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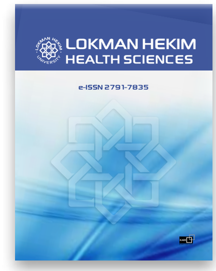

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REVIEW

Microtensile Bond Strength of Restorative Materials to Enamel and Dentin: A Narrative Review

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Abstract

The foundation of modern adhesive dentistry was laid by Buonocore's introduction of the acid-etch technique in 1955 and Bowen's development of composite resins in 1962. Today, adhesive restorative materials are widely used, offering reliable bonding to enamel. Achieving long-lasting adhesion to dentin is still challenging because of its variable structure and the presence of the smear layer. The constant evolution of adhesive systems introduces numerous products with varying compositions and clinical protocols, necessitating thorough performance evaluation. *In vivo* studies are limited by ethical and technical constraints, making *in vitro* bond strength tests a more practical alternative. While laboratory studies cannot fully replicate the oral environment, they allow for rapid, cost-effective comparisons. Various testing techniques – macroshear, macro push-out, macrotensile, microshear, micro push-out, and microtensile – are employed to assess adhesive performance, with outcomes influenced by specimen preparation, substrate variability, and environmental conditions such as thermocycling. Among these, microtensile bond strength (μTBS) has gained prominence due to its high sensitivity and ability to evaluate small bonding interfaces. It also facilitates the study of bonding behavior at different dentin depths and under localized conditions. However, translating laboratory findings into clinical success remains challenging. This review critically discusses the methodology, limitations, and clinical relevance of μTBS in adhesive dentistry.

Keywords: Adhesive systems; Enamel and dentin adhesion; Microtensile bond strength; Resin–dentin interface

Modern adhesive dentistry began in 1955 with Buonocore's acid etching technique, followed by Bowen's development of composite resins in 1962.[1] Buonocore, Gwinnett, and Matsui showed that acid etching created microporosities in enamel, allowing resin infiltration and micromechanical bonding via resin tags.[2] However, dentin bonding presents greater challenges due to its heterogeneous structure, hydrophilic properties, and smear layer. In addition, adhesion strength differs across superficial, middle, and deep dentin zones. An effective adhesive should reliably bond to both enamel and dentin, even though they differ markedly in composition.[3]

The success of esthetic and functional restorations depends on selecting suitable adhesive systems. As minimally invasive techniques gain popularity, the clinical use of direct composite restorations has expanded in parallel with advances in adhesion technology.

This review first outlines the development and classification of adhesive systems, then focuses on the μTBS. μTBS is a widely accepted technique for evaluating bond strength,

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offering benefits such as multiple specimen retrieval from one tooth and regional strength analysis.[4] The review details the application, advantages, limitations, and influencing variables of this test based on current literature.

The aim of this study was to identify and analyze the key methodological factors influencing µTBS outcomes. Despite numerous laboratory investigations, the lack of standardized methodologies limits comparability across µTBS studies.

A non-systematic literature search was conducted in PubMed, Scopus, and Web of Science using the keywords "microtensile bond strength," "adhesive systems," and "enamel-dentin bonding."

Development and Classification of Adhesive Systems

Long-term clinical success depends heavily on the adhesive technology used. Over decades of research, improvements in adhesive performance have significantly promoted minimally invasive resin-based restorations. Adhesive systems are classified by generation, as summarized in Table 1.

Fourth Generation

Developed in the 1980s–1990s, these systems employ a total-etch (TE) technique, removing the smear layer completely. They involve three steps: Acid etching, primer, and adhesive application. A hydrophilic primer applied to moist dentin forms a hybrid layer, yielding high bond strength. Although still considered a gold standard, the multi-step protocol has led to the pursuit of simpler alternatives.[5]

Fifth Generation

Introduced in the 1990s, these simplified systems combine primer and adhesive in a single bottle but maintain the TE approach. While they achieve high bond strength through a hybrid layer and resin tag formation, they are more sensitive to moisture and may be incompatible with some dual-/self-cure composites.[5]

Sixth Generation

Emerging in the late 1990s, these self-etch (SE) adhesives incorporate an acidic primer, eliminating separate etching. They are less moisture-sensitive but show weaker enamel adhesion, requiring selective-etch (SEE). Over-demineralization of dentin can reduce bond strength.[5]

Seventh Generation

These all-in-one systems combine etchant, primer, and adhesive in a single solution.[5] Although user-friendly, they are highly hydrophilic. This makes them prone to moisture uptake, hydrolytic degradation, and limited resin infiltration.[5,6] Their inability to achieve hydrophobicity after polymerization and the risk of phase separation can compromise bonding, particularly to dentin. Consequently, bond strengths are generally lower than those of previous generations.[7]

Universal (Multimode) Adhesives

Designed to overcome previous limitations, universal adhesives (UA) can be used in TE, SE, or SEE modes. The TE approach is technique-sensitive; over-drying can collapse the collagen matrix and hinder monomer infiltration,[8,9] increasing the risk of post-operative sensitivity.[10] SE techniques reduce technique sensitivity but often underperform on enamel due to low pH.[8,11] SEE offers a balance but demands precise isolation to prevent dentin over-etching.[12]

UAs are designed to work in diverse clinical situations and can be used in both direct and indirect restorations. They are compatible with self-, light-, and dual-cure composites and a variety of substrates, including zirconia, porcelain, and metal.[13] Properly applied, they deliver adequate bonding to enamel and dentin.[14]

Table 1. Summary of adhesive generations and corresponding bond strength ranges

Generation	Clinical application techniques	Steps	Bond strength (MPa)
First	Resin	Two	1–3
Second	Phosphate ester+ Resin	Two	4–6
Third	Acid+Primer+ Adhesive	Three	3–8
Fourth	ER	Three	18–30
Fifth	ER	Two	17–25
Sixth	SE	Two	13–25
Seventh	SE	One	12–30
Eighth (Universal)	SE		
SEE			
ER	One		
Two			
Two	22–37		

SE: Self-etch; ER: Etch-and-rinse; SEE: Selective-etch.

While adhesive efficacy is evaluated through clinical and *in vitro* studies, the latter are more common due to time, cost, and ethical concerns. However, *in vitro* results may not fully replicate oral conditions.[15] Thus, both study types should be used complementarily, with laboratory data supporting clinical validation. Importantly, success criteria differ between these two approaches.

Clinical Evaluation and Success Criteria

Cvar and Ryge introduced the United States Public Health Service (USPHS) criteria in 1971 to assess restorations clinically.[16] These include color match, marginal discoloration, anatomical form, marginal adaptation, and secondary caries. Outcomes are classified as Alpha (ideal), Bravo (clinically acceptable), Charlie (needs replacement), or Delta (requires immediate replacement).[17]

As these criteria were not designed to detect minor changes in marginal adaptation or discoloration, the Modified USPHS system was later developed. It includes additional parameters such as surface roughness, tooth integrity, retention, and post-operative sensitivity.[17]

Laboratory Evaluation of Bond Strength

In vitro bond strength tests are widely utilized in laboratory evaluations due to several advantages over clinical studies. These advantages include rapid data collection, control over isolated variables, and ease of implementation. They also allow comparisons between standard and experimental materials, the inclusion of multiple test groups in a single study, and lower costs.[18,19]

For reliable outcomes, careful equipment selection, proper calibration, and minimization of confounding variables are essential. In addition, repeatability, consistency within acceptable variability limits, and procedures that simulate oral conditions enhance the validity of laboratory results.[19] In clinical conditions, multiple aging factors act simultaneously. Laboratory studies should mimic this complexity through combined protocols such as enzymatic degradation, thermal cycling, and mechanical loading. Without such simulations, *in vitro* bond strength data may not reflect clinical reality.

Bond strength tests measure the resistance of tooth-material interfaces to shear, tensile, or push-out forces. Based on bonding area, tests are categorized as macro (4–28 mm^2) or micro (approximately 1 mm^2). The most commonly used device for these evaluations is the Instron universal testing machine (UTM) (Norwood, MA, USA).[19]

Macroshear Bond Strength

This test evaluates the shear resistance of dental materials on hard tissues using a bonded surface area >3 mm^2. A composite resin is applied via a cylindrical mold (2.5–3 mm diameter) and polymerized. A shear force is then applied using a chisel blade in a UTM.

Its advantages include ease of setup, minimal equipment, and frequent use. However, uneven stress distribution and concentration at the margins compromise test reliability.[20]

Macro Push-Out Bond Strength

It is used to assess resistance to dislodgement under axial loading, especially in root canals with posts or resin cements. It better simulates clinical conditions in endodontic treatments, but requires a more complex protocol.[20]

Macrotensile Bond Strength

This test measures tensile strength using larger adhesive surfaces (>3 mm^2), typically with cone-shaped specimens. While it replicates tensile loading well, the difficulty in achieving uniform adhesive interfaces undermines reproducibility and reliability.[20]

Microshear Bond Strength

Small composite cylinders are bonded to enamel or dentin, and shear force is applied using a thin wire loop. The small bonding area ensures a more homogeneous stress distribution and allows multiple specimens from one tooth. Nevertheless, slippage of the wire can lead to inaccurate measurements, necessitating a precise technique.[20]

Micro Push-Out Bond Strength

This test involves bonding micro-sized posts in 0.7–1 mm-thick root slices, followed by axial loading to evaluate push-out resistance. It also allows analysis of dentinal tubule orientation, enhancing understanding of the bond mechanism.[20]

Microtensile Bond Strength

The μTBS applies tensile stress on bonded interfaces. A composite block is built on the tooth surface, from which multiple stick-shaped specimens (~1 mm^2) are sectioned. Each is mounted and subjected to tensile force via UTM.[21]

μTBS offers high precision due to homogeneous stress distribution and the ability to assess multiple regions of a single specimen. It also evaluates localized

Table 2. Comparison of different bond strength test techniques

Test type	Surface area	Application type	Stress type	Sensitivity	Bond strength (MPa)	Reliability	Area of use
Macroshear	>3 mm^2	Cutting	Heterogeneous	Medium	Low	Medium	It is used in general restorative materials and is easier to apply.
Macro push-out	>3 mm^2	Pushing	Heterogeneous	Medium	High	Very high	It is used in post and endodontic applications with different test geometries.
Macrotensile	>3 mm^2	Pulling	Heterogeneous	Medium	Low	Low	It is the test technique used to measure the bond strength of restorative materials to tooth surfaces.
Microshear	<1 mm^2	Cutting	Homogeneous	High	High	Medium	It is used more frequently in adhesive research and is a more sensitive test technique.
Micro push-out	<1 mm^2	Pushing	Homogeneous	High	Medium	High	It is generally used to determine the bond strength of root dentin.
Microtensile	<1 mm^2	Pulling	Homogeneous	Very high	Very high	Very high	It is the most reliable bond strength test used to measure the bond strength of restorative materials to tooth surfaces.

adhesion strength. However, specimen preparation is technique-sensitive; and chipping during sectioning may impair results.[20] A detailed comparison of different test techniques is presented in Table 2.

Specimen Preparation for Testing

For µTBS, restorative materials are applied to prepared tooth surfaces. Using an Isomet cutting machine with water-cooled diamond discs, the bonded specimens are sectioned to yield a 1 mm^2 bonding area. The number of sticks per tooth depends on tooth size. Specimens typically range from 10 to 24 mm in length, half composed of tooth substrate and half of restorative material.[21]

Various geometries can be used in µTBS – stick, dumbbell, or hourglass shapes (Fig. 1). Correct alignment during tensile loading is crucial to avoid shear force interference. Special jigs or customized holders are often employed to achieve precise orientation (Fig. 2).[4,22]

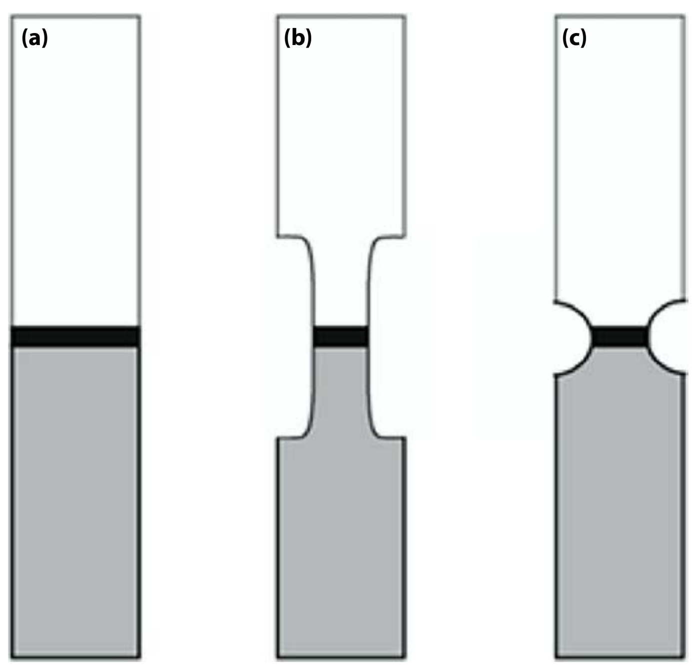

Figure 1. Shapes used in the µTBS test: **(a)** bar, **(b)** dumbbell, and **(c)** hourglass.

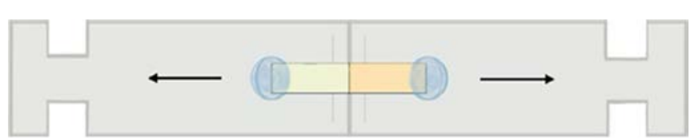

Figure 2. Schematic drawing of the holder apparatus used to measure µTBS (Drawn by the authors).

Advantages of the Microtensile Bond Strength

In contrast to macroscale tests, microtensile methods allow multiple specimens to be prepared from a single tooth, enabling the assessment of tooth-related variables and the calculation of an average bond strength value for each individual tooth. The µTBS permits assessment on rough surfaces and enables better stress distribution across the bonded interface. Structural variations within the same tooth can be examined, and testing can be performed in very small areas such as caries-affected dentin.

The influence of remaining dentin thickness on bond strength can be analyzed, as can the effects of different cavity preparations and restorative techniques on µTBS. The bond strength of root dentin can also be assessed. The bonding performance of each restorative material under investigation can be systematically evaluated. Compared to conventional tensile testing, µTBS provides a more uniform force distribution.

This technique is more suitable for artificial aging procedures and, in contrast to macroscale tests, its shorter intraspecimen diffusion path enables accelerated aging protocols to be implemented more efficiently. Shear forces are minimized, and differences between adhesive systems can be detected more clearly than with other bond strength tests.

Because smaller specimens are used, the test yields more sensitive and consistent results, exhibiting less variability. Compared to other techniques, µTBS typically demonstrates higher bond strength values in statistical analyses. The mode of failure can be easily identified using a stereomicroscope, and mechanical, morphological, and chemical analyses can be performed on the same specimen. As such, µTBS is considered a reliable technique for evaluating adhesive performance.[23]

Disadvantages of the Microtensile Bond Strength

Despite its numerous advantages, the µTBS is more labor-intensive and requires greater technical precision than other bond strength tests. Specimen geometry, adhesive layer thickness, and operator handling can significantly affect stress distribution and measurement accuracy. Preparation requires specialized equipment to produce consistent specimen shapes. Internal defects, dehydration, or damage during sectioning and testing are common, and the unpredictable location of fracture initiation further complicates data interpretation. Measuring very low bond strengths (<5 MPa) remains challenging, as interface stress during handling may artificially alter results.[24]

Roulet and Van Meerbeek[25] proposed three approaches for managing pretest failures in µTBS data:

1. Exclusion of all pretest failures, leading to overestimated bond strength values.
2. Assignment of 0 MPa to failures, resulting in underestimation.
3. Substitution with the lowest value from the relevant adhesive group – a compromise increasingly favored in recent studies for its balanced accuracy and reduced bias.[25]

Importantly, the limitations of µTBS arise not only from technical issues but also from biological variability and material-dependent factors. These aspects must be carefully considered when interpreting results or comparing data across studies.

When the advantages and limitations of µTBS are considered together, it becomes evident that the test should not be relied upon as the sole diagnostic tool. Its high sensitivity means that even minor methodological deviations can substantially influence the results, and the way pretest failures are reported can also alter the outcome directly. While some authors in the literature have argued that µTBS is a reliable method,[23] others have reported that its highly sensitive nature limits its ability to predict clinical performance.[24] These inconsistencies indicate that µTBS findings should be interpreted alongside clinically relevant aging protocols and long-term clinical evidence.

Key Factors Influencing Microtensile Bond Strength Results

Type of Substrate

Studies have shown that the µTBS values and SEM images obtained from human and bovine teeth are quite similar. This suggests that bovine teeth may serve as a practical and ethically acceptable alternative. However, it must be noted that microstructural differences between human and bovine dentin do exist, and these differences may lead to statistical deviations in bond strength outcomes.[26]

Storage Duration and Conditions of Extracted Teeth

According to ISO 11450 standards, µTBS should ideally be performed immediately after tooth extraction to ensure reliable and standardized results. However, this is often impractical in clinical and laboratory settings. As a result, extracted teeth are commonly stored under controlled conditions for later use. Prolonged storage can induce various alterations in dentin structure – particularly degenerative changes in dentin proteins – which can modify the bonding surface.

Some studies report that when appropriate storage media are used (e.g., chloramine-T or formalin), teeth can be stored for up to 2 years without a significant negative impact on µTBS values. In contrast, other studies have found that storage in 0.05% thymol solution for 3–6 months, or in sodium hypochlorite (NaOCl) solution for 2 months, significantly decreases μTBS.[27,28]

To minimize inconsistencies in bond strength data, it is generally recommended that teeth not be stored for longer than 6 months and that all storage conditions be reported in detail within the methodology.

Preparation Method and Surface Treatment

Preparation Method

μTBS values are directly associated with the quality of the hybrid layer and resin tag formation on dentin surfaces. The preparation method significantly influences dentin morphology and, consequently, adhesive penetration. Studies comparing surfaces prepared with diamond burs, carbide burs, and 600-grit silicon carbide papers have found the highest μTBS values in specimens prepared with 600-grit papers and the lowest with diamond burs. This indicates that sanding yields a smoother surface with minimal smear layer, which is more favorable for adhesive infiltration.[5] Therefore, standardizing the preparation method is essential to ensure reliability and comparability of μTBS results.

Etching Procedure

The type, concentration, duration, rinsing, and drying protocols of the acid used to remove the smear layer and demineralize dentin significantly affect μTBS. Over-etching may cause excessive demineralization, reducing resin penetration; under-etching may leave smear debris, hindering hybrid layer formation.[5] Thus, every stage of the etching process must be carefully controlled.

Primer Application

Primer plays a critical role in enabling adhesive monomer infiltration into demineralized dentin. Factors such as the amount of primer used, application time, evaporation method, surface moisture, and the bonding technique (wet vs. dry bonding) influence μTBS. Insufficient application or rapid evaporation may hinder resin penetration and compromise adhesion quality.[5] Therefore, primer application should follow manufacturer guidelines and standardized protocols.

Bond Application

Bonding performance at the adhesive interface is influenced by factors such as application amount, duration, agitation, film thickness, and polymerization. Applying a thin, uniform bonding layer and ensuring adequate light polymerization improves μTBS, whereas thick, uneven, or under-cured layers may reduce adhesive strength.[5] Thus, controlled bond application is crucial.

Configuration Factor (C-Factor)

The C-factor refers to the ratio of bonded surfaces to unbonded, free surfaces and is a critical parameter influencing adhesion due to polymerization shrinkage stress. A study comparing flat dentin surfaces with low C-factors (C=0) to high C-factor cavities (C=3/5) found that while initial μTBS values may be similar, long-term storage in water significantly reduces bond strength in high C-factor cavities.[29] Particularly in Class-V preparations (C=3), polymerization-induced stresses negatively affect adhesion, resulting in considerably lower μTBS values compared to flat surfaces. This adverse effect cannot be fully compensated for by alternative surface preparation methods and exerts a lasting weakening impact on adhesive durability.[29] Therefore, consideration of the C-factor is critical for the accurate interpretation of μTBS results.

Dentin Structure

Sclerotic Dentin

Sclerotic dentin contains two to three times more mineralized matrix compared to normal dentin. This increased mineralization, along with denatured collagen fibers, a hybridized inter-microbial matrix, and acid-resistant sclerotic plugs, significantly impedes resin tag formation, thereby reducing bond strength.[30]

Caries-affected Dentin

The presence of a thick and irregular smear layer, acid-resistant surfaces, disrupted tubule structure, and bacterial or organic debris interferes with both hybrid layer formation and resin tag penetration. Due to decreased mineral content and partially denatured collagen, bond strength is significantly compromised.[31]

Regional Variations in Dentin

Morphological characteristics such as dentin depth, tubule density, and the proportion of intertubular dentin have a direct influence on μTBS. In root dentin, reduced tubule density and increased sclerotic tissue contribute to lower bond strength values.[32]

*These findings show that differences in dentin morphology can substantially affect μTBS outcomes. Therefore, dentin type, location, and structural characteristics should be standardized to ensure comparability and reliability of research outcomes.

Vital Bleaching Procedures

Hydrogen peroxide and carbamide peroxide are widely used in contemporary vital tooth bleaching protocols. Studies have demonstrated that increasing the peroxide concentration and application time correlates with a reduction in enamel µTBS. This decrease is thought to result from peroxide-derived free oxygen, which interferes with resin polymerization by increasing polymerization shrinkage.[33] To avoid this adverse effect on µTBS in post-bleaching restorative procedures, it is recommended to allow sufficient time for the complete dissipation of residual oxygen or to delay bonding procedures accordingly.[33] Moreover, to minimize the impact on µTBS, the bleaching protocol – including peroxide concentration and exposure time – should be carefully planned and standardized.

Chlorhexidine Application

Chlorhexidine is used to enhance the long-term stability of adhesive systems by inhibiting dentin matrix metalloproteinases (MMPs), thus preventing enzymatic degradation of exposed collagen fibrils.[14] Through this mechanism, it is believed to help preserve bond strength at the resin–dentin interface over time. Several studies have reported that application of chlorhexidine in 0.2% and 2% concentrations shows no significant short-term effect on µTBS (e.g., after 24 h). However, after 6 months, the use of 2% chlorhexidine significantly slowed the reduction in µTBS values.[14] Therefore, whether or not a chemical agent (e.g., chlorhexidine) has been applied, and at what concentration, must be documented in detail, as these factors may affect µTBS outcomes.

Thermal Cycling

This procedure involves subjecting specimens to alternating immersion in water baths at different temperatures to evaluate the resistance of restorative materials to intraoral thermal fluctuations. Due to differences in the coefficients of thermal expansion between dental tissues and restorative materials, this process induces repeated thermal stresses at the adhesive interface.[34] Over time, such stresses may lead to microcracks or interfacial separation, resulting in reduced bond strength. While some studies have reported that thermal cycling significantly reduces µTBS,[23] others found the effect to be statistically insignificant.[34] These discrepancies highlight the importance of protocol parameters such as temperature range, dwell time, and number of cycles. Therefore, detailed reporting of thermal cycling conditions and standardized, clinically relevant simulation protocols are essential for improving the clinical validity of µTBS results.[34]

Specimen Geometry

The geometric shape of specimens used in µTBS has a direct influence on the accuracy and reliability of the results. Commonly prepared shapes include straight sticks, dumbbell forms, and hourglass-shaped specimens, each differing in terms of stress distribution, test sensitivity, and ease of preparation.[35] Straight stick and dumbbell shapes offer similar stress distribution and are easier to fabricate, providing an advantage in terms of standardization. In contrast, hourglass-shaped specimens are more difficult to prepare and carry risks such as microcrack formation. Therefore, the use of specialized equipment such as the Micro-Specimen Former is recommended to ensure consistent specimen preparation.[35] In addition, because µTBS values tend to be lower in hourglass-shaped specimens, the geometry of each specimen must be explicitly stated in studies, and its potential influence on the results should be carefully considered during data interpretation.[35]

Test Protocol

For µTBS results to be reliable and reproducible, the test protocol must be meticulously planned and standardized. Proper calibration of the testing machine, the use of a constant crosshead speed, and the preparation of specimens using a sharp blade, under water cooling, and at low speed, are all essential steps. Ideally, all specimens should be prepared by the same operator to eliminate operator-dependent variability. The materials and equipment used in the test – along with their brand, manufacturing lot number, expiration date, and storage conditions – must be thoroughly documented. Adhering to such technical reporting standards is crucial for ensuring cross-study comparability and scientific validity.[21]

Future µTBS research should adopt standardized reporting practices in accordance with ADM guidelines, including detailed documentation of specimen geometry, storage conditions, and crosshead speed. Such standardization will enhance reproducibility and facilitate meaningful comparisons across studies.

All of these factors significantly affect the reliability and reproducibility of µTBS and should be carefully considered during result interpretation.[24] Moreover, classification of failure modes during µTBS provides valuable complementary information in evaluating the effectiveness of adhesive systems. This is because numerical μTBS values alone may not fully reflect the quality of adhesion. Failure mode analysis helps to identify weak areas within the bonded interface and provides insight into the nature of the bond.

Failure Modes Observed During Microtensile Bond Strength

The efficacy of μTBS between two different substrates can be further evaluated by analyzing the types of failure observed following testing. These failure modes are typically classified as adhesive, cohesive, or mixed failures.

- Adhesive failures: These failures occur at the interface between the tooth substrate (enamel/dentin) and the restorative material. Adhesive failure is commonly observed in bonding systems with weak adhesion performance.[36]
- Cohesive failures: These failures occur within one of the substrates – either within enamel, dentin, or the restorative material itself. Although cohesive failures are more frequent in strong bonding scenarios, a high rate of cohesive failure does not necessarily indicate optimal adhesion.[36]
- Mixed failures: Mixed failures involve both adhesive and cohesive components, occurring simultaneously within the substrate and at the interface between different materials. The fracture patterns are typically examined either visually or under a stereomicroscope.[36]

Conclusion

μTBS remains the most sensitive laboratory method for assessing adhesive performance. However, methodological variability limits the clinical translation of laboratory results. Standardized testing protocols and integration with long-term clinical data are necessary to bridge this gap.

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