EGYPTIAN
DENTAL JOURNAL
www.eda-egypt.org

VOL. 71, 2581:2594, JULY, 2025
PRINT ISSN 0070-9484 • ONLINE ISSN 2090-2360

CONSERVATIVE DENTISTRY AND ENDODONTICS

Submit Date : 16-03-2025 • Accept Date : 29-04-2025 • Available online: 15-07-2025 • DOI : 10.21608/edj.2025.368541.3419

IMPACT OF STORAGE TIME ON MICROTENSILE BOND STRENGTH AND NANOLEAKAGE EXPRESSION OF UNIVERSAL ADHESIVES ON DENTIN: LITERATURE REVIEW

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Search strategy

The National Library of Medicine (MEDLINE/PubMed), Scopus, EBSCOHost and Elsevier were searched for published studies between 2015 and 2025 that discussed impact of storage time on microtensile bond strength (μTBS) and nanoleakage (NL) expression of universal adhesives (UAs). The search queries in the database were formulated with various Boolean operators such as AND, OR [1]. The keywords used in searching the databases were "Microtensile bond strength" OR "Nanoleakage expression" AND "Universal adhesives" AND "Composite resin" AND "Thermacycling".

How did adhesion start?

The aim of restorative dentistry is to restore the structure, functionality, and appearance of damaged or carious teeth. The manner in which dentist approaches restorative dentistry has changed over years due to the evolution of new dental material. By enabling dentists to accomplish minimally invasive operations, maintain tooth structure, and produce superior cosmetic results, adhesive dentistry caused a paradigm change in dental practice. The shift from composite restoration into amalgam has gained popularity lately due to the esthetic concerns in addition to health hazards and environmental impact of mercury in amalgam. Therefore, it's now more recommended to use more biocompatible and environmentally friendly restorative materials. [2]

The success of both direct and indirect restorative operations depends on choosing the most suitable adhesive material among all variations available.[3] The 1980s saw the introduction of the adhesive technology needed to attach restorations to tooth structure. The desire for simplicity, effectiveness, and dependability in clinical practice procedures to achieve long-lasting direct and indirect restorations has propelled the evolution of adhesive systems over the decades, from the largely ineffective systems of the late 1970s and early 1980s to the comparatively successful total-etch and self-etch systems of today.[4]

The Swiss chemist "Hagger" started the concept

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of adhesion in 1949 by using Glycerol Phosphate Dimethacrylate (GPDM) for dentin bonding only not for enamel. Actually, GPDM is still used today as a primary functional monomer in Optibond FL/ XTR Universal adhesives by Kerr.[5] For that reason, he was called the father of the modern dental adhesives as his groundbreaking innovation is still used today. He was the first to advocate that acidic monomers are aiding in interacting with tooth surface at molecular level.[6] In 1952, two scientists "McLean and Kramer" discovered an intermediate layer between tooth substrate and adhesive system which was later on identified as the "hybrid layer".[7] They described hybrid layer as changes on dentin as a narrow zone approximately 3 µm deeply stained with hematoxylin and it was found in all teeth filled with Sevitron-adhesive. They found out that these changes occuring in dentin substrate are promoted by acidic monomers in GPDM by Hagger.[8] Since the it was one of the first functional monomers used in self-etch adhesive, we can actually state that the use of GPDM may now be considered part of history.[8] Adhesion had evolved into 1954 by Buonocore who was the first to invent "acid-etching" technique to enamel. In 1955, he described the use of phosphoric acid 85% for etching enamel and producing prism like tags which are helping in micromechanical retention. However, until 1968 his concept was not published.[6]

Substrate:

After Buonocore's invention of enamel etching, he developed 85% phosphoric acid which alters the enamel surface and provides a surface suitable for bonding with resin. Both enamel and dentin are having dissimilar mechanisms of adhesion as enamel conditioning results in microporosities where resin penetrates to form "prism-like" resin tags. As a result, enamel adhesion depends more predominantly on micromechanical hence less problematic, than dentin which depends mainly on chemical adhesion.[9] Enamel substrates contains about 96% minerals which makes it a perfect substrate for to form a tight adhesive joint.[10] Until now and after over 60 years, enamel etching prior to bonding by resin-based materials is still considered the gold standard for bonding.[9]

Dentin has a more complex and humid structure which makes its adhesion challenging and less predictable.[11] Moreover, its mineral content is on average 45%, organic matrix are about 33% "most commonly type I collagen" and rest of the composition is water.[12] Besides, dentin consists dentinal tubules which are inverted cone in shape and extend from the pulp into dentin in a transverse direction with larger diameter facing the pulp.[13]

Dentin contains smear layer and organic matrix and fluid inside the dentinal tubules which make adhesion more challenging. Additionally, the number of dental tubules varies with dentinal depth since deep dentin contains more water and superficial dentin has a lower density.[14] Resulting from the low number of dentinal tubules in superficial dentin, bonding strength depends mainly on resin infiltration into intertubular dentin while in deep dentin bond strength is increased because of the intratubular resin permeability.[15]

In reality, several treatments are necessary prior to adhesion to transform the hydrophilic, crystalline, impermeable, acid-labile surface of dentin into a more hydrophobic and acid-resistant surface because of its complicated histology.[14, 16] There is no set procedure for getting adhesive solutions to adhere to dentin in a stable and ideal manner.[17] Besides, micromechanical retention can't be fully reliable as adhesives cannot infiltrate completely all the collagen fibrils of demineralized dentin.[18] These non-infiltrated collagen fibrils are more susceptible to hydrolysis and degradation.[19]

Hybrid layer degradation is considered the primary factor of failure.[20] Degradation occurs either after bonding agent application which is contributed to the activation of MMPs. Furthermore, because of the hydrolytically vulnerable groups in the molecular structure of methacrylate-based resin monomers, such as ester, urethane, hydroxyl, carboxyl, and phosphate, it can happen after bonded polymerization.[21]

One of the main challenges in dentin adhesion is smear layer. It is defined as an area of surface-dispersed tooth preparation debris.[22] Constituents of smear layer are hydroxyapatite and collagen that is denatured by the heat generation during tooth prepearation. Many studies had stated that smear layer has an influence on the bonding performance of adhesive to dentin.[22, 23, 24, 25] Smear layer may be completely or partially removed prior to bonding. [26] According to the degree of smear layer removal, dental adhesives are classified into total-etch or self-etch.

Etching and rinse or the so-called total etch strategy means using phosphoric acid prior to bond application which leads to removal of the smear layer. It had been shown that the optimum concentration for total removal of SL is 30-40% any less or more concentration had exhibited inferior bond strength.[27] It was stated that this strategy is better working with enamel due to its higher mineral content, however in dentin it causes long-term disintegration caused by the matrix metalloproteinase enzyme (MMP), an endogenous enzyme that is in charge of collagenolytic activity.[28]

On the other side, self-etch adhesives (SE) are considered simplified adhesives as they don’t require etching and rinsing and offer some advantages as decreasing post-operative sensitivity as they don’t remove smear layer completely which leads to penetration of smear layer into dentinal tubules; hence called smear plugs. The smear layer is modified and incorporated in the hybridized complex. Another advantage is that both etching and infiltration of the resin monomers of SE adhesives into dentinal tubules are occuring simultaneously.[28] Because SE adhesives contain monomers with acidic functional groups that simultaneously etch and prepare the dental substrate, they eliminate the need for the acid etching phase for dentin.[17]

Actually, it can be stated that self-etch strategy is the first strategy used in dental adhesives at the first and second generations. This is contributed to the fact that Hagger used GPDM directly over dentin without etching or conditioning. The gold standard ER adhesive is Optibond FL which contains a highly hydrophobic functional monomer called GPDM which can chemically bond to HA in enamel and dentin.[29] Meanwhile, the gold standard in SE mode is Clearfill SE.

In order to eliminate some of the disadvantages of ER, such as its increased number of stages, longer application time, method sensitivity, and difficulties in managing dentin wetness, self-etch and universal adhesive solutions were established in dentistry.[30] Although these adhesion systems are not working well with enamel, they are preferred at dentin substrate to leave some hydroxyapatite crystals around collagen fibrils for protection and allows the functional monomers to potentially interact with the substrate. ER are not preferred in dentin as they cause demineralization for several micrometers which are not fully penetrated with resin resulting in nanoleakage after aging.[31]

Meanwhile, the gold standard in SE is Clearfill SE which was the first adhesive to eliminate the use of phosphoric acid etching on dentin.[32] The most recent adhesives are those which have the flexibility to utilize in ER, SE or the so-called selective enamel etching strategy which means using phosphoric acid etching on enamel only.[33] According to the manufacturers, practitioners select bonding techniques according to the kind of tooth structure and their own preferences. This newest version of dental adhesives is called “universal adhesives”.

Classification of adhesives according to generation

From the first to eight generation of UA, an evolution in their bonding characteristics was developed. The first and second generations subjected only 1-3 Mpa and 4-6 Mpa bond strength, respectively as they directly adhered to smear layer without modifications. These bonding agents were designed to create ionic bonds with hydroxyapatite or covalent bonds (hydrogen bonds) with collagen. Coupling agents were added to increase their mechanical strength.

The second generation of dentin bonding agents was produced in the late 1970s with the goal of improving the coupling agents utilized in the first generation of adhesives. In the second generation of dentin adhesives, bis-GMA resins were primarily supplemented with polymerizable phosphates to promote adhesion to the calcium in mineralized tooth structure.

Starting from the third generation, acid etching concept was developed which modified or partially removed the smear layer. However, the unfilled resin led to difficulty in penetrating dentinal tubules which was still a problem in achieving favorable bonding strength.

The fourth generation (three-step etch and rinse) is still regarded as the gold standard because it was the first to fully remove the smear layer using powerful acid etching. Dentin and enamel are simultaneously etched with phosphoric acid (H3PO3) for 15–20 seconds as part of the total-etch method and wet dentinal hallmarks of the 4th generation systems. Collagen collapse can be avoided, though, by keeping the surface damp, a process known as "wet bonding." The bond strength, which is still regarded as low-moderate, had reached 20 MPa.

In the 5th generation (two-step etch and rinse), more simplified adhesive systems were used as it combined both primer and adhesive in one bottle with a separate etching procedure[34]. However, water sorption was a problem which raised due to the acidic and hydrophilic polymerized primer which is not covered with an additional hydrophobic layer. Those adhesives are nor compatible with chemical cure core-materials as their high acidity leads to dissolution of tertiary amines.

In the 6th generation (two-step self-etch) about in 1990s and early 2000s, self-etch adhesives were firstly introduced through a combination between etchant and primer at the same bottle. This combination was done in one (acidulated primer and bonding agent mixed prior to application) or two bottles (acidulated primer in one bottle and hydrophobic bonding agent in the other). The biggest advantage in this generation is the less dependency on the hydration state of dentin.[35]

In the 7th generation which was introduced in 2005, a true all-in-one bottle was finally released. The main drawback in 7th generation is the presence of significant amount of water in their formulations and may be prone to hydrolysis and chemical breakdown. Moreover, after polymerization it's more prone to water sorption and limits the depth of resin infiltration into the tooth which may lead to void formation.[36] In the 8th generation, nanofillers were incorporated in the adhesive composition to increase their mechanical strength and handling properties and increase the resin penetration.[37] The first launched adhesive was Futurabond DC, Voco which contains nanosized fillers in 2010.

Composition of universal adhesives:

1- Cross linking monomers (polymerizable monomers)

Those are the monomers which are corresponding to the adhesive resin. They are responsible for the hybridization of the collagen on the dentin substrate, as well as co-polymerization with the restorative composite material and functional monomers in adhesives.[38] All crosslinking monomers are hydrophobic to stabilize the hybrid layer. Cross linking monomers are classified into acrylates (–CH2–CH=COO–) and methacrylates (–CH2–C(CH3)=CH2) and methacrylamide monomers. Examples of methacrylates are BisGMA, TEGDMA, UDMA and Ethylene glycol dimethylacrylate.

Acrylates and methacrylates are more prone to hydrolytic degradation due to the presence of ester group in their formulation.[38] Therefore, replacing the ester group with amide group which is more stable was a new evolution in crosslinking monomers to increase their stability and longevity. [39] As stated in ahmed et al, acrylamides can replace HEMA and still give favorable results.[40]

2- Spacer

Either in crosslinkig or functional monomers, a spacer chain is existing which plays a crucial role in determining many properties. For example, their size determines the size of the monomer, as increasing their length leads to less volatility of the monomer, their polarity affect the monomers hydrophilicity and solubility and presence of voluminous groups within the spacer chain may modify their reactivity.[40]

3- Functional monomers

Those monomers are corresponding to dentinal primers. Since the degradation of collagen fibrils within the hybrid layer jeopardizes the long-term stability of dentin bonding, interaction with collagen is likely the most crucial factor when discussing adhesive systems. The high bond strength with dentin is believed to be explained by the chemical characteristics of functional monomers.[41] For that reason, functional monomers play a crucial role in bonding strength of adhesives. Long-term adhesion is accomplished by the adhesive and substrate chemically joining to form a three-dimensional collagen-resin biopolymer that creates a stable and continuous bond between the adhesive and the dentin substrate.[42]

Functional monomers are classified into phosphate containing (like 10-MDP, dipentaerythritol penta-acrylate phosphate (PENTA), and HEMA-P) and those containing carboxylic acids (such as 4-MET and 4-methacryloxyethyl trimellitate anhydride (4-META)). Functional monomers' acidity determines the degree adhesives acidity; with sulfonic acid is being the most aggressive, followed by phosphonic, phosphoric, carboxylic, and alcohol acids.[17] A brief about different functional monomers[17] is shown in **Table 1.**

Until now, MDP is forming the most stable salts "10-MDP-Ca salts" hence called nanolayer.[43] The endurance of bonding to tooth dentin is believed to be enhanced by self-assembled nano-layering. GPDM–Ca salt on the other side are considered weaker as GPDM does not reveal collagen; instead, it encourages the formation of a thick HL with exposed collagen.[44] Since PENTA possesses five vinyl groups instead of the one found in 10-MDP monomer, it is thought to be more stable than MDP. These four extra vinyl groups which are connected to P group[45] also make PENTA more resistant to hydrolytic destruction; however, Han et al. demonstrated the contrary.[46]

Role of HEMA in UAs:

2-Hydroxyethyl methacrylate is the most used functional monomer in primers.[47] This hydrophilic methacrylate monomer is highly used in single bottle adhesives as it has a small size which enables penetration of resin into collagen fibrils.[48] Moreover, it decreases the chance of phase separation between hydrophobic and hydrophilic components of the adhesive.

Although HEMA has a big role in wetting of the adhesives and it has high water permeability, high concentrations of HEMA in an adhesive may cause the polymer's mechanical qualities to deteriorate over time through hydrolytic breakdown..[49] Besides, it's more liable to swelling, discoloration

TABLE (1) Functional Monomers found in UAs.

Functional Monomer	**Description**
Pentamethacryloyloxyethylcyclohexaphosphazene Mono Fluoride	Monomer with five methacrylate-alkyl chains and a fluoride as a functional group
N-phenylglycine glycidyl methacrylate and N-tolylglycine glycidyl methacrylate	Monomer with tertiary aromatic amine group
Dimethylaminoethyl methacrylate (DMAEMA)	Monomer with tertiary amine group
Methacryloyloxydodecylpyridinium bromide (MDPB)	Monomer with antimicrobial agent "quaternary ammonium compound"
N-methacryloyl-5-aminosalicylic acid	Contains Salicylic acid.
Methacrylate and Methyl Methacrylate	Rarely used due to their allergic reactions.
4-methacryloyloxyethy trimellitate anhydride (4-META) and 4-MET	Hydrophobic and highly acidic with functional hydrophilic carboxyl groups with more soluble Ca salts than MDP-Ca which results in weaker bond strength. 4-META + water result in 4-MET
4-acryloyloxyethyl trimellitate anhydride	Similar to 4-MET but contains acrylate group instead of methacrylate.
Phenyl-P	Monomer with monohydrogen phosphate group
HEMA-P	Monomer with methacrylated H3PO4–HEMA esters group
GPDM	Monomer with two methacrylate groups linked by a short carbon spacer to one P acidic functional group
(Dipentaerythritol penta-acrylate phosphate) PENTA	It contains P ester monomers containing carbon=carbon double bonds and a P group [-OP(=O)(OH)2].
10-Methacryloyloxydecyl dihydrogen phosphate (10-MDP)	It was patented by Kuraray in 1981 and found in many adhesives as All-Bond Universal (Bisco, Inc., Schaumburg, IL, USA), Adhese Universal (Ivoclar Vivadent, Schaan, Liechtenstein), G-Premio Bond (GC, Tokyo, Japan), Futurabond U (Voco, Cuxhaven, Germany), Clearfil Universal Bond (Kuraray Noritake, Tokyo, Japan), and Scotchbond Universal (3M ESPE, St. Paul, MN, USA).

and hydrolysis after polymerization which are characterized by water blisters at the adhesive layer. Moreover, HEMA has some biological hazards as it leads to genotoxicitiy.[50] A Therefore, less hydrophilic monomers are recently launched to overcome those drawbacks as they have reduced water absorption and higher mechanical stability.[47]

Consequently, the development of HEMA-free dental adhesives followed in an effort to enhance their mechanical, chemical, and physical properties.[51] Replacing HEMA which is monofunctional with dimethacrylate monomers enhances the mechanical properties as it contains two polymerizable extremeties which produces crosslinked polymers and leads to less hydrolytic degradataion and increases the stability during contact with the oral environment.[52] Given the aforementioned factors, a variety of dimethacrylates are available for this use, such as GDMA, or "glycerol dimethacrylate." Also, acrylamides as methacrylamide monomer [5] and hydroxyethyl acrylamide monomer "HEAA" and diethyl acrylamide "DEAA" [53] and hydrophilic amide monomers in Clearfill Bond Quick Universal.

Solvents in UAs

A solvent is used to dissolve other components into a solution. Water, acetone and ethanol are the most common solvents used in adhesives[54] to dissolve other monomers which helps adhesive primers and/or resins become less viscous and better diffuse through etched dentin.[55] Therefore, regardless of the adhesive application method—etch-and-rinse or self-etch—or the pH of the adhesive formulation (mild/intermediately strong or strong, in the case of self-etch adhesives), solvents are added to dental adhesive formulations to aid in the establishment of micro-retention with both enamel and dentin.[56] Solvents eliminate water from collagen fibrils and dissolves the amphiphilic resin which increases the surface tension.

One important determinant of contemporary adhesives' bonding efficacy is solvent evaporation. To complete this therapeutic step successfully, a number of criteria must be taken into account. The evaporation capacity varies between solvents according to the vapor pressure (mmHg); the point at which a liquid turns into a gas. The more volatile the solvent, or the easier it is for it to evaporate, the higher the vapor pressure. Acetone has higher vapor pressure than water and ethanol.[57] Despite its despite its high volatility, acetone does not effectively enhance water evaporation because it does not form an azeotrope with water.[58] When volatilized for a longer duration than the manufacturer recommends, universal adhesives containing ethanol-based solvents exhibit increased bond adhesiveness to dentin, in contrast to those that use acetone as a solvent, where longer volatilization times do not significantly alter the bond.[59] Another important characteristic of solvents is their ability to bind to hydrogen, which allows collagen to expand once more when dehydrated, improving resin dispersion and self-bonding. The number of hydrogen bonding sites and the attraction between the polymer and solvent are influenced by the polarity of the resin.[60]

Acetone has the highest vapor pressure among all the solvents which is 200 mmHg, on the other side ethanol is 54.1 mmHg. The main function of the solvent is creating an azeotropic solution with water which facilitates vaporization of the remaining water [61] within the demineralized dentin substrate. In turn, the superior vaporization will increase the strength of hybrid layer.[54]

Modern solvents were invented to make the moist degree less sensitive as tert-butanol and iso-propanol which were mainly used by Prime and Bond, Dentsply. Both have larger molecular weight which makes collegen fibrils shrinkage less likely to occur.[54] Isopropanol has a vapor pressure close to water and its viscosity is about 400 folds less than water and about twice that of ethanol. When mixed with water, it results in a variety of solubility, wetting, and evaporation characteristics in more intricate adhesive combinations.[62]

Classification of UAs according to their pH:

The smear layer dissolution and the dentin surface's etching can be strongly impacted by the pH levels of self-etch adhesives. The acidity of universal adhesives varies; it has been demonstrated that acidity affects both bond stability and binding strength to dentin.[63] Adhesives can be categorized as ultra-mild (pH >2.5), mild (pH ≈ 2), intermediately strong (pH 1 to 2), and strong (pH <1) based on their pH value. In two-bottle systems, the higher pH of the second bottle solution is used to offset the lower pH of the first bottle solution, which is often used to promote wettability and slightly etch the tooth material. [64, 65] An adhesive with a low pH may initially appear advantageous, but the presence of acidic monomers can also cause a low pH that interferes with the polymerization reaction.[66]

Mild and ultramild universal adhesives are the most commonly used, as their retention does not only depend on the micromechanical retention but also chemical bonding via functional monomers is undergone. In mild self-etch adhesives, only superficial surface of the dentin is etched which leads to leaving hydroxyapatite crystals around the collagen fibrils open to potential chemical interaction. The smear plug is typically not entirely extracted from the dentine tubule. This results in the formation of a shallow hybrid layer with submicron measurements, as do ultra-mild self-etch adhesives. In contrast, strong self-etch adhesives demineralize dentin in a manner similar to that of etch-and-rinse adhesives. Since the mild self-etch adhesives use the smear layer as a bonding substrate and leave behind residual smear plugs that reduce dentinal fluid flow, they are thought to result in less post-operative pain than etch-and-rinse adhesives.[67, 68] Actually, mild UAs are considered the most stable adhesives even though their bond strength may be improved by selective enamel etching technique according to a previous meta-analysis.[69]

Microtensile bond strength test

µTBS is the uniform stress strain field, which is crucial to accomplish most of the failure on the bond interface, even more uniform than in shear bond strength test.[70] The main benefit of the µTBS test is that it allows researchers to focus on three-dimensional substrates that are therapeutically relevant. Furthermore, the assessment of the bond strength of relatively small specimens (less than 1 mm2 cross-sectional areas) is attributed to it. Cohesive failure is less common with μTBS than with “macro” tensile bond strength. Also, it allows more specimens from the same tooth to be measured which is which is an advantage as it’s difficult to collect human teeth.

Despite of its advantages, µTBS test is still a time consuming, operator sensitive and complicated testing. The main unresolved problem which is faced during the test is pretesting failure which needs further improvements. [71] Another limitation is the uncertainty of whether non-adhesive failures should be considered as they do not represent the clinically-relevant failure mechanism that occurs in real cavities.[72, 73] In contrast to “macro” tensile bond strength, cohesive failure is less often occurring with µTBS. Also, it allows more specimens from one tooth to be measured which is an advantage as it’s difficult to collect human teeth.

The hourglass form, which was designed to mimic the specimen shape created by Akimoto et al. [74], was the first specimen shape utilized for the µTBS test. and was used for the study to test the tensile strength of mineralised and demineralised dentin. The new sample preparations of microtensile tests are bar shaped instead of the dumbbell shaped which was used at the beginning. Bar shaped beams could overcome the dehydration problem of the test as It shortens the time between sample preparations and keeps the samples hydrated until the test is conducted.[75]

Eldamanhoury et al.[76] assessed microtensile bond strength of Adper Easy Bond, Clearfil S3 Bond, iBond, Optibond All-in-One, Xeno IV, and Adper Single Bond Plus as a control. Half of the groups were assessed immediately while the other half was subjected to 5000 thermocycles after aging. It was stated that all adhesives results dropped after aging.

Sangwichit et al.[77] investigated whether four UAs would be affected by thermocycling in ER and SE modes. The study design was divided into immediate and after 10,000 thermacycling. Regarding SE, it was concluded that all adhesives showed lower results after aging with a significant difference ($p<0.01$).

Ahmed et al.[78] investigated whether three universal adhesives would benefit from an extra bonding layer (EBL). They were divided into three thirds; after 1 month without TC, after 25,000 thermocycling and 50,000 thermocycling. Hence, aging significantly affected µTBS of all tested adheisves, however the results were not fully exhibited in this study.

Tichy et al.[79] evaluated µTBS of contemporary five universal adhesives immediately and after 15,000 thermocycling. It was stated that bond strength didn’t show significance difference after aging in three adhesives while Clearfil Universal

Bond Quick, Clearfil Universal Bond Quick without amide monomer, and Prime and Bond Universal bond, but a significant decrease was observed with G-premio bond and Ecosite bond.

Guo et al.[80] evaluated µTBS of experimental resveratrol-doped adhesive adhesives immediately and after 10,000 thermacycling. It was stated that the experimental adhesives showed non-significant difference after aging ($p>0.05$).

Yin et al.[81] evaluated the bond strength of universal adhesive systems in self-etch at the repair interface between aged and new composite resins. The microtensile bond strength was measured immediately and after 10,000 thermocycling processes. It was stated that thermocycling significantly reduced the bond strength in both immediate and delayed groups.

Tang et al.[82] assessed the microtensile bond strength of four universal adhesives in SE mode to flat dentin. Delayed groups were assigned to 50,000 thermacycles. It was stated that aging significantly decreased results of all adhesives (Healbond Max, Healbond MP, Clearfill SE) and one increased after aging (Scotchbond).

Fathy et al.[83] assessed and compared the microtensile dentin bond strength (µTBS) of three universal adhesives immediately and after 5000 thermacycling. Aging negatively affected two UAs (Clearfill universal bond quick and REGEN) and one was not affected (Single bond).

Nanoleakage pattern

It was detected by Sano et al in 1995 when he first used silver nitrate as a tracer to show it. Its rise indicates the presence of water-rich areas and the irregular infiltration of resin into the demineralized collagen, resulting in an incomplete and porous hybrid layer.[84] The primary cause of nanoleakage are pores up to 50 nanometers which occur between hybrid layer and intact dentin even without the formation of actual gaps.[85] They are too small to allow bacterial passage however it's susceptible to bacterial enzymes and acidic products which leads to degradation. In this study SEM was used to detect nanoleakage. The main limitation with using SEM is the necessity of drying samples before scanning which may lead to dehydration and cracking of the specimens.

Each of the three modes of nanoleakage has a different indication. Spot-like pattern results from permeable regions in the hybrid layer as a result of reaction between diamine silver ions and hydrophilic resin components. Reticular mode indicates incomplete water removal at hybrid layer which leads to suboptimal polymerization. Water tree on the other side is demonstrated by a delicate branching channels of silver. Water existence in UA composition is important because it allows the ionization of acidic functional monomers to enable the SE bonding potential of the adhesive.[5]

Water's primary role in adhesives is not a solvent, hence called co-solvent. Its presence is mandatory in adhesives and can't be avoided however it shouldn't be added excessively,[86] however it re-expands the collagen fibrils which allowing the resin to copolymerize with dentin. Whether in one or two-step adhesives, water bases UAs tends to degrade over time which consequently increases nanoleakage.

Chen et al.[87] 2023 assessed the effect of 4-formylphenyl acrylate (FA) on bond strength over time using two UAs. Nanoleakage was tested immediately and after 10,000 thermocycles. In conclusion, aging had non-significant effect on nanoleakage after thermacycling.

Guo et al.[88] assessed nanoleakage of experimental resveratrol-doped adhesive adhesives immediately and after 10,000 thermacycling. It was investigated that experimental adhesives showed increased nanoleakage after aging.

Han et al.[89] compared nanoleakage in GPDM and 10-MDP based universal adhesives. The groups were divided into immediate and after aging which were subjected to 10,000 thermocycles. It was conducted that both MDP and GPDM based adheisves underwent higher nanoleakage after aging.

Wang et al.[90] assessed nanoleakage of four different universal adhesives in self-etch mode. Nanoleakage was assessed immediately and after 5000 thermocycles. Thermo-cycling adversely affected nanoleakage of two adhesives (Adper Easy One and Optoibond XTR), but had no significant influence on Clearfill SE and Scotchbond Universal.

Makishi et al.[91] investigated nanoleakage and microtensile bond strength of different UAs bonded to enamel and dentin. The specimens were divided into immediate and delayed (after 1 year with 10,000 thermocycles) groups. After aging, it was investigated that nanoleakage was not avoided in all tested groups.

Aging of universal adhesives:

To stimulate physiological aging of adhesive materials, specimens are stored in artificial saliva with pH = 7 and temperature is 37°C which simulate intraoral conditions. [92] Many factors seem to affect the results of thermocycling procedure, for example size of the specimen, dwell time, temperature, number of cycles and transfer time. [93] Regardless of these variations, they found out that the possible temperature gradient which is tolerated by patients is 5°-55° C. Moreover, it was accepted by ISO TS 11405 Technical Specification for testing of adhesion to tooth structure.[94] [95] Eliasson et al. [96] was suggested that cutting the specimen into beams prior to thermocycling is better as larger specimen need longer dwell time (>60 seconds.

Theoretically, thermocycling and prolonged water storage can weaken adhesive bonds.[97] However, despite the increasing clinical success and widespread use of UAs, there remains a scarcity of comprehensive research on how their bond strength is influenced by aging.[98] Results of the previous studies are controversial as some stated a significant decrease in bond strength[99, 100] while others found no significant difference. [31, 101, 102, 103, 104, 105, 106,107].

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