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Controversies in Adhesive Dentistry – an Overview

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1. Abstract

Background/Aims:

The aim of this study is to investigate and assess the different adhesive systems and their chemical and mechanical properties on the tooth surface in order to preserve as much tooth structure as possible. Adhesive techniques and their efficiency were analyzed.

Objectives:

1. To evaluate the technical necessity of adhesive systems.
2. To evaluate the efficiency content of adhesive components.
3. To evaluate the influence of Self-Etch and Etch & Bond systems on long – term duration.
4. To evaluate the nature of the adhesive resin bond to dental materials.

Research Methods:

An Overview was implemented based on scientific articles including this topic. The databases used were Scientific & Medical ART Imagebase, CINHALL, Dentistry & Oral Sciences Source Alumni Edition using Pubmed and Elsevier. This Overview contains scientific data written in English language.

Results:

Lower results were found for self -etching adhesives than for Etch & Rinse adhesives. The marginal strength is significantly lower in the enamel of composite fillings after thermal and mechanical stress than for the Etch & Rinse systems. Dentin bond strengths varies according to the acid exposure treatment.

With longer etching times, the risk of exposed collagen fibers in deeper layers increases, where it may not be completely penetrated by the components of the bonding system.

The exposed collagen fibrils may consequently suffer hydrolytic degradation by matrix metalloproteinase.

The pH value of self – etching adhesives determines the effectiveness of penetration and dissolution of the smear layer and thus the quality of the bond to the tooth structure.

Conclusion:

This Overview discusses the precise bonding techniques needed for long-term clinical effectiveness with different tooth shapes and dental materials.

A modern adhesive system consists of a conditioner (acids, complexing agents), a primer (hydrophilic monomer in a solvent) and an adhesive (various monomers).

After applying the total-etch technique, a primer-adhesive mixture is applied. The one-bottle systems are intended to make work easier, save time and reduce technique sensitivity. Mixing up different bottles or mixing ratios is impossible. However, these advantages are offset by more difficult moisture management. Water plays the central role here, as it is required on the one hand to set up the hydrophilic collagen fiber mesh of the demineralized dentin for penetration, but on the other hand it impairs bonding to the hydrophobic composite. Overdrying to reveal the etching pattern in the enamel requires "re-wetting". In contrast, Type I adhesives supply sufficient water to the dentin dried after phosphoric acid etching via the hydrophilic primer, whereas Type III and IV Self-Etch systems avoid this problem by simultaneous etching and priming.

The solvents ethanol and acetone remove the excess water after infiltration of the adhesive into the dentin. However, in the context of wet bonding, these organic solvents must be prevented from drying out the dentin, as a collapse of the collagen mesh prevents the complete formation of the hybrid layer and leads to nanoleakage and postoperative hypersensitivity. In general, multi-bottle systems are sufficiently hydrated, but single-bottle Total-Etch or Self-Etch systems are not or not sufficiently hydrated for adequate infiltration.

2. Keywords:

Adhesive systems, Etch&Rinse-system, Self-Etch system, Enamel, Dentin

3. Introduction

The development of adhesive dentistry has revolutionized the dental field. The minimally invasive filling technique only became possible due to adhesive bonding to the tooth surface.

In the past, teeth were mainly restored with amalgam or simple cements and - if it was affordable for the patient - with cast gold fillings. The main aim was to maintain the functionality of the dentition.

The biocompatibility of restorative materials is of great importance, which led to amalgams losing their status in modern dentistry. Tooth-colored restorative materials can be divided into cements (glass ionomers) and resin-based materials (composites). The restoring tooth structure requires pre-treatment before they can be placed. During this pre-treatment, various chemicals are used to create a durable bond between the tooth structure and the composite. This specific step in restorative dentistry is known as the adhesive technique.

Since its introduction almost 60 years ago, the adhesive technique has undergone rapid development. The biggest challenge with dental adhesives is the necessity to form a good bond with two different substrates. A reliable bond with dentin is more difficult and can only be achieved with complex and time-consuming application procedures. Consequently, today's adhesives are often considered technique-sensitive, with risks in clinical application as loosening of the filling or premature degradation of the filling margin.

4. Tooth Structure Enamel

Human tooth enamel consists of 93-98% inorganic material by weight. The main component of which are calcium-phosphate crystals in the form of hydroxyapatite ¹. Approximately 100 of these enamel crystals form so-called enamel prisms, which, with an average diameter of 5.5 μm ². They extend from the enamel-dentin junction through the entire enamel mantle almost to the enamel surface ². Here, the enamel sheath usually ends with a 20-80 μm thick layer of enamel consisting crystals ¹, which can also be found in deciduous teeth, fissures and in the cervical region ³. The enamel prisms are surrounded by an interprismatic substance also formed from enamel crystals substance, in which the crystals are unstructured ³.

Another histological phenomenon are the Retzius striae that run approximately parallel to the tooth surface, which represent the growth lines of the enamel ³. They are still macroscopically visible in children and adolescents as perikymatia and imbrication lines and disappear with age due to attrition processes ^{3,2}.

According to earlier reports^{18,21}, typical rod crystals in adult enamel are more than 10 μm long and approximately 50 nm broad ⁵. Each rod has elongated crystals that are arranged parallel to one another ⁵. Every rod has a corresponding interrod, which is made up of crystals oriented at around a 60° angle to the rod axis ⁶. The direction of crystal elongation gradually changes from the rod to the interrod ⁷.

Every rod has an organic matrix sheath surrounding it, and the crystals inside the rods are abutting one another with an irregular organic meshwork in between ⁵. It is 10 times more resistant to crack growth than bone because of rods that run from the dentin-enamel junction (DEJ) to the surface of enamel. These rods' trajectories undulate in the inner enamel layer, creating Hunter-Schreger bands or a decussation pattern that has previously been imaged using electron microscopy, x-ray tomography, or x-ray fluorescence ¹⁰.

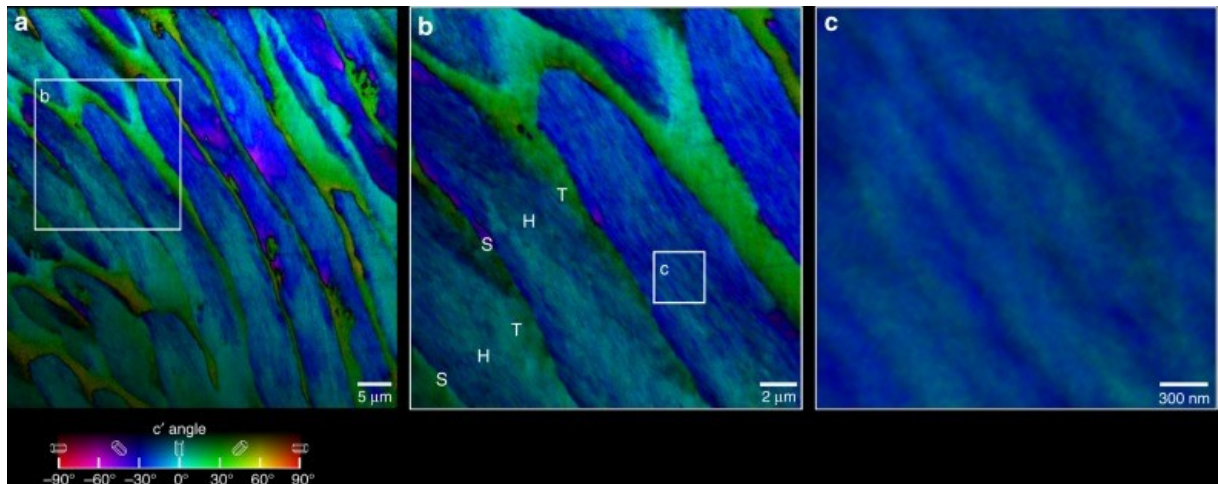


Fig.1

The inner enamel's concealed crystal orientation structure is revealed using PIC maps. a polished cross-section of human enamel at low magnification. Observing the $\sim 5 \mu\text{m}$ broad rods that are blue, indicating that a large number of crystal c-axes are oriented along the rod axis. Nonetheless, a large number of additional crystals (cyan and magenta) are orientated $\pm 30^\circ$ off the rod axis. The homogeneously green tint, with only a few orange pixels, indicates that the c-axes of interrod crystals are highly co-oriented.

In figure b, you can see that there is an organic sheath (S) separating the rod head (H) from its interrod tail (T), which changes in crystallographic orientations gradually, from the interrod to the next rod head, which transitions abruptly. c Zoomed-in area in b, where individual crystals inside the rod are parallel to one another but do not have co-oriented c-axes; as a result, one or more co-oriented crystals exhibit distinct colors, such as blue encircled by cyan or vice versa. Typically, the crystal width is about 50 nm, the resolution and pixel size in b and c are both 22 nm, and in a, 60 nm.

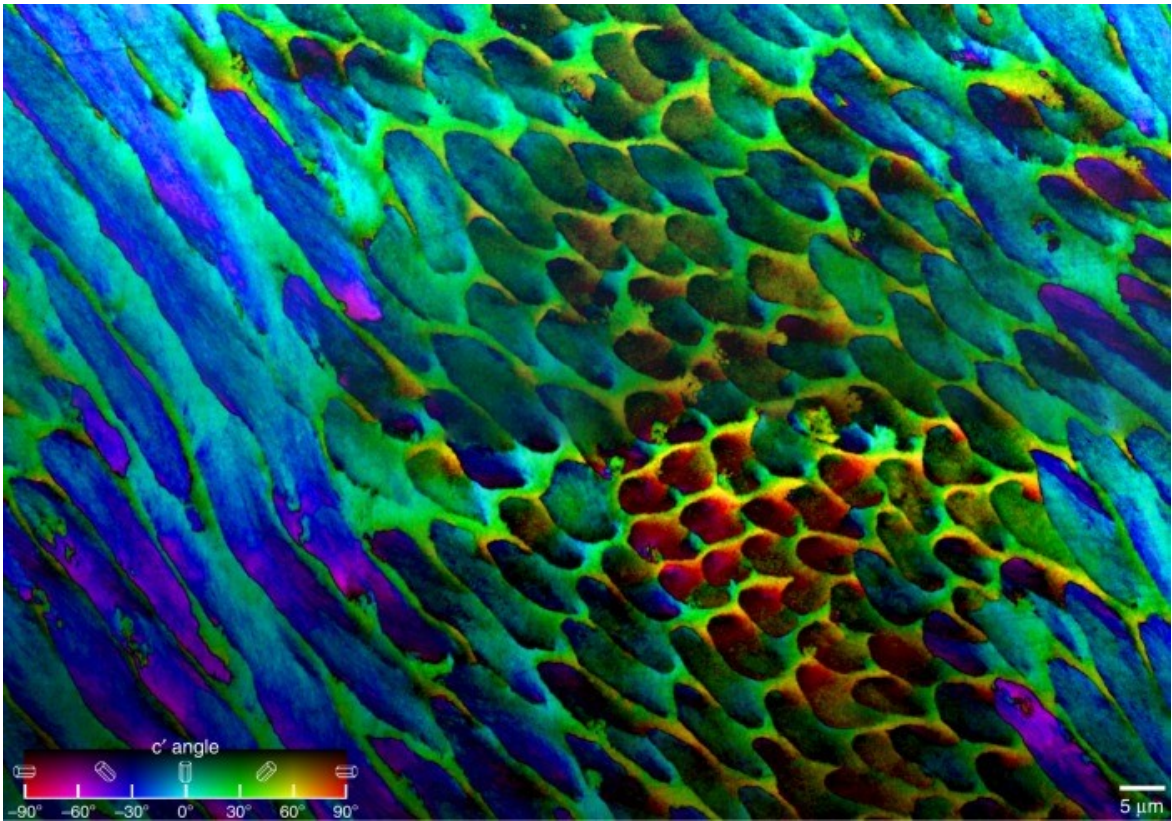


Fig. 2

PIC mapping reveals the hidden crystal orientation structure in a large area of inner enamel.

The map shows Hunter–Schreger bands, or decussation pattern, in inner enamel, with three groups of rods exposed on this polished surface: in longitudinal (left), transverse (right of center), and oblique (center, right) cross-sections.

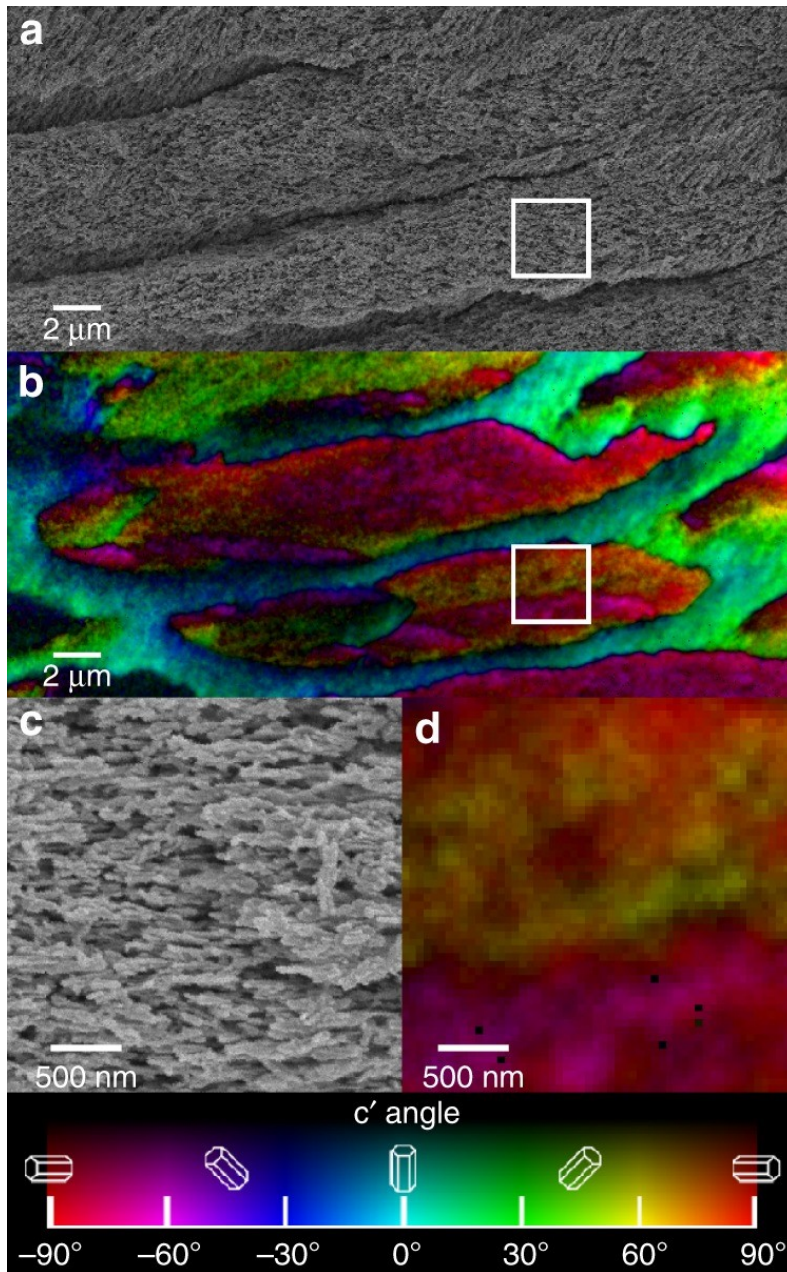


Fig. 3

Following etching, the SEM picture displays two distinct rods that are surrounded by other partial rods and divided by deeper and interrod grooves. b As with all other rods photographed in this work, the PIC map reveals that these two well-defined rods have numerous orientations inside them.

When the same region of enamel is imaged using PIC mapping and SEM, respectively, with the same magnification and orientation before and after etching, it becomes clear that the crystals are significantly misoriented.

4.1. Adhesion to Enamel

Microretention can be used to create an adhesion of materials for bonding dental restorations to the enamel. The adhesion of the composite filling material to the tooth enamel can be achieved by the preparatory step of enamel etching¹⁴. The procedure itself is called the acid etching technique (SAET). Microretention significantly increases the impermeability of a filling. It led to the acceptable use of resins in restorative dentistry.

Etching with 30 to 40% phosphoric acid creates an ideal surface morphology for the micromechanical anchoring of composite¹³. The different acid solubility of the enamel prisms in the center and in the periphery creates a rough structure that allows unfilled and filled adhesives to flow in and leads to an intimate interlocking with the enamel through polymerization. The etching process irreversibly removes approx. 10 µm of the enamel surface and creates a roughness depth of up to 50 µm, the so-called etching pattern. This retentive etching pattern is characterized by a high surface energy and the wettability of the enamel is increased¹³.

Common phosphoric acid concentrations are 35-40% and concentrations below 27% produce less soluble precipitates, so it is not true that lower concentrations are "gentler" and just as effective. A concentration of approx. 37 % with an etching time of 30 s is recommended as ideal for prepared enamel. Shorter etching times are possible on prepared enamel, while non-prepared enamel (fissure sealing, proximal sealing, tooth widening) should be etched for 60 s. The enamel prisms should be etched transversely to their direction; in the case of longitudinal prisms, the adhesive can only flow laterally into the loosened prism areas¹³.

Therefore, the necessity of enamel chamfering in the posterior region has been repeatedly discussed. Although in vitro results prove the superiority of chamfering¹³, there is no clinical evidence for this paradigm, as other parameters are added and chamfering is not necessary if the prisms are already cut

transversely/obliquely during preparation. The actual enamel bond is achieved using functional adhesives such as monomers based on bis-GMA, possibly diluted with TEGDMA.

The adhesion is built up via so-called tags and intercrystalline retention¹⁴. Primers applied separately after phosphoric acid etching should not be actively put into the etched enamel surface for longer than 15 s, as otherwise the adhesion values will deteriorate due to possible destruction of the etching pattern. "Conditioning" is a separate work step that is usually associated with a rinsing process. This applies to all etch-and-rinse adhesive systems, including those used in the past, which did not use phosphoric acid but other acids (e.g. 10% maleic acid, 10% oxalic acid).

Self-etching adhesive systems were originally developed primarily for the gentle pretreatment of dentin - today, however, these adhesive systems are also used for the "etching" of enamel¹⁵. Self-etching adhesive systems contain acidic primers or acidic monomer mixtures (pH <1 to pH 2)¹⁵ that can create an etching pattern in the enamel. The etching patterns produced by these self-etching systems are significantly less pronounced than after phosphoric acid etching. The effectiveness and, above all, the durability of the enamel bond generated by self-etching adhesives are disputed in this research¹⁴.

To date, studies have shown lower results for self-etching adhesives than for Etch & Rinse adhesives. The marginal efficiency in the enamel of composite fillings on molars was significantly lower after thermal and mechanical stress than for the Etch & Rinse systems¹⁵. It was shown that selective conditioning of the enamel with phosphoric acid improves the effectiveness of self-etching systems¹⁶. However, care must be taken to ensure that the enamel is etched selectively¹⁷.

5. Tooth Structure Dentin

Dentin consists of 70% by weight of inorganic material, which is preeminently present as phosphate and calcium in the form of apatite crystals¹⁸. In contrast to enamel, dentin is a living tissue that is formed and maintained by odontoblasts. In contrast to amelogenesis, dentinogenesis is a lifelong process¹⁸. According to the time of dentin formation, a distinction is made between primary dentin, which is formed until the end of root growth, and secondary dentin, which is formed regularly thereafter¹⁹. The latter causes the pulp chamber to shrink with increasing age until the root canals are completely obliterated. If dentin is formed as a defense barrier due to a stimulus such as caries, attrition or erosion, tertiary dentin is formed²⁰. It has an inhomogeneous structure and shows irregularly shaped dentin tubules¹⁹.

The odontoblast bodies are located in the pulp and extend through the entire dentin⁸. The odontoblast processes run in the dentinal tubules, which extend with decreasing diameter from the pulp-dentin to the enamel-dentin junction²¹. Their diameter reaches maximum values of 2.5 μm near the pulp and decreases peripherally to 0.8 μm ^{18,22}. The Tomes fibers are connected to each other via branching thin lateral branches so that intercellular communication can take place between the odontoblasts^{19,21,22}. The number of dentin tubules per unit area varies from up to 90,000 tubules per mm^2 near the pulp to only 15,000 tubules per mm^2 at the enamel-dentin junction^{18,22}. This makes dentin a highly permeable tissue^{24,25}.

The periodontoblastic space around the Tomes' fiber is filled with dentin liquor. This extracellular fluid is secreted by the pulp and consists of water and organic components, including predominantly proteins²⁶. In the dentinal tubules, there is an outward pressure of approximately 30 mmHg^{19,26,27}, which can rise to 40 mmHg in the inflammatory state²⁹. This fact significantly impairs adhesion to the dentin, as the outward flow of fluid does not allow absolute desiccation.

Stimuli such as cold, heat, compressed air, osmotic fluid, or mechanical stimulation cause fluid displacement in the dentinal tubules^{23,25,30}. According to the hydrodynamic theory, even minimal movements of the dentinal fluid stimulate the A- δ fibers by deforming the mechanoreceptors or lead to aspiration of the odontoblast nuclei into the tubules and can thus cause a pain sensation^{31, 32}. Sclerosed or obliterated tubules impair the movements of the dentin liquor and lead to desensitization of the dentin³¹.

During dentinogenesis, phases of irregular mineralization occur, which can be recognized as histological structural features. The Ebner lines correspond to the growth lines of the dentin and run approximately parallel to the enamel-dentin boundary. They represent hypomineralized areas and reflect the resting phases of the odontoblasts²⁰. If general diseases restricting odontoblast secretion occur during dentin development, the so-called Owen lines are found as widened and more hypomineralized growth lines²⁰. The neonatal line results from a longer resting phase of the odontoblasts of approx. 15 days and is found on deciduous teeth and first molars²⁰.

5.1. Adhesion to Dentin

Successful implementation of dentin as an adhesion substrate took longer than with enamel. Two main problems needed to be solved, on the one hand the hydrophilicity of the dentin including the tubules filled with dentin liquor, on the other hand the presence of a smear layer, which develops after

mechanical processing. The first development stages of dentin adhesives were never able to meet the requirements for clinical use, as only a bond to the smear layer was achieved.

Although an infiltration of the smear layer was achieved, the adhesion of the composite was limited by the low adhesion of the smear layer to the dentin. In addition, the smear layer is exposed to hydrolytic degradation processes²⁰. Clinically relevant dentin adhesion could only be achieved with the next stage of dissolving of the smear-layer and the superficial demineralization of dentin³³. The prepared enamel was conventionally conditioned with phosphoric acid, i.e. a selective enamel etching was carried out. Only in the second step a self-etch primer was applied, an acidic monomer solution that was able to dissolve the smear layer. The next stage of development was characterized by the simultaneous etching of both dental resins with phosphoric acid.

This used to be called "total etching", today more correctly "etch & rinse technique". The primer applied after phosphoric acid etching now has the task of preparing the hydrophilic surface with the aid of amphiphilic molecules. The hydrophobic adhesive is then applied for chemical bonding to the composite to be subsequently applied. Thus, two separate components (primer, adhesive) are characteristic of this development stage of adhesive systems. This was followed by the introduction of the so-called "one bottle bonds". The first manifestation of this group was the compomer adhesive Dyract PSA (Dentsply). From a materials science perspective, it is plausible that the combination of properties (penetration capability vs. mechanical stability) represents a compromise, as not both points can be fully developed. Irrespective of this, all Etch & Rinse systems have one problem in common: the collagen network that is exposed after phosphoric acid application must be penetrated by a hydrophilic monomer.

To achieve this, the collagen network should not collapse. In the Etch & Rinse technique, colored 35% to 40% phosphoric acid gels are usually used, as for enamel conditioning. The acid penetrates preferentially along the dentin tubules, which are opened by the conditioning. The intertubular dentin is demineralized to a depth of 3 to 10 μm , according to some authors even up to 20 μm . The acid attack is more effective peritubularly than intertubularly. The average irreversible loss of dentin is around 10 μm , while the depth of collagen exposure is described as around 20 μm , depending on the acid concentration and etching time³⁴.

The acid penetrates a maximum of about 30 μm into the dentin and therefore has no damaging effect on dentin per se³⁵. Depending on the duration of etching with phosphoric acid, different depths of demineralization result, which also depend on whether the etching gel was kept in motion during application or not³⁶. Thus, after 10 s without movement, virtually no demineralization of the compact

dentin was observed, while etching gel kept in motion exposed a collagen fibre layer approx. 3 μm deep within 10 s.

After 60 s, a demineralization depth of 13 μm was measured with movement. In general, an application time of 15 (-20) s for phosphoric acid on dentin is recommended. With longer etching times, there is a risk that the collagen will also be exposed in deeper layers, where it may not be completely penetrated by the components of the bonding system. The collagen areas of the demineralized dentin that are not infiltrated with monomers are considered to be particularly critical with regard to possible degradation. Excessive exposure to the acid can therefore weaken the bond. The collagen network exposed by phosphoric acid etching must subsequently be safely penetrated by monomers.

However, this has a low surface energy, so that the application of surface-active components in the form of primers is particularly important for adhesion to conditioned dentin. In order to visualize the success of the enamel etching, which is sensibly carried out at the same time, at least the enamel margins must be dried. However, this removes important moisture from the unstable collagen fiber mesh, which results in collagen collapsing and sticking together. "Wet bonding" i.e. leaving visible moisture on the dentin surface, can prevent this collagen collapse and promote penetration into the interfibrillar spaces ³⁷.

However, the term "wet bonding" originates from studies conducted with acetone-based systems (e.g. Prime&Bond NT) ³⁷. When acetone is used as a solvent, only moist dentin functions as a bonding partner for such systems. Remoistening the dentin ("re-wetting") is also possible and usually easier to carry out. Primers containing ethanol or acetone are therefore significantly less effective on dried dentin. Tert-butanol is a less technique-sensitive solvent than in XP Bond (Dentsply DeTrey, Constance), but re-wetting is still generally recommended ³⁹.

Incomplete penetration of the hydrophilic primers into the collagen network means that unfilled areas of the nanoscopic interfibrillar spaces remain, leading to "nanoleakage" ³⁷. Clinically, postoperative hypersensitivities then occur more frequently. Water-based (e.g. Adper Scotchbond Multi-Purpose, 3M Espe) and water/alcohol-based systems (e.g. OptiBond FL, Kerr) achieve acceptable rehydration even without re-wetting due to the water contained in the primer ^{39,40,14}.

SelfEtch-based adhesive systems have been developed to avoid the risk of collapse of the collagen network. They contain primers with an acidic pH value that can demineralize enamel and dentin and at the same time penetrate these conditioned surfaces. The acids applied during this type of dentine pre-treatment are deliberately not rinsed off. There are 2-step and 1-step systems. In the 2-step system,

a self-etch primer is applied to the enamel and dentin and dried before the adhesive is applied and light-cured in the second step.

For further simplification, the two components were then combined to form self-etching primer adhesives, also known as all-in-one adhesives. They contain a balanced mixture of hydrophilic and hydrophobic monomers and are so acidic that they simultaneously fulfill the function of etchant and primer in addition to that of adhesive. A distinction is made between self-conditioning preparations with a strong (pH approx. 1), moderate (pH approx. 1.5) or mild (pH approx. 2) etching effect according to the degree of dissolution of hydroxyapatite ⁴¹.

Neither the adhesion to dentin nor the enamel bond strength can be deduced from the pH of the self-conditioning solutions in terms of the bond strength achieved or their long-term stability. The solvent of these self-etching adhesive systems must consist of at least a large proportion of water, as acids can only dissociate and thus develop their etching effect in aqueous solutions. SelfEtch primers only need to be dried after a certain reaction period of usually 30 s in order to volatilize the solvents they contain (acetone, alcohol, and above all water). The dissolved smear layer and the inorganic components of the decalcified dentin are integrated into the bond ^{42,43}.

6. Adhesive System

Adhesives are substances that bond different materials together utilizing mechanical or chemical adhesive forces and evenly dissipate the load forces exerted on the bond ²⁴. They play a central role in composite filling therapy with resins because they enable the hydrophobic composite groups to bond with the hydrophilic tooth structure. They are also used in the treatment of hypersensitivity ^{24,45}. Their effectiveness depends on the ratio of their bond strength to the polymerization stress ^{45,46}. The basis of every adhesively cemented restoration is sufficient drying, which should ideally be carried out "absolutely" under a rubber dam ⁴⁷.

While the now outdated classification of adhesive systems into seven generations provides a chronological overview of the development of bonding agents ^{20,48,49,50}, the current classification according to working steps is functional and practice-oriented ^{51,52}. Here, the adhesives are categorized according to the number of working steps according to acid conditioning.

	One-step system	Two-step system	Three-step system	Four-step system
Self-Etch	Type IV	Type III		
Etch & Rinse Total Etch		Type II	Type I d	Type I c
Etch & Rinse Selective Etch			Type I b	Type I a

Type I systems in this category comprise three to four application steps. Separate conditioning is followed by primer, dentin adhesive, and if necessary, an enamel adhesive²⁰. The bond strength of these multi-bottle systems has been clinically proven^{20,53}.

Phosphoric, Citric, glutaric, nitric, maleic, or dicarboxylic acid as well as complexing agents such as EDTA are used as conditioners of the dental hard substances^{20,54}. The concentrations vary between 2 % for maleic acid and 40 % for phosphoric acid. Usually, 37% phosphoric acids are used for enamel conditioning and mild acids such as maleic or glutaric acid for dentin conditioning^{20,51}.

To avoid acid-induced pulp damage, only the enamel can be etched selectively before the dentin is moistened with the primer (type I a and b). This then contains mild acids such as maleic acid as a self-conditioning primer. Since selective enamel etching is difficult to carry out in practice, especially in the context of minimally invasive cavities⁵¹, the Total-Etch technique is often used (type I c and d). Here, both enamel and dentin are conditioned in one step⁵¹.

As the dentin is faster to demineralize due to its lower inorganic content, the acid is first applied to the enamel and then extended to the dentin²⁰. After exposure times of 30 or 15-20 seconds, the acid, smear layer, and tooth structure residues are rinsed off for approx. 30 seconds^{51,47}.

In the case of prism-free enamel (e.g. diastema sealing, fissure sealing), the etching time should be 60 seconds⁵¹. The acid application dissolves the 1-5 μm thick smear layer²⁴, increases the permeability of the dentin for the penetration of the bonding agent⁵⁵, and conditions the peritubular dentin of the tubule entrance⁵⁵. This can result in exposure of the collagen to a depth of approximately 30 μm , whereby a dentin-layer of 10 μm is irreversibly lost^{20,52}.

The primer can then be absorbed into the collagen network prepared in this way and almost completely free of hydroxyapatite⁵⁷. It consists of hydrophilic mono- and dimethacrylates such as hydroxyethyl methacrylate (HEMA), phosphonated mono-, di- and polymethacrylates as well as acid

monomers. Other components are photoinitiators, stabilizers and fillers²⁴. Since the collagen network remains erect as long as moisture is present^{55,58}, water as a solvent can prevent premature collapse. Particularly in the case of primers with organic solvents based on acetone or ethanol, the dentin must be kept moist as part of the so-called "wet bonding", as insufficient infiltration into the collagen network would otherwise prevent the complete formation of the hybrid layer^{20,60,55,52,59}.

The difficulty is in drying the enamel to produce the etching pattern and re-wetting the collagen meshwork of the dentin⁵¹. The water is then displaced by the acetone²⁴. After a reaction time of 10-30 seconds, the primer is blown off to remove the solvent. A sufficient layer thickness ensures adequate adhesion. The hydrophilic small-chain groups such as HEMA penetrate the interstices of the collagen network in the nanometer range⁵³. The primer thus forms the basis for a bond with the dentin, which is always moist, and prepares it for the absorption of the adhesive^{20,60,52}.

The dentin adhesive contains amphiphilic mono- and dimethacrylates, polymethyl methacrylate (PMMA) and phosphonated mono-, di- and polymethacrylates and thus acts as a link between the hydrophilic primer and the hydrophobic composite^{20,61}. In addition to additives such as bisphenol A diglycidyl methacrylate (Bis- 27 GMA), triethylene glycol dimethacrylate (TEGDMA) or urethane dimethacrylate (UDMA), water, acetone or alcohol are also used as solvents⁵². The adhesive with rather long-chain monomers stabilizes the complex of collagen and primer and forms a hybrid layer with these after polymerization of 20 - 40 seconds^{56,62}.

In the process, the so-called tags^{54,55} are created as plastic cones protruding into the dentinal tubules. Achieving a sufficient layer thickness, which can still be adequately cured despite oxygen inhibition, is of great importance. If the adhesive film on the dentin surface is too thin, the majority of the methacrylates present are not polymerized and do not allow sufficient adhesion²⁰. In addition to the dentin adhesive, an enamel adhesive in the form of unfilled, low-viscosity dimethacrylate can be applied before polymerization²⁰. The composite is then applied.

Type II (etch & rinse, 2-step systems) Similar to type I, separate acid conditioning is carried out first in the form of selective enamel etching or the total-etch technique. However, a primer-adhesive mixture is then applied. These one-component materials are applied once or twice, depending on the manufacturer's instructions, with the first layer acting as a primer and the second layer as an adhesive^{20,53}. Moisture management after dentin etching is a particular challenge with these systems⁵¹.

Type III (self-etch, 2-step systems) Self-conditioning, self-priming adhesive systems do not require separate acid etching. The acidic primer mixture dissolves the smear layer with the help of self-

etching, adhesive monomers and subsequently conditions the enamel and dentin^{53,64}. The etching pattern is always less pronounced than with phosphoric acid etching⁵¹. Since this non-rinse technique does not require the acid to be rinsed off, the problem of overdrying the dentin is avoided⁵³. Simultaneous dentin etching and infiltration of the first methacrylates avoid a discrepancy between the demineralization front and the penetration depth of the hybrid layer^{65,64,54}.

In the case of self-etching adhesives, the pH value determines the effectiveness of penetration and dissolution of the smear layer and thus the quality of the bond to the tooth structure. A stable hybrid layer is only formed at pH values < 1.2⁶⁶. Depending on the pH value, Self-Etch adhesives are divided into strong (pH < 1), moderate (pH around 1.5) and weak systems (pH around 2)^{65,24,52}.

The subsequent application of the bonding agent is intended to stabilize the hybrid layer, establish a secure bond to the enamel and enable adhesion to a composite⁵⁸. For this purpose, crosslinking monomers with polymerizable groups are used as functional methacrylates, which are often HEMA. Since the methacrylates are not hydrolytically stable in acidic, aqueous solutions, the preparations in this category are offered as 2-step systems²⁰. First, the self-conditioning primer is applied, then the adhesive is applied and polymerized⁵³.

The solvents used in self-etching adhesive systems are mostly water and alcohol to improve application to the dentin on the one hand and to facilitate the blowing off of water, which impairs polymerization, with the aid of alcohol on the other²⁰. The problem of a permanent enamel bond without phosphoric acid etching remains with these systems⁵¹.

Type IV (self-etch, 1-step systems) All-in-one adhesives combine acid, primer and adhesive in one application step⁵². The 1-bottle systems in this category are water-based and use bifunctional acrylamides²⁰. More rarely, the adhesive is mixed from two components directly before application⁵¹. In many preparations, only one layer of the self-conditioning and priming mixture is applied²⁰. The problem with these systems is to simultaneously fulfill the requirement of hydrophilicity for dentin adhesion and hydrophobicity for bonding to the composite⁵¹.

The adhesive-tooth bond is still the weak point in restorations with composites⁶³ and must be further examined and improved, as no material can yet guarantee permanent microleakage-free marginal adaptation⁵⁷. In addition, chemical reactions with collagen, hydroxylapatite or untreated dentin should also be more closely integrated into research²⁴.

The advantages of type III and IV adhesives compared to category I and II adhesive systems are easier handling with less technique sensitivity and less time required^{47,46,66}. As a result, application-related errors can be minimized⁵⁷. Disregarding the manufacturer's instructions can lead to a significant

increase in microleakage ^{61,67}. Etch & Rinse systems are still superior to Self-Etch systems in terms of marginal integrity and bond strength ^{20,57,51,63}.

Better results were found for the three-step systems than for the two-step systems with separate conditioning, which in turn should be preferred to the Self-Etch systems ^{20,56}. In purely dentin-restricted cavities, the Self-Etch systems achieved significantly better results ⁶⁷. In terms of enamel adhesion, phosphoric acid etching continues to be the most effective ⁵¹.

List of commercially available products of the various adhesive system classes according to primer functionality.

Systems with dentin-conditioning Primer (sole fusion etching with phosphoric acid and subsequent spraying)	Systems with Etch & Rinse - Technique	Systems with Enamel and Dentin conditioned Primer
Primer to mix: <i>Ecusit Primer/mono (DMG)</i> <i>A. R. T. Bond (Coltene)</i> Primer ready for use: <i>Syntac Optibond (Kerr Hawe)</i> <i>Microbond (Saremco)</i> <i>James-2 (Saremco)</i>	Three-steps-system: <i>OptiBond (Saremco)</i> <i>MicroBond</i> <i>Ecusit Primer/Mono</i> <i>Gluma solid Bond (Heraeus Kulzer)</i> <i>Paama 2 (SDI)</i> <i>Quadrant Uni Bond (Cavex)</i> <i>Solobond Plus (Voco)</i> <i>Cmf adhesive System (Saremco)</i> <i>Syntac (Ivoclar Vivadent)</i> Two-steps-systems: <i>Adper Scotchbond IXT (3M Espe)</i> <i>Admira Bond (Voco)</i> <i>Clearfil Photo Bond (Kuraray)</i> <i>Clearfil New Bond (Kuraray)</i> <i>Cosmedent Complete (Cosmedent)</i>	Seperate application of Primer and adhesive Primer to mix: <i>Crearafil Liner Bond 2V (Kuraray)</i> <i>FL-Bond (Shofu)</i> <i>Resulcin AquaPrie & MonoBond (Merz Dental)</i> Primer ready for usage: <i>Adper Scotchbond SE (3M Espe)</i> <i>AdheSE (Ivolcar Vivadent)</i> <i>Clearfil SE Bond (Kuraray)</i> <i>Clearfil Protect Bond (Kuraray)</i> <i>OptiBond Solo Plus (Kerr Hawe)</i> <i>Contax (DMG)</i>

	<p><i>Excite (Ivoclar Vivadent)</i> <i>Dentamed P&B (Dr. Ihde)</i> <i>Fantestic Flowside (R-Dental)</i> <i>James-2 (Saremco)</i> <i>Gluma Comfortbond +</i> <i>Desensitizer (Heraeus Kulzer)</i> <i>Microbond (Saremco)</i> <i>One Coat Bond (Coltene)</i> <i>Mirage Dentin Adhäsiv</i> <i>(Tanaka)</i> <i>Optibond Solo Plus (Kerr</i> <i>Hawe)</i> <i>PQ1 (Ultradent)</i> <i>Stae (SDI)</i> <i>Solobond Mono (voco)</i> <i>TECO (DMG)</i> <i>XP Bond Dentsply)</i></p> <p>Special forms: (Etching/ 2x Primer-adhesive): <i>Ambarino Bond (Creamed)</i> <i>Bre.bond (Bredent)</i> <i>Ena Bond (Loser&Co)</i> <i>Cumdente Adhesive (Cumdente)</i> <i>Solist (DMG)</i></p>	<p><i>One Coat Self Etch Bond(</i> <i>Coltene)</i> <i>Unifil (GC)</i></p> <p>All-in-One adhesives: To Mix: <i>Adper Prompt L-Pop (3M</i> <i>Espe)</i> <i>One-Up Bond F (Tokuyama)</i> <i>Xeno III (Dentsply DeTrey)</i> <i>Flowsive SE (R-Dental)</i> <i>Futurabond NR (Voco)</i></p> <p>Without Mixing: <i>AdheSE One (Ivoclar</i> <i>Vivadent)</i> <i>Adect* (Bonadent)</i> <i>Adper Easy Bond (3M Espe)</i> <i>AQ-Bond (Morita)</i> <i>artCem One (Merz)</i> <i>Clearfil Tri S Bond (Kuraray)</i> <i>Bond Force (Tokuyama)</i> <i>Futurabond M (Voco)</i> <i>G-Bond (GC)</i> <i>Hybrid Bond (Morita)</i> <i>iBond Self Etch (Heraeus</i> <i>Kulzer)</i> <i>One Coat 7.0 (Coltene)</i> <i>OptiBond all-in-one (Kerr)</i> <i>Xeno V (Dentsply)</i> <i>Scotchbond Universal ** (3M)</i></p> <p>Double application needed*</p> <p>With or without separate</p>
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		dentine etching with phosphoric acid**
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6.1. Biocompatibility to Enamel and Dentin

Since the adhesive systems are applied almost close to the pulp depending on the cavity depth, their biological compatibility is of central importance. The dentinal tubules opened by the etching procedure are sealed with the aid of the bonding agent to prevent the penetration of noxious substances to the pulp and to block fluid movement²⁰. This reduces postoperative hypersensitivity. Overall, pulp tolerance can be classified as good, but direct contact should be avoided^{20,60}. Reversible mucosal reactions have been described when touching the gingiva. Systemic allergies to individual components of the adhesives may occur in rare cases. Mutagenic, carcinogenic, toxic or hormonal effects have not yet been proven, but cannot be ruled out with certainty due to the numerous ingredients and different compositions²⁰.

7. Self – Adhesive Composite for Direct Restorative Therapies

Due to the great success of self-adhesive luting composites, attempts were made to establish the same bonding mechanisms for direct restorative materials - i.e. a self-adhesive restorative material without an actual adhesive. The first commercially available self-adhesive product was Vertise Flow (Kerr Corporation, Orange, USA). This product contains a special monomer (GPDM = glycerol phosphate dimethacrylate), which consists of a functional phosphate group. In dissociated form, this can form chemical bonds with the calcium in the dental hard tissue.

The molecule thus acts as a bonding agent, which etches the surface on one side due to the acidic phosphate group, thus creating a microretentive surface and forming the chemical bond, but has

methacrylate groups at the other end of the molecule, which can crosslink with other monomers. Another product, Fusio Liquid Dentin (Pentron Clinical, Orange, USA), uses 4-META as an adhesive monomer which, similar to glass ionomer cement, forms an adhesion to the tooth structure via carboxylate groups. Unlike traditional flowable composites, self-adhesive flowables do not require pre-treatment with an adhesive.

For a self-adhesive (flowable) composite to come into close contact with the bonding surfaces, it must be actively applied. Vertise Flow is applied with a brush or microbrush, which uses a rubbing motion to spread the flowable composite in an approx. 0.5 mm thick layer on the bonding surface. This active application method enhances the interaction of the acidic monomers with the contact surface. This layer, which should be as uniform as possible, must be cured by light initiation. For Vertise Flow, the manufacturer recommends a polymerization time of 20 s, as adhesive monomers exhibit a delayed reaction during light curing⁶⁸.

While the hydrophilic phosphate group is responsible for the micromechanical and chemical bonding to the tooth structure, the mechanical strength is achieved by the cross-linking of the methacrylate groups. However, the hydrophilic phosphate group is also responsible for water absorption, which is significantly higher with Vertise Flow than with non-adhesive flowable⁶⁸. The hydrophilicity of an adhesive monomer depends on the chemical structure, namely on the spatial distance between the methacrylate groups and the phosphate group. This hydrophilicity is necessary to ensure wettability so that the monomer can come into intimate contact with the substrate surface. On the other hand, this hydrophilic end is susceptible to hydrolysis as well as absorbing water.

However, since the phosphoric acid ester formed by the GPDM molecule is more stable than other esters, such strong hydrolysis of this compound should not be expected, which could impair the long-term stability of the adhesion⁶⁹. In laboratory studies, however, such a decrease in adhesion after thermal stress is observed. Directly after application, adhesion values are measured that are comparable to those of adhesive systems using the etch-and-rinse technique. After thermal cycling, statistically significant reductions were observed⁷⁰.

Clinical use is therefore still viewed very critically. The increased water absorption of Vertise Flow has also been confirmed in studies. Compared to conventional flowable and pasty composites, Vertise Flow showed the highest values, which can be attributed to the higher proportion of monomers per se and especially to the hydrophilic monomers⁷¹. Laboratory tests have measured a rather lower adhesion to the enamel⁷². The adhesion values to the enamel can be improved by prior selective enamel etching with phosphoric acid.

In dentin, however, this pre-treatment harms the bond strength values. This can be explained by the limited wettability of the self-adhesive flowable, as the flowability is not sufficient to penetrate the collagen network exposed by the phosphoric acid etching. As a result, these collagen fibers are exposed to subsequent degradation⁷³. In addition, the phosphoric acid etching of the dentin reduces the calcium content of the bonding surface to such an extent that the chemical adhesion of the adhesive monomer is massively restricted, similar to the case with self-adhesive cements⁷⁴. If the working instructions are followed, i.e. without phosphoric acid etching, the results in the dentin are comparable to the marginal behavior of etch-and-rinse systems. Since Vertise Flow exhibits hygroscopic expansion due to the hydrophilic monomers, the good marginal behavior could be due to the compensation of polymerization shrinkage⁷¹.

On the other hand, some studies found less favorable results and recommended the use of self-adhesive flowables in combination with an adhesive for cervical defects⁷⁵. However, the advantage compared to the use of conventional flowables is then questioned. Studies in Class I cavities showed that Vertise Flow also withstood thermal and mechanical stress when used as a liner before filling the cavity with a composite filling material. However, when the standardized Class I cavities were filled with the self-adhesive flowable using the layering technique, the proportion of marginal leakage was significantly lower at approx. 60 %.

This result was also confirmed in minimally invasive occlusal cavities. The results of a clinical study are now available, which found no postoperative sensitivity in the treatment of minimally invasive occlusal defects after 6 months⁷⁶. The not particularly high adhesion values to the enamel could even be an advantage when bonding orthodontic brackets. Similar to self-etching adhesive systems both in vitro⁷⁷ and in vivo⁷⁸, orthodontic brackets could be bonded with Vertise Flow. Removal is gentler on the enamel than when using Etch-and-Rinse systems⁷⁰.

In clinical use, this could represent a particular advantage, enabling the bonding of brackets with a one-step technique, as the active application of the self-adhesive flowable is certainly easier to carry out on a smooth labial or buccal surface than in a cavity. Even the previous phosphoric acid etching did not increase the adhesion to the enamel at the beginning but only reduced the drop in adhesion values after thermal stress. In this study, a drop in adhesion values was reported after storage in water, which the authors attributed to the hydrophilic monomers⁷⁰.

A similar problem arises with adhesion to precious and non-precious metals, which the manufacturer claims is given for Vertise Flow [Vertise Flow - Technical Bulletin]. This statement could not be confirmed in an in-vitro study, in which the bond to the brackets was lost after exposure to water⁷⁰.

8. Conventional adhesive cementation of restoration vs. filling restoration

Classical adhesive cementation means the implementation of the techniques as mentioned earlier in the course of indirect restoration. With proven multi-bottle adhesives such as Syntac (Ivoclar Vivadent, Ellwangen, Germany), an additional 15-second etching of the dentin with phosphoric acid during enamel etching significantly increases the dentin bond strength ⁷⁹. With all-in-one adhesives (not to be confused with self-adhesive luting cements), the enamel and dentin surfaces are pretreated in one step. Based on the components used, there are the terms multi-bottle and single-bottle systems, whereby the former are still considered to be very reliable and less technique-sensitive ⁸⁰.

All systems that involve pre-treatment of the enamel and dentin require reliable drying. The durability of conventional adhesive luting of restorations has been extensively proven in clinical studies ⁸¹. Adhesive cementation of silicate ceramic inlays and partial crowns is a particularly effective means of preserving the remaining tooth structure during cavity preparation and stabilizing it with the aid of adhesive cementation ⁸². Lithium disilicate ceramics must also be adhesively cemented as inlays and partial crowns. Adhesive cementation can be used for lithium disilicate crowns (especially if there is insufficient retention). Adhesive bridges made of metal or all-ceramic can also be adhesively cemented ⁸³.

For conventional crown and bridge restorations (precious metal or zirconium oxide), the advantage of adhesive cementation (with the associated additional effort) compared to conventional cementation with sufficient retention is controversial. One advantage of adhesive cementation of all restorations is the secure sealing of the dentin ⁸¹.

9. Adhesion to partially restored restorations

9.1. Restoration of composite

The complete removal of a composite restoration is very time-consuming and is usually accompanied by an additional loss of tooth structure. Especially in the case of purely composite-limited defects, it

makes sense to repair effectively. Previously, only shear tests were available in the literature to estimate the potential of repairs. Based on these studies, the repair strength was assumed to be approx. 65% of the cohesive strength of intact composite samples. Roughening with silicon carbide stones or preparation diamonds on the one hand or sandblasting on the other were classified as promising methods ⁸⁴.

Studies on teeth with the repair of aged composite fillings showed that a deliberate extension of the repair cavity into the adjacent enamel is not recommended ^{37,44}. Although this frequently occurring situation can also be solved, it should not be aimed for a priori, as the exposure of several bonding surfaces (composite / enamel / dentin) creates more problems than it solves ^{37,44}.

Another study investigated the influence of the preparation geometry on the integrity of the repair bond in aged composite fillings. The main result here was that a dovetail preparation does not make sense, as this significantly increases the C-factor and increases system-immanent stresses ⁸⁵. Minimally invasive preparations showed the best results, whereby the lining technique with flowable composites further improved the marginal quality ⁸⁵. Several approaches are discussed for the pretreatment of the aged composite to be repaired. Among many alternatives, intraoral sandblasting with corundum (27 µm or 50 µm) has proven to be the best method, as intraoral silicatization works well, but only composite should be present as the sole adhesion substrate ^{86,87,88}.

Rotating tools (silicon carbide stones, coarse diamond grinders) also produce a measurable increase in surface area on the composite to be repaired, but it is difficult to penetrate the barely accessible marginal areas without damaging adjacent teeth. Therefore, in addition to effectiveness, access to hard-to-reach cavity margins is also an advantage of sandblasting ^{88,84}. To date, there is only little clinical data available on new fabrication vs. repair: In the case of marginal discolored composite restorations, no difference was found between repair, sealing or new fabrication after several years of observation ^{89,90}.

9.2. Restoration of ceramic

Since by far, the majority of ceramic failures are caused by fractures, the question of reparability is just as interesting and important as for composites⁹¹. In the case of large fractures (bulk fracture), a

new restoration is often necessary, but in the case of the much more common chipping, a repair would be preferable to a presumably tooth structure-robbing and expensive new restoration. Even in the case of esthetically delicate ceramic veneers, Peumans et al. showed that an attempt to repair partial fractures is preferable to a new restoration ⁷³. In a laboratory study simulating a two-year clinical wear period, only hydrofluoric acid and CoJet pretreatment were able to permanently retinue repair fillings.

Unbuffered hydrofluoric acid is highly toxic and can lead to profound tissue damage. Its intraoral use is therefore no longer indicated today, as buffered hydrofluoric acid products are approved for intraoral use. An alternative is the universally applicable method of intraoral silicization (Cojet, 3MESPE) ^{85,88}.

10. Specific forms of adhesions

10.1. Infiltration of enamel lesions

In addition to fissure sealing, infiltration of labial and proximal enamel lesions is the least invasive treatment option for initial caries lesions ⁹². Here, too, the tooth structure is conditioned (10% hydrochloric acid) and an adhesive (infiltrant) is applied, so in principle, this can also be considered an adhesive technique. However, the application times of 120 s (hydrochloric acid) and 180 s (infiltrant) are considerably longer than with adhesive restorations. The application of a specially developed double foil also allows direct etching and infiltration both proximally and labially ^{92,88}.

The clinical potency for arresting enamel lesions has been proven in clinical studies. However, after three years, there has not yet been any major penetration of the market. The broader international perception is only just beginning ^{93,94}.

10.2. Infiltration of carious dentin

Infiltration can also occur in caries-altered dentin, which is now also targeted therapeutically. It has been known for some time that both caries-softened and therefore infected dentin and dentin affected by caries, which is partially sclerosed and can be penetrated with conventional adhesive techniques without a high bacterial load. Interaction structures may even be more pronounced here than when bonding to freshly cut, healthy dentin ⁹⁵. Although the "resin tags" become shorter due to mineral deposits in the dentin tubules, the thickness of the hybrid layers is sometimes considerably greater than when bonding to unmodified, freshly cut dentin ⁹⁶.

This is important for the assessment of the adhesive technique in these areas for two reasons: 99% of the published studies on the evaluation of dentin bonding phenomena were conducted on healthy permanent teeth, which would not have "required" ⁴⁰. Although this is an important substrate for the establishment of adhesion and its standardized examination, carious or sclerotic dentin usually remains after caries excavation. However, this plays an important role in the sealing of relevant areas in contrast to the pulp dentine complex. After caries excavation, areas with caries-altered dentin usually remain ⁸⁶.

From a modern cariological point of view, a more gentle caries excavation (e.g. with polymer drills) is desirable today, as remineralizable dentin can usually still be preserved ^{97,96}. The remaining carious dentin can also be specifically sealed with adhesives. This preservation helps to protect the tooth structure and the vital pulp.

10.3. Adhesion in structural disorders

Adhesive measures have also become widely accepted in the treatment of teeth with structural anomalies (e.g. amelogenesis imperfecta, dentinogenesis imperfecta, molar incisor hypomineralization, fluorosis, etc.), even if only to bridge important periods in paediatric dentistry. These measures are not comparable to the effectiveness of permanent dentition, but they are expedient and make therapeutic sense.

10.4. Adhesion to iatrogenically altered surfaces

In the context of endodontic procedures (rinsing agents), color corrections (bleaching agents) or bleeding management in extremely deep cavities (astringent substances, etc.), changes to the surface of the tooth structure can occur that influence adhesion. For example, it is known that various agents used to promote hemostasis can dramatically reduce adhesion to composites under certain conditions. Therefore, after iatrogenic alteration of tooth structure surfaces, further intermediate steps may need to be taken before an adhesive technique can be used successfully. This has not yet been billed either.

11. Discussion

An adhesion promoter system must be used that allows a hydrophobic material to be attached to a hydrophilic substrate. Therefore, dentin bonding agents were developed to form a chemical bond with the organic or inorganic part of the dentin.

New adhesive systems have therefore been developed that enable micromechanical bonding of the hydrophobic composite material to the moist dentin surface.

The adhesive components provide penetration capability and mechanical stability.

To not destroy the collagen mesh 40% of phosphoric acid gels are used for enamel conditioning. Self-etch-based adhesive systems contain primers that avoid the risk of collapsing of the collagen network. All-in-One adhesives contain a balanced mixture of hydrophilic and hydrophobic monomers which simultaneously fulfill the function of etchant and primer. To prepare the enamel a phosphoric concentration of 37% with an etching time of 30s is recommended as ideal.

Furthermore, the enamel prisms should be etched transversely since the adhesive can only flow laterally into the prism areas.

12. Conclusion

This Overview of adhesive dentistry depicts the seven generations and types (Etch & Rinse-adhesives, Universal-adhesives, self-etching adhesives) of adhesive components that have been introduced during the last 30 years. Providing patients with minimally invasive dentistry is an

ethical obligation, leading to an ongoing improvement aiming to simplify the process, improving stability over time and their bond durability.

It is necessary to understand the three main components of the bonding systems (Etchant, Primer, bonding resin) in order to comprehend the hybrid layer formation using universal etch technique and Self-Etch technique.

To prepare the enamel and dentin, an etchant with 35-40% phosphoric acid, creates microporosity and micromechanical retention to the composite restoration.

To penetrate into the hydrophilic dentin a primer is used, composed of hydrophilic monomers, which influence the bond strength.

The bonding agent can be defined as a thin layer of resin applied between the conditioned dentin and a resin composite restorative material or cement, connecting the hydrophilic resin primer and the hydrophobic resin composite.

Since dentin adhesion is more challenging than enamel adhesion, the etch-and-rinse technique is considered as a highly sensitive and critical technique, due to the risk of over-dried or over-wetted dentin, causing low monomer diffusion, collapsing of collagen fibers, incomplete monomer polymerization and early degradation.

Self-Etch adhesives, which are considered simplified adhesive materials, are assumed to create less post-operative hypersensitivity, using the smear layer as a bonding substrate. Because self-etch adhesives use the smear layer as a bonding substrate insufficient enamel etching occurs resulting from their less acidity and less injurious effect on the dental substrate.

Universal adhesives have been developed that allow pre-etching with phosphoric acid in the total-etch or selective-etch approaches to achieve a durable enamel bond. As a result of its important complex formulation and their specific properties, it allows universal adhesives to create a bridge over the gap between hydrophilic tooth structure and the hydrophobic resin restorative.

It is up to the skill and indication of the clinician to decide whether the adhesive restoration technique works. Even the most modern techniques fail if less time is used for adequate treatment or the ignorance of basic concepts during treatment.

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