the transition between the hybrid layer and the unaffected dentin suggests that an abrupt shift from hybrid tissue to mineralized tissue occurs, without any empty space or pathway that could result in leakage. The demarcation line seems to consist of hydroxyapatite crystals embedded in the resin from the hybrid layer. For self-etch systems, the transition is more gradual, with a superficial zone of resin-impregnated smear residues and a deeper zone, close to the unaffected dentin, rich in hydroxyapatite crystals (Sturdevant, 2014).

Two-step etch and rinse adhesive system

In vitro dentin bond strengths have improved so much that they approach the level of enamel bonding. Therefore, much of the research has focused on the simplification of the bonding procedure by reducing number of steps into twostep etch-and-rinse adhesive system. They are sometimes called "one-bottle" systems because they combine the primer and bonding agent into a single solution and a separate etching step still is required (Swift et al, 2001). With this system, primer and adhesive are mixed in one bottle and the application of two coats are recommended. The first coat applied on etched dentin works as a primer-it increases the surface free energy of dentin The second coat acts as the bonding agent used in three-step systems-it fills the spaces between the dense network of collagen fibers (Freedman and Goldstep, 1997).

Two-step self-etch adhesive system

An alternative bonding strategy is the self-etch approach. a type of acidic conditioner was introduced -the self-etching primers (SEPs)- and has proved to be successful. These acidic primers include a phosphonated resin molecule that performs two functions simultaneously; etching and priming of dentin and

enamel (Perdigao and Lopes, 1999).

In contrast to conventional etchants, SEPs are not rinsed off. The bonding mechanism of SEPs is based on the simultaneous etching and priming of enamel and dentin, forming a continuum in the substrate and incorporating smear plugs into the resin tags. In addition to simplifying the bonding technique, the elimination of rinsing and drying steps reduces the possibility of over-wetting or over-drying, either of which can affect adhesion adversely. Also, water is always a component of SEPs because it is needed for the acidic monomers to ionize and trigger demineralization of hard dental tissues; this makes SEPs less susceptible to variations in the degree of substrate moisture but more susceptible to chemical instability due to hydrolytic degradation (Fukuoka et al, 2011).

One disadvantage of SEPs that are currently available is that they do not etch enamel as well as phosphoric acid, particularly if the enamel has not been instrumented. The seal of enamel margins in vivo might be compromised (Patil et al, 2013).

When enamel bonds are stressed in the laboratory by thermal cycling, SEPs are more likely than etch-and-rinse systems to undergo deterioration. This decrease in bond strengths with thermal fatigue might be a sign that a potential exists for enamel microleakage when SEPs are employed to bond to enamel (Sturdevant, 2014).

SEPs have been classified in three categories: mild, moderate, and aggressive. Mild SEPs tend to provide excellent dentin bond strengths and poorer enamel bonds, whereas more aggressive self-etch systems provide the reverse (Ermis et al., 2009).

SEPs are less technique sensitive than are etch-and-rinse adhesives. Additionally, SEPs are less likely to result in a discrepancy between the depth

of demineralization and the depth of resin infiltration because SEPs demineralize and infiltrate dentin simultaneously. SEPs do not remove the smear layer from dentin completely, which is the main reason that they might result in less postoperative sensitivity compared with etch-and-rinse adhesives (Christensen, 2002).

One-Step Self-Etch Adhesives

Continuing the trend toward simplification, no-rinse, self-etching materials that incorporate the fundamental steps of etching, priming, and bonding into one solution have become increasingly popular. these one-step self-etch or "all-in-one" adhesives contain uncured ionic monomers that contact the composite restorative material directly. Their acidic unreacted monomers are responsible, in part, for the incompatibility between these all-in-one adhesives and self-cured composites. Additionally, one-step adhesives tend to behave as semi-permeable membranes, resulting in a hydrolytic degradation of the resin-dentin interface (Tay et al, 2004).

Because these adhesives must be acidic enough to be able to demineralize enamel and penetrate dentin smear layers, the hydrophilicity of their resin monomers, usually organophosphates and carboxylates, also is high. Some of these resin monomers are too hydrophilic, which makes them liable to water degradation (Tay and Pashley, 2003).

1.4 Mechanisms Of Bonding

1.4.1 Bonding to Enamel

In enamel, etch & rinse technique is still the most effec- tive approach to achieve efficient and stable bonding and requires selective dissolution of hydroxyapatite crystals through etching (Van Meerbeek,2003). Two types of resin tags interlock within the etch-pits. Macro-tags fill the space surrounding the enamel prisms, while numerous micro-tags result from resin infiltration and polymerization within the tiny etch-pits at the cores of the etched enamel prisms. The latter are espe- cially thought to contribute the most with regard to retention to enamel (Van Meerbeek, 2003).

Self-etch adhesives use acidic monomers to condition tooth structure rather than traditional phosphoric acid, how- ever they do not produce the same degree of porosity in enamel surfaces as that attained with phosphoric acid etch-ing in etch & rinse systems (Hannig M,2002). Since enamel bonding is pri-marily based on micromechanical interlocking of a low viscosity resin into microporosities, the extent and depth of the etching pattern should logically influence the bonding performance of an adhesive. Scanning electron microscopic studies indicate that an enamel etching pattern caused by self-etch adhesive is not as deep and appears to be less reten- tive compared with the etching pattern resulting from phos- phoric acid treatment and that the degree of enamel etching depends on the pH of the self-etch adhesive (Hanning M,1999)(Kanemura N,1999). The weak acidity of these self-etch adhesives raises the question of whether the adhesives are able to penetrate the enamel surface and yield durable bonding with the restored tooth. Self-etch adhesives create an irregular, non homogenous etch pattern, whereas phosphoric acid removes the enamel smear layer and leads to a honey-comb structure sur- face (Kanemura N,1999)(Breschi L,2003). The demineralization depth of enamel surface is lower for self-etch adhesives compared to the etch & rinse approach (1.5-3.2µm vs. 6.9µm) (Hannig M,2002). The shallower etching pattern on enamel and subsequent reduced micromechani- cal retention might jeopardize bonding. Thus bonding of self-etch systems to enamel still remains critical and is con- troversially discussed by various authors (Brackett WW,2006)(Watanabe T,2008).

1.4.2 Bonding to Dentin A-Dentin conditioning

Two mechanisms for dentin conditioning are available, in the fourth and fifth generation of adhesives which are termed 'etch and rinse adhesive systems' the smear layer is completely removed and the dentinal tubules are opened by acid etchants. In the sixth and seventh generations which are termed 'self-etch adhesive systems' the smear layer is incorporated in the hybrid layer by the acidic primer which partially demineralize and infiltrate it simultaneously (Breschi et al., 2004; Pashley et al., 2011a).

The demineralization effect of acid etchant used in etch and rinse adhesive systems depends on its type, concentration, time of application, wettability, viscosity, water content and PH (Perdigao, 1995; Scheffel et al., 2012). Time of acid application is not proportional with the degree of penetration into dentin due to the buffering action of dentin that prevents acid infiltration to deep layers during dentin demineralization (Hashimoto et al., 2002).

Acid etchant also affects surface free energy, proteins exposure after dentin conditioning leads to a drop in surface free energy and low wettability that is difficult to provide good adherence to its surface (Yesilyurt and Bulucu, 2006).

After acid etching with the etch and rinse technique; a collagen network will be exposed as shown in Figure (1-1) and must be infiltrated by resin to form the hybrid layer. If dentin is air dried before bonding, collagen network will collapse and prevent the infiltration of the bonding resin and poor quality hybrid layer will be formed (Nishitani et al., 2006a



Figure (1): Scanning electron micrograph of etched dentin showing exposed collagen fibrils (Sturdevant, 2014)

When dentin is kept moist, collagen network will be distended and allow easy infiltration of the resin monomer and high quality hybrid layer will be formed with higher bond strength of the restoration as shown in Figure (1-2) (Cardoso et al., 2005).

With air drying of the etched dentin, evaporation of the water inside he collagen fibrils will happened and rewetting can expand these fibres again and a bond strength will be restored to normal levels (Perdigao et al., 2002).





B- Priming of dentin

Is the intermediate agent that is applied between conditioning and bonding. After demineralization of dentin; a protein layer is exposed which reduce surface free energy and hence wettability. The primer raises the free energy and increase affinity of dentin to resin infiltration efficiently and form a hybrid layer for bonding. Primer consists of bifunctional molecules, each molecule has two functional groups, one of them is hydrophilic and has affinity for dentin while the other is hydrophobic with affinity for the adhesive resin (Van Meerbeek et al., 1998). A main constituent of the primer in many adhesive systems is hydroxyethyl methacrylate (HEMA). HEMA molecule resembles methyl methacrylate except that the methyl ester group was replaced with ethoxy ester group to make it hydrophilic (Carvalho et al., 2003). Modern adhesive systems are hydrophilic and contain resin monomers dissolved in acetone, ethanol, water or combination of these with organic solvents and termed as water chasers. They expel water from dentin and replace it with resin monomer (Reis et al., 2004).

C-Bonding resin application

After the priming of dentin, an adhesive resin based on Bisphenol A glycol dimethacrylate (Bis-GMA) is applied which will infiltrate between collagen fibrils and into open dentinal tubules to form the hybrid layer and the resin tags which are the micromechanical retentive mean for the restoration (Van Meerbeek et al., 1998).

i- Hybrid layer

Polymerization of infiltrated resin into collagen fibrils form the hybrid layer as mentioned previously and along with the resin tags form a hermetic seal on the opened dentinal tubules (Swift et al., 1995). There are three different zones within the hybrid layer. The top of the hybrid layer consists of an amorphous electro-dense phase, which has been ascribed to denatured collagen. At the middle of the hybrid layer there are cross sectioned and longitudinally sectioned collagen fibrils which separated by electro-lucent spaces, these spaces represent areas where hydroxyapatite crystals had been removed and replaced by resin as a result of the process of hybridization. The base of the hybrid layer represents the gradual transition to the underlying intact dentin with partially demineralized zone of dentin that contain hydroxyapatite crystals enveloped by resin (Perdigao, 1995). The ideal percentage of resin in the hybrid layer is 70% by weigh which is difficult to achieve in most situations due to collapse in the collagen fibrils, incomplete resin infiltration (Eick et al., 1997).

ii- Resin tags

Resin infiltration inside dentinal tubules and polymerization in situ leads to the formation of resin tags. Resin can penetrate to 2-4 µm distance inside the tubules,

these tags increase area available for retention to 30-40% (Prati et al., 1998). Resin tags are important to get a good seal to prevent pulpal irritation and improve retention of the restoration. The percentage of the strength added by the resin tags to the total bond strength depends on the diameter of the tags and the cohesive forces binding the resin molecules (Pashley and Carvalho, 1997). Micromechanical retention in superficial layers of dentin occur primarily by the hybrid layer with little participation of the resin tags because of the low number of resin tags in the superficial dentin. In deep layers of dentin, the number of tubules are increased with larger diameter and limited amount of intertubular dentin; for these reasons the retention will be majorly by resin tags and minimally by intertubular dentin (Pashley and Carvalho, 1997).

1.5 Dental composite

Resin based composite had evolved greatly since its introduction. The evolution of resin based composite was toward reducing the particle size of the filler to produce a material with high wear resistance and high polishability, then the focus was shifted toward improving the matrix of the composite to reduce the polymerization shrinkage and more importantly polymerization shrinkage stress and to make it self-adhesive to tooth structure (Ferracane, 2011).

1.6 Bond strength

The bond strength is defined as the force per unit area needed to break up two bonded surfaces at or close to the adhesive interface (Versluis et al., 1997).

Longevity of a restoration is predicted to some extent by its adhesive ability, and this in turn can be measured by bond strength testing. Though the validity of bond strength tests to predict clinical performance of dental adhesives is questionable, existing evidence shows that clinical performance can be predicted by appropriate types of laboratory study results (Sirisha et al., 2014)

1.6.1 Factors affecting bond strength

A- Enamel prism orientation

Composite resin restorations will have a longer life span if the enamel walls are given marginal forms to produce transverse sections of enamel prisms rather than longitudinal sections (Munechika et al., 1984)

B-Dentin depth and permeability

The dentinal tubules are fluid filled from the pulp to the DEJ. If the dentin is exposed to the external surface for any reason, fluid will move inside the tubules and activate pulpal nerves which cause pain (Pashley et al., 1993). The diameter and number of tubules per square millimetre (density of tubules) increase with increasing depth in dentin while approaching the pulp, this lead to an increase in fluid movement through the tubules and increase in permeability. permeability differs from tooth to another and from age to age (Ulu Guzel et al., 2018).

Dentin permeability increases almost logarithmically with cavity depth. In majority of studies, lower shear bond strengths were reported with increased dentin depth and permeability, and this permeability is less when smear layer was retained (Tagami et al., 1990; Prati and Pashley, 1992).

Dentin permeability sophisticates the bonding process which is more difficult than enamel, after acid etching; the smear layer and plugs are removed which increase the permeability (Soderholm, 1991).

To optimize dentin bonding, resin must infiltrate dentin in both

intratubular and intertubular way to form a continuous layer of hybrid layer with resin tags. In superficial dentin which contains fewer tubules; intertubular resin infiltration will be responsible of the bonding process (Nakabayashi et al., 1991).

In the deep layers of dentin where the density of dentinal tubules is high, intratubular infiltration of resin will be responsible of a large part of the bond strength, intratubular infiltration may encounter difficulties in the deep layers due to pulpal pressure that push the fluids away from the pulp and reduce resin infiltration (Pashley and Carvalho, 1997).

Dentin affected by caries has low permeability due to bacterial presence and calcium deposits within dentinal tubules, therefore a low bond strength will be achieved by bonding to carious dentin. Sclerotic wedge shaped defects in the cervical dentin is another example of a low bond strength substrate due to minerals deposition inside the tubules that reduce resin infiltration (Eick et al., 1997).

C- Dentinal tubules orientation

Dentinal tubules that run parallel to the bonded surface has greater amount of peritubular dentin than dentin with tubules running perpendicularly to the bonded surface providing more surface area for conditioning and a thicker hybrid layer will be formed resulting in higher bond strength values (Sattabanasuk et al., 2004).

D- Degree of dentin mineralization

Degree of mineralization may be increased in dentin in response to physiological or pathological stimulus, if the dentin is exposed to the oral cavity in non-carious cervical lesions or if it is close to caries for a prolonged period of time; tricalcium phosphate deposits and obliterates dentinal tubules.

Hypermineralized dentin is termed "sclerotic dentin" and it interferes with resin infiltration and produce a weaker bond strength than with normal dentin (Hosoya, 2006)

E- Surface area of the bonded interface

Studies had demonstrated that there is no linear correlation between the bond strength and the dentinal surface area at the bonded interface. In other words, doubling the interface available for bonding will not double the force needed to break the bond and this can be explained by increase in surface defects and flaws with increase in surface area available for bonding (Phrukkanon et al., 1998).

1.6.2 Factors related to adhesive material and technique

A- Surface wetness

Studies had shown that wet bonding, in which, dentin is dried but left visibly moist gives bond strength greater than dry bonding technique (Irmak et al., 2016).

Pooled moisture should not remain on the tooth because excess water can dilute the primer and render it less effective (Van Dijken, 2000).

B- Etching time

When etching time is prolonged a wide zone of demineralized dentin will be created and it is difficult to be infiltrated completely by resin, thus a resin free zone will be crated between the hybrid layer and the intact dentin and this zone will create a pathway for nanoleakage and bond degradation (Perdigão and Lopes, 2001).

Studies had shown that prolonged etching time (over etching) did not increase bond strength but actually reduce it and specimens fractured from the demineralized zone that was not infiltrated by resin (Zafar and Ahmed, 2015)

C-Number of adhesive resin application

An important way to improve bond strength is by increasing the number of adhesive resin application to improve resin infiltration and creating a high quality hybrid layer. However, there is no consensus about the number of applications needed to achieve good results (D'Arcangelo et al., 2009). Some studies found that two applications are enough to produce a good bond strength while other studies found that several applications lead to better results (Carvalho et al., 2017)

D- Use of hydrophobic coating

Since the incorporation of hydrophilic monomer blends in simplified adhesives (two-step etch-and-rinse and one-step self-etch adhesives) dramatically reduced bond longevity, the need of a hydrophobic coating with a not-solvated bonding layer seems to be pivotal to reduce water sorption and stabilize the hybrid layer over time, i.e., etch-and-rinse three steps and selfetch two-step adhesives should be preferred to simplified ones. Also applying a hydrophobic layer on one-step self-etching adhesives could improve bond strength and durability (Reis et al, 2008).

E-Extended polymerization time

Extending the curing times of simplified adhesives beyond those recommended by the manufacturers resulted in improved polymerization and reduced permeability and appears to be a possible means for improving the performance of these adhesives (Samimi et al, 2017).

F- Use of MMPs inhibitors

The use of MMPs inhibitors as additional primer has been claimed to reduce interfacial aging over time by inhibiting the activation of endogenous dentin enzymes which are responsible for the degradation of collagen fibrils in the absence of bacterial contamination (Li et al, 2018).

i- Chlorhexidine

Chlorhexidine is a bisbiguanide antiseptic consisting of two biguanide groups and four chlorophenyl rings. The biguanide groups are bonded by a central hexamethylene bridge (Figure (1-4)). Chlorhexidine is a strong base with two positive charges (cations) on both sides of the hexamethylene bridge (Albert and Serjeant, 1962).



Figure(3): Chlorhexidine molecule (Albert and Serjeant, 1962).

Chlorhexidine as a matrix metalloproteinase inhibitor

CHX has a potential MMPs inhibitory effect by calcium and zincchelation mechanism (Gendron et al., 1999).

Application of 2% chlorhexidine for 1 minute between etching and bonding steps inhibits hybrid layer degradation for at least 14 months. CHX was also found to inhibit cysteine cathepsins enzyme that presents in mineralized and non-mineralized dentin which is responsible for MMPs activation and extracellular matrix degradation (Nascimento et al., 2011)

G-Improved impregnation

Various methods have been recently proposed to enhance dentin impregnation, i.e., prolonged application time, vigorous brushing technique, and electric impulse assisted adhesive application. The latter technique recently revealed increased bond strength and reduced nanoleakage expression if adhesives are applied under the effects of an electric signal. Junior et al (2008) improved impregnation of dentinal collagen by adhesives via the evaporation of adhesive solvent by a stream of warm air. The use of a warm airdry stream to evaporate the solvent of adhesives seems to be a clinical tool to improve the bond strength and the quality of the hybrid layer (less nanoleakage infiltration).

Another approach to improve impregnation of collagen by the adhesive after acid etching was the simultaneous acid etching and deproteinization suggested by Nassif and El Korashy. The simultaneous etching and deproteinization by NaOCl/phosphoric acid for 15 s showed a hybrid layer with improved bond strength. This was attributed to removal of shredded collagen found in the smear layer that could not be removed by acid etching only. Removal of this disorganized collagen would give more open structure to the collagen network and improve its impregnation by the adhesive (Nassif and El-Korashy, 2009)

H- Filler contents in the adhesive products

Characteristics of fillers like shape, size, type and interaction with matrix and solvents may affect bond strength. Filled adhesives may act as a shock absorber that dissipates functional forces thus increasing bond strength of composite to dentin (Swift et al., 2001).

The loading of adhesive resin with fillers and nanoparticles has led to a significant reinforcement effect of the adhesive. Carboxylic acid functionalized titanium dioxide (Sun et al., 2017), copper (Gutierrez et al., 2017), silver (Torres-Mendez et al., 2017), and zinc oxide (Barcellos et al., 2016) nanoparticles have been used to reinforce the organic matrix of resin adhesives, thus improving physicomechanical properties of the material, and, consequently, bond strength between the restoratives and dental substrates. In another study by (Lohbauer et al., 2010), zirconia nanoparticles were incorporated into the primer or adhesive of a commercial three-step etch-andrinse adhesive system (SBMP, Scotchbond[™] Multipurpose[™]; 3M ESPE, St. Paul, MN, USA) resulting in increased dentin bond strength. The formation of a strong adhesive interface is usually associated with a higher resistance to hydrolytic phenomenon, which may enhance bond durability. Once the hybrid layer is strong, water uptake is reduced, hydrolysis is diminished, and proteases activity is retarded, thereby reducing the rate of bond degradation over time (Carvalho et al., 2016).

I- Type of organic solvent

Contemporary total-etch adhesives mainly contain ethanol/water or acetone as a solvent, in which resin monomers are dissolved (Van Landuyt et al., 2007). Solvents are responsible for water displacement from collagen network and infiltration of resin monomers into spaces previously occupied by

water (Kanca, 1992). Composition of the adhesive and solvent type requires different moisture spectrums (Tay et al., 1998). Acetone based systems evaporate much residual water than ethanol/water based systems; however, they are more sensitive to air-drying as they cannot re-expand the shrunken collagen fibrils (Pashley et al., 2002). Ethanol/water based systems are less moisture sensitive and good at re-expanding collagen matrix and produce higher bond strengths in dried dentin (Reis et al., 2003). Wet bonding may be the ideal technique for current adhesives; however, this moisture concept varies widely among clinicians and manufacturers. Drying time and air-syringe distance, air pressure are variables that also have an effect on bond strengths and not easy to control (Kanca, 1996).

An adhesive containing a different solvent, tertiary butanol is on the market. According to its manufacturer, this adhesive system has a high degree of technique robustness due to chemical composition of tertiary butanol (XP BOND. Konstanz, Germany: Dentsply DeTrey, 2006) (Irmak et al., 2016)

1.7 Matrix metalloproteinases

Matrix metalloproteinases (MMPs) are zinc and calcium dependent endopeptidases that can degrade extracellular matrix proteins, growth factors, lipoproteins and cell adhesion molecules (Osorio et al., 2011). They are cell derived proteolytic enzymes that are secreted as inactive form (proenzyme) which don't cleave extracellular matrix proteins. The MMPs are trapped in the dentin during its formation (Mazzoni et al., 2007). MMPs consist of a prodomain, a catalytic domain with a highly conserved zinc binding site, a hinge region and a hemopexin domain (Figure (1-4)). The catalytic domain contains cysteine rich repeats that are necessary for the binding and cleaving activities of these proteolytic enzymes (Van Wart and Birkedal-Hansen, 1990; Visse and Nagase, 2003)



Figure (4): Structure of MMP (Visse and Nagase, 2003).

1.7.1 Durability of bond strength with etch and rinse adhesives

Even though water is necessary at the beginning of resin infiltration, it must be eliminated from dentin after serving its purpose otherwise it will cause hydrolytic degradation of the resin matrix by esterase and degradation of collagen by the collagenolytic and the gelatinolytic enzymes (Liu et al., 2011). Another problem related to the etch and rinse technique is that resin concentration is reduced as we go deeper towards the bottom of the hybrid layer, this phenomenon is more prevalent in carious dentin than sound dentin which is the usual cause for tooth restoration. When resin infiltration is not complete, denuded collagen fibrils will be the result which are susceptible to enzymatic attack and fatigue failure (Wang et al., 2007). In vitro researches had shown that resin dentin interface deteriorates with time due to degradation of the collagen fibrils at the interface (De Munck et al., 2009); in addition to the hydrolytic degradation of the adhesive resin (Hashimoto et al., 2000). Proteolytic degradation of collagen fibrils may also be responsible for the diminished dentin bond strength over time (Hashimoto, 2010). In vivo studies have also found that collagenous part of the hybrid layer undergoes a gradual hydrolytic degradation (Hashimoto et al., 2003a).

Conclusion

Reliable bonding of resins to enamel and dentin has revolutionized the practice of operative dentistry. Improvements in dentin bonding materials and techniques are likely to continue. Even as the materials themselves become better and easier to use, however, proper attention to technique and a good understanding of the bonding process remain essential for clinical success

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