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Influence of Storage Temperature on the Bond Performance of Universal Adhesives: A Narrative Review

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Abstract

Background: Universal adhesives revolutionized restorative dentistry by enabling clinicians to employ a single material in self-etch, etch-and-rinse, or selective enamel-etch modes. However, their efficacy is dependent on the stability of whole formulations that incorporate water, volatile organic solvents, acidic functional monomers, hydrophilic resins and photoinitiators. Storage temperature can affect viscosity, solvent evaporation, monomer mobility, phase stability, and polymerization, which may influence the quality and durability of the resin–dentin contact.

Objective: This narrative review explored the influence of refrigerated storage, room-temperature storage, prolonged heat exposure, and short-term prewarming on the bonding performance of universal dental adhesives.

Results: Available evidence indicates that temperature effects are strongly formulation dependent. Refrigeration may reduce chemical deterioration and preserve volatile components, although adhesives used immediately after removal from cold storage may exhibit increased viscosity, reduced dentin wetting, and slower solvent evaporation. Controlled prewarming may improve flow, monomer diffusion, and solvent removal in selected formulations, whereas prolonged exposure to elevated temperatures may accelerate solvent loss and destabilize reactive components. Artificial aging decreases bond strength and increases interfacial permeability. Differences among studies are attributable to variations in solvent systems, functional monomers, water content, storage duration, application protocols, and testing methods.

Conclusion: No single storage temperature can be recommended for all universal adhesives. Clinicians should follow product-specific instructions, avoid prolonged thermal exposure, allow refrigerated materials to reach the recommended working temperature, and ensure adequate active application, air thinning, and light curing.

Keywords: universal adhesives; storage temperature; refrigeration; prewarming; solvent evaporation; bond strength; nanoleakage.

Introduction

Adhesive dentistry is transforming the modern practice of restorative dentistry, providing durable bonding of restorative materials to dental hard tissues, while adhering to the principles of minimally invasive dentistry.[1] Reliable adhesion is paramount for the long-term clinical success of direct and indirect restorations, enhancing restoration retention, minimizing microleakage and postoperative sensitivity and strengthening weakened tooth structure.[2] Universal adhesives, also known as multimode adhesives, are the most recent generation of adhesive systems. Compared to conventional adhesives they may be applied with etch-and-rinse, self-etch or selective enamel-etch approaches depending on the clinical situation.[3] This versatility is obtained with a complex formulation containing functional acidic monomers, resin monomers, organic solvents, water, photoinitiators and in some products silane coupling agents.[4]

Functional monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) have attracted much attention among these components, as they can induce micromechanical retention and persistent chemical bonding with hydroxyapatite, which helps improve the durability of the resin-dentin interface.[5] Performance of universal adhesives, however, depends not only on their chemical composition but also on proper clinical handling and storage conditions.[5,6] Universal adhesives have high susceptibility to environmental factors before clinical use due to the presence of volatile solvents and chemically reactive monomers in one formula. One of the most important variables that influence physicochemical properties of these materials is temperature. Changes in storage temperature might affect adhesive viscosity, solvent evaporation, monomer mobility, phase stability and the kinetics of polymerization, which will finally affect resin infiltration, hybrid-layer formation, bond strength and the long-term durability of the interfacial zone.[7-9] Moreover, repeated bottle opening and prolonged storage under less-than-ideal conditions may accelerate solvent loss and alter the original composition of the adhesive, potentially affecting its clinical performance. Several studies have assessed the influence of refrigeration, room temperature storage, elevated temperature exposure and prewarming on the bonding effectiveness of universal adhesives. However, the findings reported so far are still

inconsistent. Moderate heating has been shown to improve adhesive flow and solvent evaporation in some studies, while other studies have shown little benefit or even degradation of bond performance from thermal exposure.[1012] Similarly, refrigeration has been reported to preserve material stability for some formulations but to increase viscosity and adversely affect dentin wetting when adhesives are used
immediately after removal from cold storage.[13,14]

These contradictory results show that the temperature response is highly dependent on the material and controlled by parameters such as solvent composition, hydrophilicity, functional monomer chemistry, and storage time. Considering the increasing clinical use of universal adhesives and the absence of a clear consensus on the ideal storage conditions, a comprehensive assessment of the available evidence is justified. The aim of the present narrative review is to critically summarize the current knowledge about the impact of storage temperature on the physicochemical properties and bonding performance of universal dental adhesives, regarding solvent behavior, bond strength, nanoleakage and the durability of the resin-dentin interface. The review also discusses the clinical implications of adhesive storage and handling to provide evidence-based recommendations for optimizing the adhesive performance in the daily dental practice.

1. Universal Adhesives: Composition

Universal adhesives, also known as multimode or multipurpose adhesives, have been used in clinical practice since 2011. These adhesives have been recently called eighthgeneration adhesives, based on the historical perspective of dental adhesives.[15] Their primary objective was to simplify chairside procedures while maintaining the clinical efficacy of restorations that rely on the formation of a robust hybrid layer. These adhesives are designed for high versatility, allowing adaptation to various clinical scenarios and bonding strategies.[16] Their

formulation is carefully balanced to function across different substrates and moisture conditions.[17]

1.1 Resin monomers

The polymeric matrix is mainly formed by cross linking methacrylate monomers such as bisphenol A glycidyl methacrylate (Bis-GMA), urethane dimethacrylate (UDMA) and triethylene glycol dimethacrylate (TEGDMA).[6] After light activation these monomers polymerize and form the structural framework of the adhesive layer and create a chemical bond with the overlying resin composite restoration.[6] Disadvantage of high viscosity of high-molecular-weight monomers like BisGMA that give mechanical strength. Thus, lower viscosity diluent monomers are added to improve handling characteristics and to facilitate penetration into conditioned dentin.

1.2 Fillers

Some universal adhesives contain a small amount of inorganic nanofillers that reinforce the polymerized adhesive layer, increase the film thickness and improve the distribution of stress at the resin-dentin interface. Fillers can improve elastic modulus and resistance to polymerization and functional stresses when well dispersed in the resin matrix.[6] However, higher filler loading can increase the viscosity and decrease the monomer penetration into dentin.[18] Thicker adhesive layers may also influence adaptation in narrow cavities or hinder complete solvent removal. Therefore, the effect of fillers is determined by the particle size, concentration, surface treatment and compatibility with the resin matrix and not simply by the presence of fillers.

1.3 Photoinitiators and stabilizers

Universal adhesives contain photoinitiator systems which produce free radicals and initiate polymerization after activation by light. The most common system is camphorquinone (CQ) and a tertiary amine co-initiator. This combination is effective in the wavelength range of blue light used in modern curing units. The acidic and hydrophilic nature of universal adhesives, however, can decrease the efficiency of conventional amine-based initiation systems, especially in the presence of residual water or solvent within the adhesive layer.[19] Some of the more recent formulations thus have included other initiators, such as acylphosphine oxides, which are better suited for polymerization under acidic circumstances and minimize the yellow color of CQ.[6] The performance of these systems is dependent on the spectral

output of the curing light, initiator concentration, adhesive acidity and solvent content. Inhibitors and stabilizers are also added to prevent premature polymerization in storage and to prolong shelf life.[9,20] They can also degrade if exposed to heat or light over a long period.

1.4 Silane coupling agent.

Some universal adhesives have added silane to improve adhesion to resin composites and silica-based ceramics fillers. Silane molecules contain a silanol-reactive end that reacts with silica-containing surfaces and a methacrylate end that copolymerizes with resin cement or composite.[21] Theoretically this allows a universal adhesive to be a tooth adhesive and a ceramic primer simultaneously. However, the long-term stability of silane in acidic, water-containing universal adhesives remains to be questioned. Acidic pH and water can lead to premature silane hydrolysis and condensation, which can reduce its reactivity before clinical use. Yoshihara et al.[22] and Yao et al.[21] observed that silane in universal adhesives could be less effective and less stable than a silane primer applied separately. Melo et al.[23] also found that separate silanization can provide more reliable bonding to silica-based ceramics. Therefore, while the presence of built-in silane enhances clinical convenience, a

dedicated silane primer is still often recommended for bonding.

1.5 Functional monomers

Universal adhesives are characterized by the presence of acidic functional monomers which provide micromechanical and chemical adhesion. The monomers simultaneously condition the hard dental tissues and chemically interact with the remaining hydroxyapatite surrounding the collagen fibrils. Among them 10-MDP is the most successful and well-studied functional monomer. The phosphate group of 10-MDP reacts with calcium ions to form stable MDP-calcium salts that are resistant to hydrolysis and can form organized nano-layering at the adhesive interface. This chemical interaction complements micromechanical interlocking and is a major factor in the longevity of the bond.[5,24,25]

Other phosphate-containing monomers include glycerol phosphate dimethacrylate (GPDM) and dipentaerythritol pentaacrylate phosphate (PENTA) and are used in some commercial products. While these monomers are responsible for

chemical adhesion, their calcium salts are less stable than 10-MDP calcium salts, and thus, more susceptible to hydrolytic degradation and loss of bond strength over time.[26] Carboxylic acid monomers, such as 4-META and Phenyl-P, have also been used in adhesive formulations; however, the available evidence indicates that phosphate-based monomers generally have better long-term chemical stability.

1.6 HEMA-containing and HEMA-free formulations

Universal adhesives often contain 2-hydroxyethyl methacrylate (HEMA), a hydrophilic resin monomer that improves dentin wetting, allows resin incorporation into the collagen network, and reduces phase separation between hydrophilic and hydrophobic components. These qualities improve handling and help creation of hybrid layers especially in simple one bottle systems. HEMA is also one of the key drawbacks of simpler adhesives as it is extremely attracted to water. Increased water sorption may increase permeability of the polymerized adhesive layer leading to polymer swelling, plasticization, hydrolytic degradation and degeneration of the resin-dentin interface.[5] Furthermore, HEMA has been shown to correlate with increased cytotoxicity and lower long-term mechanical stability.[5,27] The development of HEMA-free universal adhesives overcomes these constraints by replacing this monomer with more stable alternatives.[28] The replacement of HEMA by dimethacrylate monomer, e.g. glycerol dimethacrylate (GDMA), increases the crosslinking of the polymer network and thus decreases water sorption and improves hydrolytic stability in the oral environment.[29] Newer acrylamide-based monomers such as methacrylamide, hydroxyethyl acrylamide (HEAA), and diethyl acrylamide (DEAA) show less water uptake, less hydrolytic breakdown, and more durable bonds over time.[30]

1.7 Solvent systems

Organic solvents are also important components, as they reduce the adhesive viscosity, deliver the resin monomers to the demineralized dentin matrix and allow evaporation of the residual moisture before polymerization. The most common solvents are water, ethanol, acetone and, more recently, isopropanol, each with specific physicochemical properties.[31] The ionization of acidic monomers requires the presence of water and the highest vapour pressure of acetone leads to rapid evaporation, so the adhesive performance is extremely sensitive to evaporation during storage and during clinical use.[9,32] Ethanol is less technique sensitive than

acetone, has better collagen reexpansion and evaporates more slowly. Isopropanol is intermediately volatile and has recently attracted attention due to its superior formulation stability and controlled evaporation behavior.[6]

2. Shelf Storage Temperature: Importance and Implications

Previous literatures reported that immediate resin–dentin bond strengths as well as hybrid layer formation can be affected by the shelf temperature of the adhesive.[13,14,33,34] whereas other studies have reported that the temperature of the adhesive before it is used does not significantly affect the bond strength.[10,33] Manufacturers usually advise keeping them between 2 °C and 25–28 °C, with a typical shelf life of about two years.[9,35] This recommendation reflects the balance needed to preserve the chemical stability of solvents, monomers, and initiators. It could be useful to use an ethanol/water-based adhesive at 37°C or 50°C and an acetone-based adhesive at 37°C to improve adhesive performance.[11] Once storage conditions drift outside this range (during transport in hot climates or prolonged storage in clinics) the integrity of the adhesive begins to suffer. For instance, elevated temperature conditions could lead to hydrolytic degradation, particularly in adhesives containing hydrophilic monomers like HEMA or acidic functional monomers such as 10-MDP.[24,36] This degradation breaks critical bonds in the adhesive polymer structure, resulting in reduced bond strength, increased nanoleakage at the resin-dentin interface, and phase separation of the adhesive components.[20,37] On the contrary, adhesives free from HEMA and incorporating methacrylamides tend to be more stable under suboptimal storage condition.[9] For example, a HEMA-free adhesive, demonstrated minimal changes in nanoleakage even after expiration in comparison to HEMA containing.[24]

Another significant issue related to improper storage temperature is the evaporation of solvents, such as acetone or ethanol, which are essential for dissolving monomers and ensuring proper substrate wetting.[12] High temperatures accelerate solvent loss, leading to increased viscosity of the adhesive.[38] This thicker consistency hampers the adhesive ability to penetrate dentin effectively, compromising the formation of a durable hybrid layer.[39] So, adhesives exposed to high temperatures might exhibit reduced bond strength, increased susceptibility to enzymatic degradation (due to MMP activation), and a higher risk of restoration failure.[40,41] Additionally, the stability of photoinitiators (CQ) can be negatively affected by heat, especially in acidic adhesive formulations.[19] Moreover, high

temperatures may trigger neutralization reactions between acidic monomers and amine co-initiators, reducing polymerization efficiency and weakening the adhesive's performance.[42] Some studies suggest that alternative photoinitiators, like TPO, may offer better stability under adverse storage conditions.[43]

a. Storage in Refrigeration (≈4 °C)

Refrigeration has been proposed as a means to extend adhesive shelf life by slowing solvent evaporation and chemical degradation of functional monomers like 10-MDP.[133] Some studies report that refrigerated adhesives maintain bond strength longer than those stored at room temperature,[44] although increased viscosity at lower temperatures can reduce wettability and complicate solvent evaporation.[13,45-47] Consequently, incomplete polymerization and compromised hybrid layer formation may result.[48,49] Reports on clinical outcomes are mixed: while some studies indicate reduced bond strength under refrigeration,[34,46,50] others found no significant differences compared to room-temperature storage.[13,51]

b. Storage at Room Temperature (≈20–25 °C)

Room temperature storage reflects typical clinical conditions. Within manufacturer-recommended timeframes, most studies report stable bond strength and nanoleakage performance.[52,53] However, prolonged storage at ambient conditions may accelerate solvent evaporation and monomer degradation, resulting in gradual reductions in bond strength. Stability therefore depends on both adhesive composition and duration of storage.

c. Storage at 25 °C

Although close to "room temperature," studies that have specifically examined controlled storage at 25 °C show divergent findings. Some indicate that when adhesives are held at 25 °C for up to 6 months, bond strength remains stable and nanoleakage is at acceptable levels.[54] Others, however, experienced early deterioration of the adhesive effectiveness under the same conditions as a consequence of faster water sorption and solvent loss.[55] The differences emphasize the necessity of distinguishing between nominal "room temperature" storage and controlled 25 °C conditions.

d. Elevated Storage at 40 °C

The adhesive is often said to perform less well when stored at 40 °C. Donmez et al.[56] stated that storage at 4 °C and 23 °C had a slight influence on the bond strength, but a year of storage at 40

°C significantly decreased it. Increased temperatures promote time-dependent hydrolysis in moisture-laden adhesives, resulting in decreased binding strength and increased nanoleakage.[33] The inconsistent results on adhesive storage and temperature are undoubtedly caused by many factors, such as differences in adhesive composition (e.g. ethanol- vs acetonebased solvents), packing, testing procedures and assessment methods.

3. **Pre-Warming Adhesives: Pros and Cons**

Loguercio et al.[11] evaluated the effect of adhesive temperature on bond strength and reported that increasing the temperature of Single Bond to 37 °C or 50 °C enhanced resin–dentin bonding.[47,50] Controlled heating reduces viscosity, improves monomer infiltration into dentin, and promotes more uniform hybrid layer formation.[8,47,57] It also accelerates solvent evaporation, minimizing the risk of residual solvent entrapment within the adhesive layer.[47] Pre-warming has also been associated with a higher degree of conversion, which may contribute to improved mechanical stability of the adhesive interface.[58] Nevertheless, the benefits of pre-warming depend on maintaining physiological temperature ranges. When heating is excessive (>50 °C), premature solvent loss and destabilization of heat-sensitive components occur.[47] Prolonged exposure to elevated temperatures has additionally been linked to photoinitiator degradation, increased solvent evaporation, and shortened shelf life, ultimately compromising bond strength and increasing nanoleakage. Despite these potential benefits, prewarming also has drawbacks. The prolonged heating could lead to an excessive evaporation of solvent before clinical use, increasing the viscosity of the adhesive and limiting the penetration into dentin.[59] Moreover, elevated temperatures may affect the stability of some adhesive components, especially photoinitiator and other heat sensitive materials, resulting in decreased degrees of conversion and degraded long-term bond durability.[60] Moreover, prewarming does not affect all types of adhesives in the same way. Heating can be beneficial for ethanol-based adhesives, but acetone-based solutions, which are more volatile, frequently exhibit only modest improvements or a decrease in performance.[55] Finally, practical adoption is hindered by the absence of standardized protocols, as variations in heating devices, duration and temperature control lead to variable results and have led to conflicting findings in the literature.[61]

4. **Manufacturer Recommendations vs. Clinical Practice.**

Universal adhesives are marketed as simplified bonding systems, with manufacturers promoting short application times. These protocols are primarily designed to facilitate convenience in clinical settings. However, accumulating evidence demonstrates that such minimal procedures may not guarantee complete solvent evaporation, adequate monomer infiltration, or optimal polymerization.[25,62] This discrepancy is particularly relevant in laboratory evaluations focusing on μTBS and nanoleakage. Manufacturer instructions often recommend application times ranging between 10–20 seconds, followed by gentle air-drying. Nevertheless, several in vitro and vivo studies indicate that extending the application to ≥20 seconds, employing active rubbing during placement, and using more vigorous air-drying significantly improve dentin infiltration, solvent evaporation, and hybrid layer formation. These adjustments have been directly associated with increased μTBS values and reduced nanoleakage.[3,63] Additionally, adhesives containing more volatile solvents, such as acetone, are especially sensitive to incomplete evaporation. In such cases, the literature emphasizes the need for intensive air-drying to avoid entrapment of residual solvent and water, which can negatively influence both μTBS and nanoleakage.[62,64,65]

5. **Bond Strength Testing of Universal Adhesives**

The performance of adhesive systems is frequently assessed using laboratory bond strength tests, which provide valuable insights into the adhesion of resin materials to dental substrates. Although these tests cannot perfectly replicate intraoral conditions, they remain the most accepted means of comparing different adhesives and predicting their clinical behavior.[66,67] The most commonly used tests are the microtensile bond strength (μTBS) test and the shear bond strength test, with other variations such as macro-tensile, push-out, and pull-out tests being less frequently applied. In contrast, the shear bond strength test is a simpler method in which a composite cylinder bonded to tooth substrate is subjected to shear forces parallel to the adhesive interface until debonding occurs.

Despite the availability of these alternative methods, μTBS testing is often preferred for dentin adhesion studies due to its higher sensitivity and versatility. This technique allows for the use of small, uniformly shaped specimens, typically less than 1 mm^2 in cross-sectional area, which minimizes stress distribution discrepancies and enables more accurate measurement of true interfacial bond strength. Additionally, μTBS testing permits the

evaluation of multiple specimens from a single tooth, increasing statistical power.[68-71] The principal studies evaluating the effect of storage or application temperature on adhesive bond strength are summarized in **Table 1**. Taken together, these studies suggest that the effect of temperature is not uniform among adhesive systems. In certain materials, refrigeration preserved bond strength; in others, refrigeration limited or had no effect on performance.

Table 1. Summary of studies evaluating the influence of storage or application temperature on the bond strength of dental adhesive systems.

Study	Temperature-related condition	Bond-strength method/outcome	Main interpretation
Spohr et al.[13]	Refrigeration	Tensile bond strength	Refrigeration produced material-dependent effects
Sadr et al.[7]	Storage time and temperature	Properties and bond performance	Both duration and temperature influenced some systems
Alexandre et al.[34]	Adhesive temperature	Enamel bond strength	Temperature effect depended on the adhesive
Reis et al.[10]	Cold/ambient/warmed adhesive; immediate and 6 months	Dentin bond strength	Thermal effects could persist after aging
Donmez et al.[56]	Refrigerated, room, and elevated storage	Bond strength to pulpchamber dentin	Prolonged elevated temperature was more detrimental
Loguercio et al.[11]	Prewarming	Resin–dentin bond strength and adhesive properties	Moderate warming benefited selected systems
Cardoso et al.[35]	Shelf-life simulation	Dentin bond strength	Shelf aging affected materials differently
Pongprueksa et al.[12]	Evaporation/repeated use	Universal adhesive shelf performance	Solvent loss altered adhesive performance
Sharafeddin et al.[50]	Different application temperatures	Shear bond strength	Temperature response was product dependent
Cuevas-Suárez et al.[9]	Shelf-life simulation	Bond strength, conversion, nanoleakage	Universal adhesives showed formulation-dependent deterioration
Mazzitelli et al.[20]	Shelf aging	Dentin bond performance	Storage history influenced delayed bonding behavior

6. **Nanoleakage and Aging of Adhesive Interfaces**

Nanoleakage refers to the penetration of ions, fluids, or tracer molecules—such as silver nitrate into nanometer-sized channels within the resin–dentin hybrid layer.[72] Unlike microleakage, which occurs at the external margins of restorations, nanoleakage develops internally within the adhesive interface. This phenomenon is primarily attributed to incomplete resin infiltration, which leaves voids around the exposed collagen fibrils and compromises the structural integrity of the hybrid layer. [72] Several factors have been identified as contributing to

nanoleakage. One common cause is the retention of water or solvents within the adhesive layer, which can interfere with the polymerization process and lead to the formation of voids or porosities.[73] Additionally, phase separation between the hydrophilic and hydrophobic components of some adhesive systems can result in the development of nanometerscale channels that allow fluid penetration.[74]

Another significant factor is the collapse of the collagen network which can occur during air drying or owing to poor wet bonding procedures.[75] The collapse of collagen fibrils makes resin monomer infiltration into the demineralized dentin inappropriate, increasing the potential of nanoleakage.[75] These mechanisms, together, decrease the endurance of the adhesive contact and may contribute to the long-term breakdown of resindentin bonds. Clinically, this process is substantially associated with lower bond strength and higher chance of restoration failure over time.[75] Several strategies have been offered to counteract these effects. These are solvent evaporation prior to polymerization, ethanol-wet bonding strategies to enhance resin penetration, and matrix metalloproteinase (MMP) inhibitors to reduce enzymatic collagen breakdown. Also, the development of adhesives with optimized hydrophilicity seeks to minimize phase separation and water sorption, decreasing the danger of nanoleakage and improving the lifetime.[73,74]

Nanoleakage evaluation is commonly performed using a variety of analytical techniques to visualize and characterize leakage pathways within the resin–dentin interface. One widely used method is tracer penetration, particularly with ammoniacal silver nitrate staining, followed by visualization under scanning or transmission electron microscopy (SEM/TEM) using backscattered electron imaging to detect silver deposits in nanochannels.[72] Another powerful approach is confocal laser scanning microscopy (CLSM), which employs fluorescent dyes to trace fluid movement and identify leakage pathways in three dimensions with optical sectioning capability.[75] Additionally, micro-Raman spectroscopy provides valuable chemical insights by identifying areas of incomplete resin infiltration and detecting changes in the molecular composition of the adhesive interface, further contributing to a detailed understanding of nanoleakage phenomena.[73]

There is scarce evidence of the relationship between storage temperature and nanoleakage in comparison with bond strength tests. However, Cuevas-Suárez et al.[9] observed that shelf-life simulation influenced the nanoleakage, degree of conversion and bonding performance of universal adhesives in a material dependent manner. Their results suggest that the storage-induced deterioration is not uniform across products and is dependent on the overall adhesive formulation. Likewise, Mazzitelli et al.[20] observed formulation-dependent alterations in dentin bonding during shelf ageing, which corroborates the hypothesis that the stability of solvent and monomer before to application may affect the subsequent interfacial integrity. Sadr et al.[7] have previously demonstrated that storage duration and temperature influence the parameters of self-etch systems, although nanoleakage was not the predominant effect.

7. **Factors Influencing Long-Term Bond Stability**

Adhesive systems require hydrophilic monomers such as HEMA to ensure adequate wetting and penetration into moist dentin. However, this hydrophilicity renders the polymerized adhesive network permeable to water, acting like a semipermeable membrane. As a result, continuous water diffusion leads to nanoleakage and hydrolytic breakdown of the hybrid layer, significantly affecting bond durability.[36,59,76] Incomplete solvent evaporation (water, ethanol, or acetone) and oxygen inhibition compromise the degree of conversion of adhesive monomers. Residual solvents and unreacted monomers plasticize the resin matrix, reduce cross-linking density, and accelerate hydrolytic degradation.

This weakens the hybrid layer and increases its susceptibility to nanoleakage and collagen degradation.[75-77] These processes result in reduced bond strength, increased nanoleakage, and compromised restoration performance over time. There are many internal and external elements that make resin-dentin adhesive interactions less stable over time. Hydrolytic breakdown of methacrylate resins is one of the main ways this happens. This process involves breaking down ester bonds in the resin matrix with water, which causes the polymer to break down, the structure to degrade, and the mechanical qualities to slowly get worse.[76] Enzymatic degradation is also very important.

MMPs and cysteine cathepsins are two types of enzymes that can arise from the host. They can become active during the bonding process or when acidic monomers get into the host. These enzymes break down collagen fibrils that are exposed to

and have lost minerals in the hybrid layer, making the adhesive contact even less stable.[40,74] In addition, the adhesive interface is always under mechanical and temperature stress in the mouth. Repeated chewing and changes in temperature cause microcracks to grow and the bonded contact to become weaker with time.[5,78,79] These tensions speed up the damage that happens at the interface over time. These aging processes all work together to weaken the bond strength, promote nanoleakage, and, in the end, make adhesive restorations less effective in clinical settings. To make adhesive-based dental treatments last longer and be more predictable, it is important to understand and stop these degradation mechanisms.

Water storage is one of the most used in vitro methods to simulate the aging of dental adhesive systems. In this process, specimens are immersed in distilled water or artificial saliva for varying durations—ranging from a few days to several years— to replicate the moist conditions found in the oral environment.[52] This method is valued for its simplicity and ability to provide insight into the long-term stability of resin–dentin bonds. Recent studies have shown that adhesives containing hydrophilic monomers like HEMA exhibit significant reductions in bond strength after prolonged water storage. In contrast, HEMA-free adhesives tend to retain better mechanical properties over time, highlighting how formulation plays a critical role in resistance to hydrolytic degradation.[80] When stored in water, dental adhesives undergo several degradation processes. One major concern is hydrolytic degradation, where water infiltrates the adhesive interface and disrupts the polymer matrix. This can result in plasticization of the polymers, making the material softer and more prone to mechanical failure. Additionally, unreacted monomers and solvents can leach out over time, further weakening the adhesive bond.[81] Water exposure also affects the collagen network within dentin, leading to enzymatic breakdown of collagen fibrils if the hybrid layer is not well-protected. Recent literature emphasizes that water trapped within the hybrid layer can both interfere with proper resin infiltration and promote long-term degradation of the interface, especially in adhesives with high water sorption and solubility.[82] Despite its widespread use, water storage has several limitations. It does not fully replicate the dynamic and complex nature of the oral cavity, where materials are constantly exposed to fluctuations in temperature, pH, enzymatic activity, and mechanical stresses. These variables can interact synergistically, accelerating the degradation of adhesive interfaces in ways that water storage alone cannot

capture. Studies have shown that the inclusion of thermal cycling, pH cycling, and enzymatic exposure can significantly alter the durability of adhesive systems, offering a more realistic simulation of clinical aging conditions.[83]

Thermocycling is a laboratory-based aging method wherein dental samples are alternately exposed to cycles of cold and hot baths—typically between 5 °C and 55 °C—for thousands of repetitions.[84] This technique is designed to simulate the temperature fluctuations that occur within the oral environment during daily activities like consuming cold drinks and hot meals. Integrating thermocycling into artificial aging protocols is increasingly recognized as essential for accurately developing and evaluating the durability of novel dental composites in vitro.[84] The alternating thermal stress induces repeated expansion and contraction at the resin–dentin interface, leading to mechanical fatigue and potential microcrack formation. Over time, these microstructural changes facilitate water penetration, exacerbate nanoleakage, and accelerate hydrolytic degradation of the hybrid layer. Although this process recapitulates some oral stressors, it remains a simplified model—lacking enzymatic, bacterial, and mechanical load factors that also contribute to bond degradation in vivo. Contemporary studies have consistently highlighted the detrimental effects of thermocycling, particularly when combined with mechanical loading, on the durability and strength of adhesive bonds.[85]

8. **Summary and Rationale for the Current Study**

The durability of resin–dentin bonding remains one of the greatest challenges in restorative dentistry.[39,74,77,86] Although adhesive systems have evolved considerably, their long-term clinical performance is still compromised by hydrolytic and enzymatic degradation, incomplete polymerization, and susceptibility to nanoleakage.[85,87] Numerous laboratory studies have highlighted the influence of adhesive composition, solvent type, and application protocols on bond strength and stability. However, there is a growing recognition that external factors, such as storage temperature, shelf life, and pre-warming of adhesives**,** also play a critical role in determining the effectiveness of resin–dentin adhesion.

9. **Future Perspectives**

Future work should include standardized storage, ageing, and bond testing techniques to allow for better comparability between investigations. More emphasis should be paid to universal adhesives without HEMA based on methacrylamide, isopropanol

and tert-butanol. Studies should also distinguish between long-term storage and short-term prewarming, and correlate bond strength testing with nanoleakage, degree of conversion, and advanced imaging. Long-term clinical trials are required to find out if laboratory findings transfer into improved repair durability.

10. Conclusion

- Storage temperature can alter the viscosity, solvent evaporation, monomer mobility, polymerization, and interfacial quality of universal adhesives.
- Its effect is formulation dependent; therefore, no single storage temperature is suitable for all products.
- Refrigeration may preserve chemical stability but can impair handling if the adhesive is used while still cold.
- Controlled prewarming may improve flow and solvent removal in selected materials, whereas prolonged heat exposure may accelerate solvent loss and reduce stability.
- Long-term durability also depends on residual solvent, water sorption, hydrolytic and enzymatic degradation, and nanoleakage.
- Clinicians should follow manufacturer-specific storage instructions, avoid excessive heat exposure, allow refrigerated adhesives to reach the recommended working temperature, ensure active application, adequate air thinning, and sufficient light curing.
- Further standardized laboratory and clinical studies are needed to establish product-specific storage recommendations.

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