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# Bonding Agents in Orthodontic

**(A Review Study)**

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

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# Certification of the Supervisor

I certify that this project entitled "**Bonding Agents In Orthodontic**" was prepared by the fifth-year student **Noor Riyadh Mohammed Amin** 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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B.D.S., M.Sc. (Orthodontics)

May, 2022

# Dedicated

To my devoting parents, to my best friends for their continues support and love Zahraa and Fatima, to my cousin Rana Al-Shaarbaf for her special advice, to Zahraa Al-Taweel who always be my side, to colleague Hiba Adel who helped me in editing this project, for study partner May M., Maryam A., Kawther A., Huda A., Mina Q., Nabaa Q., Nadiaa F., Mena M., Farah A., Noor AL-Huda A., Hadiea A., Farah B., Hiba A.

And for all those who encouraged me to fly toward my dreams.

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# List of abbreviations

| | |
|---|---|
| SE | Self etch |
| SEP | Self etch primer |
| E&R | Etch and rinse |
| Bis-GMA | Bisphenol-glycidyl methacrylate |
| HEMA | 2-Hydroxyethyl methacrylate |
| 10-MDP | 10-Methacryloyloxy decyl dihydrogen phosphate |
| 4-META | 4-Methacryloxyethyl trimellitate anhydride |
| NPG-GMA | N-phenylglycine glycidyl methacrylate |
| PENTA | Dipentaerythritol penta-acrylate monophosphate |
| TEGDMA | Triethylen glycol dimethacrylate |
| DBa | Dentin bonding agent |
| pH | Power of hydrogen |
| °C | Degree Celsius |
| µm | micrometer |
| % | Percentage |
| H. | Hour |
| min | minute |
| SBS | Shear bond strength |
| S. | Second |

| | |
|---|---|
| MPa | Mega pascal |
| E | Etch |
| P | Primer |
| PB | Primer and bond |
| SEAs | Self etch adhesive system |

# Introduction

From the inception of fixed-appliance orthodontic treatment, brackets traditionally have been welded to gold or stainless-steel bands. The band encompassed the tooth circumferentially, requiring the creation of interproximal space to accommodate the width of the band material. This separation process, which was accomplished initially by placing wires and later elastomeric, was time consuming for the orthodontist and uncomfortable for the patient. At the conclusion of treatment, these interproximal gaps had to be addressed again. In addition, banded appliances frequently caused gingival trauma when fitted, and decalcification under bands sometimes occurred during treatment. Therefore, the obvious solution to these problems was for the clinician to attach the brackets directly to tooth enamel, thus eliminating the need for bands. The development of adhesives which will successfully bond orthodontic attachments directly to enamel has been greatly influenced by the research work directed towards improving adhesive properties of materials used in conservative dentistry **(Gange, 2015)**.

Bonding brackets has some advantages, including ease of placement and removal, minimal soft tissue irritation and hyperplastic gingivitis, minimal danger of decalcification with loose bands, and being more esthetic **(Attar *et al.*, 2007)**. Current adhesive materials simplify bonding procedures by reducing the number of application steps and time required for application. This simpler protocol makes them less technique sensitive and allows for better application standardization **(Van Meerbeek *et al.*, 2005)**.

# Aim of the study

- ❖ Review the history of bonding agents from its beginning to the generations that's available nowadays by manufactures including its components, properties, advantages, drawbacks and the longevity of the bonding strength.

# Chapter one:
# Review of literatures

## 1.1 Enamel

The hardest biological tissue in the body of human; it caps the crowns of the teeth providing the contour, shape, and the ability to resist the force of mastication as well as protection against any harmful stimuli in the oral environment **(Chiego, 2014).**

Enamel is a thick layer made from calcified material covering the dentin and varies in thickness in different areas of tooth, at the incisal is thicker than occlusal areas of the tooth and becomes sequentially thinner until it ends at the cemento-enamel junction **(Paulsen, 2010).**

The greater mass of enamel is made of prisms, except for a few microns on the outer surface, which is frequently prism less, the enamel prisms extend from the dentine-enamel junction to the outer surface at varying angles which differ from tooth to tooth and from one surface to another within the same tooth, and they are oriented in such a way that the head portion are directed toward the incisal and cuspal regions and tail portions towards the cervical region of the tooth crown **(Nanci, 2013).**

The response of the enamel to acid etching agents as a result of the direction of the crystals within the prisms, compositional differences on its surface within a tooth, morphological differences between teeth, presence of prism less enamel on certain areas of tooth surface, structural defect in either the organic and inorganic of enamel components and due to the presence of acquired organic pellicle **(Van meerbeek *et al*., 2003)**.

Two distinct surface changes were noted after etching. First, a shallow layer of enamel was removed along with plaque and surface and subsurface cuticles. Second, the remaining enamel surface was rendered porous by the acid solution. It is this porous region that the resin can penetrate and micromechanically bond with the enamel **(Silverstone *et al.*, 1975).**

The cumulative enamel loss of pumicing, bonding, debonding, and clean ups is difficult to estimate precisely as minerals are not dissolved in a uniform way. Residual adhesive may persist in surface enamel after debonding. Resin tags can reach more than 20 μm into the enamel after bonding based on the acid-etching principle, and alteration of the prism structure even further **(Øgaard and Fjeld, 2010).**

## 1.2 Adhesion

When two substances are brought into intimate contact with each other, the molecules of one substance adhere, or are attracted to, molecules of the other substance. This force is called adhesion when unlike molecules are attracted and Cohesion when molecules of the same kind are attracted. The material or film used to cause adhesion is known as the adhesive; the material to which it is applied is called the adherend. In a broad sense, adhesion is simply a surface attachment process. The term adhesion is usually qualified by specification of the type of intermolecular attraction that may exist between the adhesive and the adherend **(Anusavice et *al.*, 2003).**

### 1.2.1 Wettability and Contact angle

**Anusavice *et al.* (2003)** demonstrated an important requirement for the occurrence of any of these interfacial phenomena is that:

- Two materials being joined must be sufficiently wetted with close and intimate relation.
- An intimate contact, sufficient wetting of the adhesive will occur only if the surface tension is less than the surface energy of the adherend.

- Wetting of a surface by a liquid is characterized by the contact angle of a droplet placed on the surface. The smaller contact angle, the better the wetting ability of the adhesive, as shown in **(Fig. 1)**.

Chemical match between the adhesive and the adhered surface and thin film thickness of the adhesive also result in good wetting **(Roberson *et al.*, 2002).**

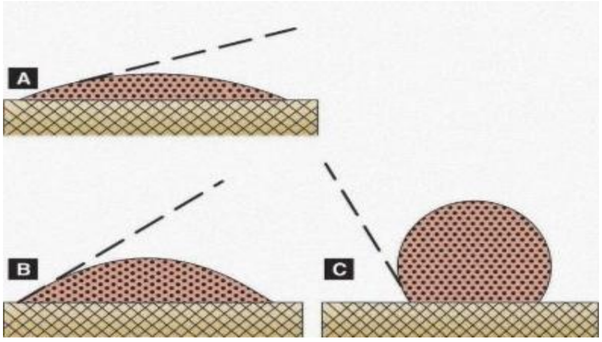


**Fig. 1:** Degree of contact angle influnces the wetting of surface; (A) when contact angle is small, wettability of the adhesive is better; (B) and (C) when contact angle is large, liquid does not wet the surface completely. [Modified by **(Sikri, 2017)**].

### 1.2.2 Possible mechanisms of adhesion

**Ritter *et al.* (2019)** mentioned mechanisms of bonding resin-based materials to tooth structure are four, as follows:

I. Mechanical penetration of resin and formation of resin tags within the tooth surface.

II. Adsorption chemical bonding to the inorganic component (hydroxyapatite) or organic components (mainly type I collagen) of tooth structure.

III. Diffusion precipitation of substances on the tooth surfaces to which resin monomers can bond mechanically or chemically.

IV. A combination of the previous three mechanisms.

## 1.3 Basic of bonding

Successful bonding requires careful attention to three essential components: the tooth surface and its preparation, the design of the bracket base, and the bonding agent **(KH *et al.*, 2014).**

### 1.3.1 Ideal requirements of orthodontic adhesives by (Singh, 2007)

1. To have suitable flow properties.
2. Penetration without undue slumping or bracket drift; this rheological characteristic is often expressed as thixotropy.
3. Provide high bond strength to enamel and dentin.
4. Provide an immediate and durable bond.
5. Prevent of the ingress of bacteria.
6. Be safe to use, biocompatible.
7. Be simple to use.
8. To minimize setting shrinkage, their overall water absorbing tendency should be minimum.
9. Aesthetic.
10. Color stability.

### 1.3.2 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. It is usually reported in units of Mega pascal **(Versluis *et al.*, 1997).**

Bond strength tests are almost always classified as shear or tensile bond strength tests. Tensile strength is the amount of force required to stretch a material in a single impact before it breaks, whereas shear strength is the maximum shear stress that a material can withstand in a single impact before failure **(Albers, 2002)**. It has been suggested that bond strengths between 8 and 9 MPa are sufficient to withstand normal orthodontic forces **(Sunna and Rock,**

**1998).** The maximum bond strength should be less than the breaking strength of the enamel, which is about 14 MPa **(Retief, 1974; Bowen and Rodriguez, 1962).** Regardless of the adhesive system, SBS were significantly higher at 24 H. after bracket bonding procedure than after 15 min **(Vinagre *et al.*, 2014).**

However, the maximum bond strength should be inferior to the tensile strength of enamel, which ranges between 11 and 25 MPa, depending on the prismatic orientation **(Carvalhoa *et al.*, 2000).**

## 1.4 The steps of bonding of orthodontic attachments:

### 1.4.1 Cleaning

Before bonding brackets, it is essential to remove the organic pellicle that normally covers all teeth **(Aboush *et al.*, 1991).** Investigators have found that these remnants might interfere with the etching process, resulting in subsequent lower resin adhesion **(Kaneko *et al.*, 1986; Clasen *et al.*, 1997).**

This is accomplished by cleaning the enamel surface using a mix of pumice and water, or prophylaxis paste, with a rubber cup or a polishing brush mounted on a low-speed rotary instrument as shown in **(Fig. 2),** it has been observed that prophylaxis alone can double the bond Strength **(Bishara *et al.*, 2005)**. The tooth is subsequently rinsed with water to remove any pumice debris, and thoroughly dried with a stream of oil-free air, during this procedure, cheek and lip retractors, saliva ejectors and cotton or gauze rolls should be used **(Melsen, 2012).**

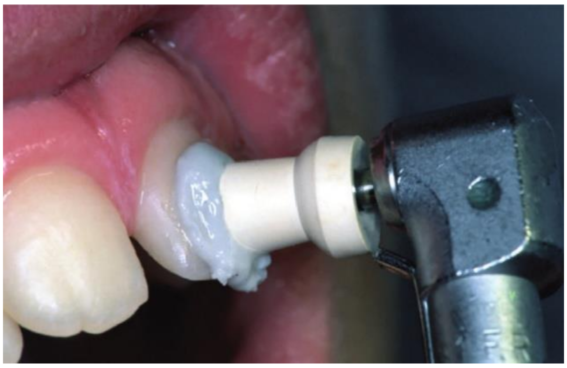

**Fig. 2:** Initial prophylaxis with pumice [modified by **(Melsen, 2012)**].

### 1.4.2 Enamel conditioning:

Unprepared enamel surface is hydrophobic, with limited wettability. Thus, bonding to untouched enamel poses a challenge. For successful bonding, enamel surface conditioning or pretreatment is necessary. Typically, this is performed by etching the surface using a variety of acids **(Nanda and Kapila, 2010).**

#### 1.4.2.1 Phosphoric acid etching

**Buonocore (1955)** envisioned the use of acids to etch enamel for sealing pits and fissures. And he suggested the use of 85% phosphoric acid for 2 minutes on the enamel surface to etch it **(Ritter *et al.*, 2019). Silverstone *et al.* (1975)** demonstrated that the optimum concentration of phosphoric acid should be 30% to 40% for 60 seconds to achieve good retention in enamel. Presently a 37% concentration of phosphoric acid is preferred (**Raghu and Srinivasan, 2013; Garg and Amit Garg, 2013).** And time reduced to 15 seconds as studies using scanning electron microscopy shows it produces the same roughness was achieved previously within 60 seconds **(Raghu and Srinivasan, 2007; Ritter *et al.*, 2019).** Over etching is thought to occur beyond 60 seconds, resulting in compromised tooth structure and bond strength **(Wang and Lu, 1991)**. Etching time may be increased in the case of primary teeth and in teeth with fluorosis **(Raghu and Srinivasan, 2013; Shen *et al.*, 2022).** Deciduous teeth require

120seconds of etching to achieve the same etching pattern as permanent teeth because of a lower mineral content and higher internal pore volume **(Angmar *et al.*, 1963; Silverstone *et al.*, 1975).** Etching removes 10 µm of surface enamel and creates a micro porous layer which is 5 to 50 µm deep **(Lopes *et al.*, 2007).**

The surface should be washed for at least 20 seconds **(Shen *et al.*, 2022).** The prolonged water lavage is necessary to remove contaminant residue, consisting mainly of soluble calcium salts, from the treated enamel surface before bonding. The surface of the etched enamel should be very thoroughly dried. This imperative that the air directed across the surface of the tooth be free of oil or moisture, as they act as contaminant and reduce the bond strength with resin. Although chemical drying agents may be used, warm air drying is preferred **(Singh, 2007).**

**Raghu and Srinivasan (2013)** and **Garg and Amit Garg (2013)** found microscopically three types of etch patterns have been described, type I dissolution of the prism core leaving the prism peripheries intact as shown in **(Fig. 3)**, Type II dissolution of the prism peripheries leaving the prism core intact, type III no prism structures are evident.

Recently type IV and type V patterns have been added. Type IV pattern displayed only a random distribution of depressions with no preferential distraction of either cores or peripheries. These pitted areas occasionally occurred in little patches over the enamel surface. This type of etching pattern is commonly seen in cervical areas and rarely on occlusal. Similarly, to type IV pattern, type V etching shows no evidence of prism outline. The regions of enamel are flat and smooth and lack micro irregularities for penetration of resin. Such type is seen in high fluoride **(Sikri, 2017).**

For increasing the simplicity of etching procedure, acid gels are preferable to acid solutions: gels provide better control of the acid and restriction of the working field to avoid insulting the gingival margin and initiating bleeding, although there is no apparent difference in the degree of surface irregularities **(Brannstrom *et al.*, 1982).**

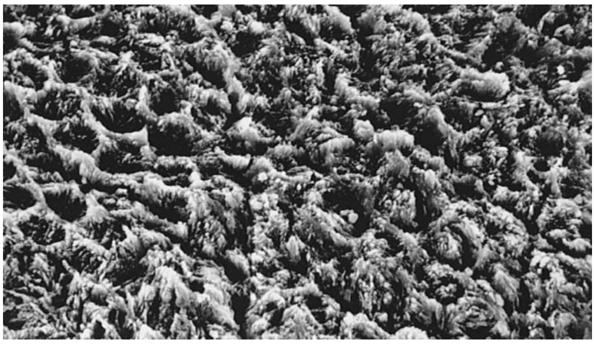

**Fig. 3:** Surface of etched enamel in which the centers of enamel rods have been preferentially dissolved by the phosphoric acid. (Courtesy of K-J. Söderholm). **[**Modified by **(Anusavice *et al.*, 2003)**]**.**

### 1.4.2.2 Maleic acid

Research studies showed that 10% maleic acid potentially decreased the Mineral loss and produced similar bond strengths to 37% orthophosphoric acid **(Olsen *et al.*, 1997).** However, its use remained unpopular **(Nanda and Kapila, 2010).**

### 1.4.3 Sealing

A liquid resin applied with a small foam pellet or brush with a single gingivo-incisal stroke on each etched tooth. The resin is able to penetrate into the irregularities created in the etched enamel surface, allowing the bonding material to mechanically interlock with the tooth surface **(Fig. 4).** Scanning electron microscopy studies show that the non-rinse conditioner produces a more conservative bonding pattern than that with conventional phosphoric acid **(Melsen, 2012).**

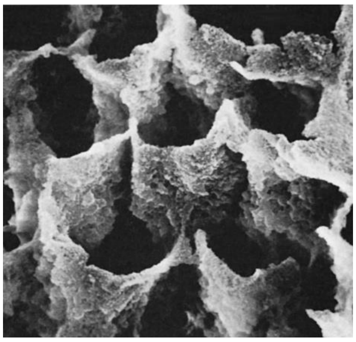

**Fig. 4:** Scanning electron microscopy image of tags formed by the penetration of resin into etched areas of enamel. The resin was applied to the etched enamel, and the enamel was then dissolved by acid to reveal the tags. (×5000.) (Courtesy of K-J. Söderholm.) [Modified by **(Anusavice *et al.*, 2003)**].

### 1.4.4 Primers and coupling agents:

These substances seek to make the surface of the substrate more amenable to accepting a bond. Primers are hydrophilic monomers, carried in a solvent **(Singh, 2007).** According to **Van Landuyt *et al.* (2007),** some of frequently used monomers are: HEMA, Di-methacrylates, 10-MDP, 4-META, Di-HEMA-phosphate and HEMA-phosphate. There is generation improvement in development of bonding primers, as shown in **Table 1.**

**Table 1: Generation improvement in the development of bonding primers in restorative dentistry (Birnie and Harradine, 2012).**

| Generation | Characteristic | Surface treatment | Properties |
|---|---|---|---|
| 1 | NPG-GMA | Etched enamel | Dry bonding |
| 2 | Bis-GMA\HEMA | Etched enamel | Dry bonding |
| 3 | 4META\BPDM | Etched enamel | Dry bonding |
| 4 | Hydrophilic primer | Etched enamel | Wet bonding |
| 5 | 1-bottle system | Etched enamel | Wet bonding |

The most commonly used solvents in primers are water, ethanol and acetone. Addition of hydrophilic monomers on one hand, and a solvent on the other hand dramatically improves the wetting behavior of the primer, as shown in **Table 2 (Van Landuyt *et al.*, 2007).**

**Table 2: Comparison between different types of solvent (Singh, 2007).**

| Solvent | Advantage | Disadvantage |
|---|---|---|
| Acetone | Dries quickly | Can evaporate from container, multiple applications require sensitive to wetness dentin |
| Water | Slow evaporation, not sensitive to wetness of dentin | Long drying time, water interferes with adhesive if not removed |
| Ethanol\water | Less sensitive to wetness of dentin, evaporate slowly | Long drying time |
| Solvent free | Single coat, no drying | High film thickness |

## 1.5 Adhesive

The adhesive is essentially an unfilled or lightly filled resin, similar in composition to the resin in composites except that hydrophilic molecules have been added **(Singh, 2007).**

## 1.6 Scientific Classification of Modern Adhesives

As a major component of orthodontic therapy, researchers tended to optimize the bonding of brackets by improving the quality of the adhesive systems used throughout the evolution of different generations of products, as shown in **(Fig. 5) (Van Meerbeek *et al.*, 2003).**

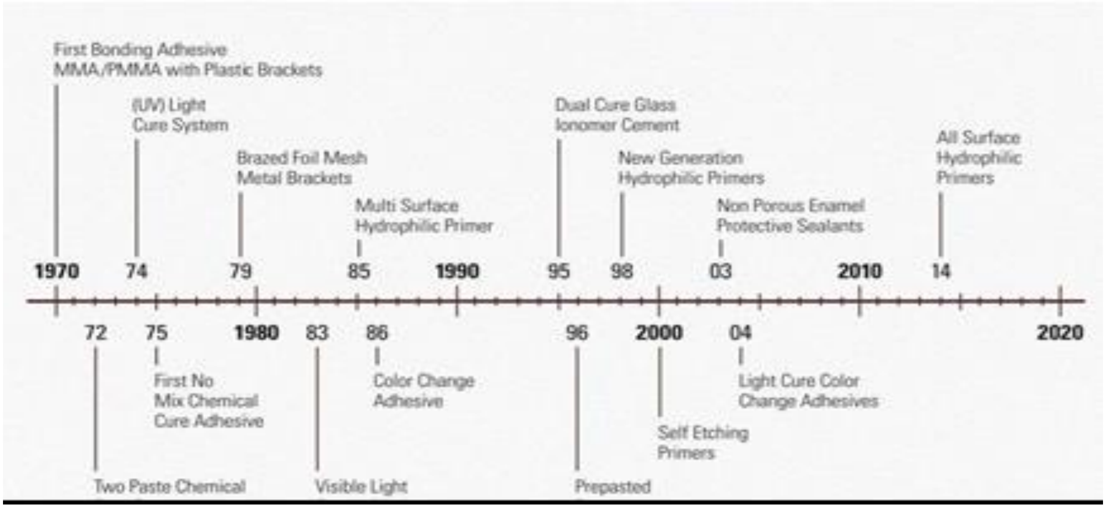


**Fig. 5:** Orthodontic bonding timeline. [Modified by **(Gange, 2015)**].

### 1.6.1 Based on generations

- First generation bonding agents.
- Second generation bonding agents.
- Third generation bonding agents.
- Fourth generation bonding agents.
- Fifth generation bonding agents.
- Sixth generation bonding agents.
- Seventh generation bonding agents.
- Eighth generation bonding agents.

### 1.6.2 Based on number of steps

- Three step
- Two step
- Single step

### 1.6.1.1 First generation bonding agents

Developed in 1960s **(Garg and Amit Garg, 2013).** The initial trial was using a surface active Co-monomer, N-phenyl-glycine-glycidyl methacrylate (Npg-GMA), which acted as a primer and adhesion promoter between enamel and resin materials by chelating with surface calcium (**Raghu and Srinivasan, 2013).**

Since calcium ions of the tooth substance are a mediator in the bond formation, agents of this type are expected to form stronger bonds to enamel than to dentin. Studies with this system have not shown good retentive values. A commercially introduced system, Cervident (Ss White) has shown bond strengths to dentin of only 2.8 MPa and no improvement in marginal leakage when compared to conventional unfilled bonding agent **(Sikri, 2017)**.

### 1.6.1.2 Second generation bonding agents

Most of the second generation bonding agents were phenyl phosphorous, phosphorous/chloro phosphorous esters of unfilled resins such as Bis-GMA or HEMA. The bonding mechanism involved improved wetting of the surface and ionic interaction between the phosphate group and calcium of the tooth. Clearfil (kuraray, Japan) was the first agent introduced in this series. It composed of an ethyl alcohol solution containing tertiary amine as the activator. The catalyst liquid was Bis-GMA monomer containing a phenyl phosphate ester, benzoyl peroxide and methyl methacrylate. The interfacial bond was established through attraction between the negative charges of the oxygen on the phosphorous group and the positively charged calcium ions in the dentin surface. scotch bond, (halo phosphorous ester of Bis-GMA) is formed by reaction between Bis-GMA and phosphorous oxychloride (pocl3). Bonding to tooth calcium occurs through chlorines having a partial negative charge **(Sikri, 2017)**.

Mean shear bond strengths to dentin have been reported to be 2-7 MPa for the second generation bonding agents. Bond strengths in this range are considered to be quite week **(Sikri, 2017).**

### 1.6.1.3 Third generation bonding agents

In the late 1980s, the third generation DBa, two component primer and adhesive systems were introduced. conditioning and priming before application of bonding agent **(Garg and Amit Garg, 2013).** Third generation used milder acids like 2% nitric acid, 2.5% maleic acid with HEMA, 10% citric acid with 3% ferric chloride, 10% phosphoric acid **(Raghu and Srinivasan, 2013).**

**Garg and Amit Garg (2013)** described many advantages of third generation bonding agents over first and second generation bonding agents:

- Higher bond strengths (8–15 MPa).
- reduced micro-leakage.
- form a strong bond to both sclerotic and moist dentin.
- reduced need for a retentive tooth preparation.
- can be used for porcelain and composite repairs.
- Erosion, abrasion and abfraction lesions can be treated with minimal tooth preparation.

They also mention drawbacks of third generation bonding agents:

- Decrease in bond strength with time.
- Increase in micro-leakage with time.

### 1.6.1.4 Fourth generation bonding agents

In the early ‘90s, 4th generation bonding agents transformed dentistry. It was developed by Fusayama and Nakabayashi in Japan in the 1980s. Described as etch [Phosphoric Acid]+primer [NTG-GMA, N-Tolyglycine–Glycidyl Methacrylate] dissolved in acetone or ethanol+bond [Bis-GMA/TEGDMA] **(Singh, 2007)**.

**Singh (2007)** demonstrate several Advantages:

• It has high bond strength to dentin in the range of 17-25 MPa.

•decreased postoperative sensitivity in posterior occlusal restorations encouraged many dentists to begin the switch from amalgam to direct posterior composite fillings.

• with this ‘generation” the concept of hybridization at the interface of the dentin and the composite began.

• moist dentin bonding, earlier, air drying was recommended for the dentin but now the adhesives are designed to work best on well hydrated or moist dentin.

### 1.6.1.5 Fifth generation bonding agents

Developed in mid 1990s (**Garg and Amit Garg, 2013).** One component system manufacturer introduced systems that combine the primer and adhesive agents. These materials consist of hydrophilic and hydrophobic resins simultaneously dissolved in solvents like alcohol or acetone, displacing water and achieving an intimate contact to dentinal structures. This system may be described as, E[phosphoric acid]+PB [PENTA, methacrylated phosphonates] **(Singh, 2007)**. According to **Raghu and Srinivasan (2013),** these agents are in inferior to fourth generation in their bond strength.

**Singh (2007)** decided several advantages:

• These materials adhere well to enamel, dentin, ceramics, and metal.

• A single component, single bottle characterizes them. There is no mixing, and thus, less possibility for error.

• Bond strengths to dentin are in the range of 20-25 MPa.

• These bonding agents easy to use and predictable.

• Postoperative sensitivity has been reduced appreciably.

### 1.6.1.6 Sixth generation bonding agents:

Developed in early 2000s **(Garg and Amit Garg, 2013).** No etch, no rinse, no cure technology, an effort to eliminate etching or to include it chemically in one of the other steps, this system can be described as EPB [Methacrylated Phosphates] **(Singh, 2007).** According to **Raghu and Srinivasan (2013)** this system manufactured into 2 types**:**

- Self-etching primers: etchant and primer are in one bottle and applied firstly to the tooth surface which then followed by the application of the adhesive agent. Subsequently, the bonding agent is light cured. **Garg and Amit Garg (2013)**, mentioned water is the solvent in these systems.
- Self-etching adhesive: the primer, etchant and adhesive are all in one package but require mixing before application on tooth surface.

**Garg and Amit Garg (2013),** evaluated bond strength lower than fourth and fifth generation bonding agent and reduced postoperative sensitivity. According to **Singh (2007)** the bond strength to the dentin is around 18-25 MPa, while it is bond to the unetched, unprepared enamel. The pH values did not influence the shear bond strength significantly in the between 6$^{th}$ and 7$^{th}$ adhesive systems **(Jamadar *et al.*, 2020).**

### 1.6.1.7 Seventh generation bonding agents

They are true "all in one adhesive", also, in the form of self-etch adhesives, a simpler procedure, a non rinse approach, was introduced. By combining hydrophilic and hydrophobic acid functional monomers, organic solvents, and water into a single-bottle solution, this method incorporates all the etching steps, priming, and bonding into one step, making it more user-friendly and less technique sensitive **(Talan *et al.*, 2020).** Bonding agents also have disinfecting and desensitizing Properties **(Garg and Amit Garg, 2013)**.

Drawbacks:**(Raghu and Srinivasan, 2013)**

- More prone to phase separation as they contain both Hydrophilic and Hydrophobic components in one bottle.
- Act as semipermeable membranes as they allow water to move in and out of the adhesive layer.
- Provide lower bond strength than the fourth and fifth generation adhesives.

### 1.6.1.8 Eight generation bonding agents

A modified version of seventh generation bonding agent is introduced by Voco America as Futurabond DC. This is one-step dual-cured, non-filled, self-etch adhesive available in single use blister packs **(Sikri, 2017)**.

Advantages: **(Sikri, 2017)**

• Can be used with light cure and dual cure and self-Cure composites.

• Provides high bonding strength. **Khudhur *et al.* (2021),** Concluded that the eighth-generation bonding agent showed higher mean bond strength to dentin than the seventh.

• Moisture tolerant.

• Contains fluorides.

• Nano-fillers help in better cross linking of the bonding Resin components.

**Ganesh (2020),** concludes this generation gives highly long-lasting esthetic, wear resistant, and it is ideal for hypersensitive tooth.

### 1.6.2.1 Three steps etch and rinse adhesive

Etch and rinse adhesives have been familiarized since the early 1990's, 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 considered the most durable and predictable method of bonding esthetic materials to tooth structure **(Van Meerbeek *et al.*, 2003)** (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 **(Ritter *et al.*, 2019).** Their mechanism is principally based on the shared effect of hybridization and formation of resin tag **(Tyas and Burrow, 2004).**

### 1.6.2.2 Two steps:

### A) etch and rinse adhesive system

The research has focused on the simplification of the bonding procedure by reducing number of steps into two steps etch and rinse adhesive system. They are sometimes called "one-bottle" system because they combine the primer and bonding agent into a single solution and a separate etching up still is required **(Landuyt and Lamberchts, 2005).**

This etch and rinse strategy is the most effective to achieve efficient and stable bonding to enamel. Etching, usually with a 30% to

40% phosphoric gel that is rinsed away, promotes the dissolution of enamel rods, creating porosities that are filled by a bonding resin through capillary action, and then followed by resin polymerization. And the application of two coats is recommended **(Shen *et al.*, 2022).**

**B) Self-etch adhesives**

Self-etching adhesive systems differ from etch-and-rinse adhesives in several aspects, such as the initial pH, type of acidic monomer, number of bottles and steps, concentration of water and solvents, and the hydrophilicity of the bonding Layer **(Gomes-Silva *et al.*, 2008; Moura *et al.*, 2009).**

According to **Migliau *et al.* (2017),** that SE classified into two or one step systems depending on the number of procedures required for.

**1- 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 enamel and dentine **(Perdigao and Lopes, 1999).** 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).** later on this agreed by the conclusion of **Kulkarni and Mishra (2016),** that self-etching adhesives were not negatively affected by the presence of water on the enamel surface because of that, **Masarwa *et al.* (2016)** conclude SEPs are less technique sensitive than are etch and rinse adhesives. On the other hand, the hydrophilic feature of these monomers increases the water sorption of this material, which may render the adhesive interface weaker and lead to bracket debonding **(Reis *et al.*, 2007).**

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)**. **Patil *et al.* (2013)** his conclusion agree with several studies made previously by **Hannig *et al.* (2002), Perdigão and Geraldeli (2003)** and **Brackett *et al.* (2006),** 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**.** Numerous in vitro studies have shown that this system provides shear bond strengths similar to the values achieved with etch and rinse systems. **(Perdigão and Geraldeli, 2003; Miguez *et al.*, 2003; Kiss Moura *et al.*, 2006). Hu *et al.* (2013)** found low-quality evidence that was insufficient to conclude whether or not there is a difference in bond failure rate between SEPs and conventional etching systems when bonding fixed orthodontic appliances over a 5 to 37 month follow-up. The self-etching primer can successfully be used for bracket bonding. The thermo-cycling protocol did not affect shear bond strengths **(Vinagre *et al.*, 2014).**

**Fleming *et al.* (2012)** conclude there is strong evidence that a self-etch primer is likely to result in a modest time savings (8 minutes for full bonding) compared with acid etch.

**2- One-step self-etch adhesives**

The self-etching approach has been proposed in an effort to simplify the enamel and dentin bonding systems. These materials combine tooth surface etching and priming steps into one single procedure. The elimination of separate etching and rinsing steps simplified the bonding technique and has been responsible for the increased popularity of These systems in daily practice containing ethanol as a solvent or co-solvent showed higher SBS compared with the other self-etching bonding agents. The bond strength values of these adhesives to dentin are significantly higher than those to enamel **(Ageel and Alqahtani, 2019)**.

Mechanism of action in contrast to conventional adhesive systems that contain an intermediate light-cured, low-viscosity bonding resin to join the composite restorative material to the primed dentin–enamel substrate, these one step SEAs contain uncured ionic monomers that contact the composite restorative material directly **(Ritter *et al.*, 2019)**. One-step self-etch adhesives are highly hydrophilic; therefore, they attract water and may increase the potential for degradation **(Ito *et al.*, 2005; Nishitani *et al.*, 2006; Ritter *et al.*, 2019) .**

## 1.7 Universal adhesive systems

One of the most recent novelties in adhesive dentistry was the introduction of universal adhesives that have been used since 2011 in clinical practice. These new products are known as “multi-mode″ or ″multi-purpose″ adhesives because they may be used as self-etch (SE), etch-and-rinse(ER) adhesives, or as SE adhesives on dentin and ER adhesives on enamel [a technique commonly referred to as “selective enamel etching”] **(Migliau *et al.*, 2017).** These adhesives have ability to bond methacrylate-based restorative, cement, and sealant material to dentine, enamel, glass ionomer, and several in direct restorative substrate, including metals, alumina, zirconia and other **(Hilton *et al.*, 2013).**

Mechanism of action described: **(Ritter *et al.*, 2019)**

- The major difference between traditional one-step SEAs and universal adhesives is that most universal adhesives contain 10-MDP (and/or other monomers), which is capable of bonding Ionically to hydroxyapatite through Nano layering.
- The 10-MDP molecule forms stable calcium-phosphate salts without causing strong decalcification. The chemical bonding formed by 10-MDP is more stable in water than that of other monomers used in the composition of SEAs, such as 4-META and Phenyl-P.
- The active application (rubbing) of 10-MDP-containing adhesives results in more intense Nano layering than passive application. Make higher concentration of 10MDP in intimate contact with hydroxyapatite crystals. And this makes improvements on the bonding strength.
- Reduced Nano layered formed in enamel because parallel orientation of hydroxyapatite crystals makes the enamel less receptive to chemical interaction with 10MDP.

**Hirokane *et al.* (2020)** find Phosphoric acid pre-etching and double-layer application of universal adhesives resulted in increased enamel bond strength in the early phase of specimen bonding. Later on **Fraga *et al.* (2021)** conclude conventional 'etch and rinse' and 'self-etch' adhesives had the highest shear bond strength, but they were associated with more enamel damage compared to universal adhesives. The application of universal adhesives with the time recommended by the manufacturer ensures satisfactory bond strength and enamel integrity.

# Chapter two: Discussion

We discussed in the previous chapter the bonding mechanism, adhesive systems and mention its bonding strength, we found that all of them reach the optimal strength need for placement of orthodontic brackets. According to **Nystrom *et al.* (1998),** a commonly encountered problem during orthodontic treatment is bond failure. This is because bonding procedures require multiple clinical steps, and that success with these adhesive systems can depend on technique sensitive and material related factors. Neither vivo or in vitro study the longevity of the strength of SEP, E&R, universal and mixture in short or long term used in orthodontic, in vitro testing does not closely simulate the oral environment, which includes the possibility of contamination with saliva, the stresses placed on the teeth during mastication and occlusion, decalcification, the degradation of the adhesive when exposed to the saliva, the temperature variations introduced by food or drinks, as well as the skill of the clinician.

The storage and shelf life is important because it could affect the material physical and chemical properties, storage temperature and relative humidity several studies from conservative department conclude improvement in quality of resin penetration to dentine bond in E&R if stored at room temperature than keeping it in the refrigerator, authors explain that due increase in the viscosity which leads less wettability. We can relate that to orthodontic as we mention in the beginning of this project less wettability means less penetration, less adhesion results bond failure. Future more, etching with phosphoric acid as separated process suitable pattern for penetration of resin tag than others also its acidity don't affect the residual component of the bond during storage. After application to tooth surface the bond strength increase within time due polymerization of deeper resin tag.

In the Self etch system that storage effect still contraverse because it contains HEMA which is Hydrophilic could absorb water in cured an un cured [methacrylate monomer undergo hydrolysis under acidic aqueous **(Tay and Pashley, 2001)**] starts from the moment of manufacturing, As we know when the temperature increase the chemical interaction occurs in faster rate, that's why Manufacturers recommend to keep product in the refrigerator to minimize this chemical interaction, some authors find there's no difference in the storage condition but others disagree. this maybe due different chemical formula of the tested product some have all components in 1 bottle others 2 bottle system, also some had water as solvent others have acetone or ethanol. SE after curing process will have a continuous polymerization which makes the bond strength higher within days after that the continues esterification will degrade the bond it self. SE they don't affect by the humidity because it's a hydrophilic material.

Universal bond despite their convenient clinical use, the combination of a variety of components with different chemical natures into a single container makes the stability of the bonding agent controversial. The HEMA free universal bond should be stable and less affected by hydrolysis universal bond with HEMA stability still unknown but depending on the chemical formula if esterification occur the polymer subdivision into smaller monomer.

# Chapter three: Conclusion and Suggestions

1- Conventional Etch and rinse 5th generation bonding system have most stable evidence than others. (Have longer bonding strength).
2- Phosphoric acid treatment gives well defined pattern, for deep penetration by the resin and much more effective than others.
3- Etch and rinse and self-etch reach the optimal bonding strength.
4- The longevity of Universal bond strength still needs more clinical trials.
5- The bond strength of universal bond in SE technique couldn't with stand orthodontic force because it has less chemical interaction with hydroxyapatite crystals in parallel enamel prism.
6- Application of 2 layers of Universal bond (HEMA-Free) with rubbing motion after treating tooth surface with phosphoric acid which is helpful in making irregularities that could increase the ability of bond chemically with hydroxyapatite crystals.
7- Best condition for application any bond is clean enamel surface.
8- After applying bond to enamel surface make a very fast air blowing to allow thin layer thickness because thicker or un even thickness leads to fracture later.
9- Follow manufacture instructions.
10- Avoid leaving cap of any adhesive open more than 1 minute to prevent evaporation of its components.
11- Keep clinic temperature between 25-30°C.
12- Reaching optimal bond strength doesn't mean this material will withstand orthodontic force during mastication and long treatment duration.
13- Longevity of the bonding strength not relevant to higher bond strength.

14- Longer Shelf life of the material doesn't mean it have stable properties till the expiration date it depends on multiple factor.

15- There is highly need for more Vitro & Vivo studies covers newly produced adhesives like universal bond and eight generation bond and how could be effective to use for long term orthodontic treatment

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