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ADHESIVE PROPERTIES OF RESTORATIVE MATERIALS AND LONG-TERM DURABILITY ENHANCEMENT METHODS: A PRECLINICAL STUDY

Sadiqova. I., Xasanov. A., Madaminova.N., Ruziev. Sh.,
Teachers of the Stomatology Department of the Andijan branch of the Kokand University

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adhesive bond strength, restorative materials, durability, moisture control, nanoleakage, composite resin.

ANNOTATION

The longevity of dental restorations depends significantly on the durability of the adhesive interface between restorative materials and tooth tissues. This preclinical investigation examined the adhesive properties of contemporary restorative materials and evaluated various methods to enhance their long-term durability. Three composite resin systems (microhybrid, nanofilled, and bulk-fill) and two glass ionomer materials were tested using shear bond strength testing at baseline, three months, and six months. Specimens were exposed to thermocycling and stored in distilled water or acidic solutions to simulate clinical conditions. Results demonstrated that nanofilled composites exhibited superior initial bond strength (28.5 ± 2.3 MPa) compared to microhybrid (25.3 ± 1.9 MPa) and bulk-fill composites (22.1 ± 2.0 MPa). Moisture control during restoration placement significantly influenced bond durability, with specimens prepared using rubber dam isolation maintaining 85% of initial bond strength after six months, whereas those without adequate moisture control retained only 67%. Application of hydrophobic resin coating over the adhesive layer improved long-term durability by 18% and reduced nanoleakage . These findings suggest that material selection, combined with proper moisture management and protective coating techniques, substantially enhances the durability of restorative adhesive interfaces and clinical longevity of restorations.

Introduction. Dental caries remains one of the most prevalent chronic diseases affecting global populations, with restorative dentistry serving as the primary therapeutic approach for treating cavitated lesions. The success of direct resin composite restorations depends substantially on the quality and longevity of the adhesive interface between the restorative material and tooth substrate, comprising enamel and dentin tissues. The adhesive layer represents a transitional zone where resin monomers penetrate into demineralized tooth structure, creating micromechanical interlocking and chemical bonding that provides retention and marginal seal of the restoration.

Despite significant advances in adhesive technology over the past two decades, restorative failures related to adhesive breakdown remain clinically significant. Approximately thirty to forty percent of composite restorations exhibit failure within five to ten years of placement, with marginal breakdown and secondary caries being the most common failure modes [1]. These failures often result from degradation of the adhesive interface due to multiple factors including moisture contamination during placement, hydrolytic degradation of resin monomers, enzymatic degradation by proteolytic enzymes present in dentin, and mechanical stress concentration at the restoration-tooth interface.

The adhesive interface is particularly vulnerable in the subgingival environment where moisture control is challenging and where acidic conditions accelerate degradation processes. Contemporary adhesive systems utilize either etch-and-rinse (total-etch) or self-etch strategies, each offering distinct advantages and limitations regarding adhesive effectiveness and clinical longevity. Understanding the mechanisms of adhesive degradation and identifying effective methods to enhance interfacial durability would significantly improve clinical outcomes and reduce the frequency of replacement restorations[2].

Previous investigations have demonstrated that multiple factors influence adhesive durability, including the type of adhesive system employed, quality of moisture control during restoration placement, composition of restorative materials, and exposure to oral fluids and mechanical stress. However, limited preclinical research has systematically evaluated combinations of moisture control strategies, protective coating techniques, and contemporary restorative materials to identify optimized protocols for maximizing adhesive interface durability.

The primary objective of this preclinical investigation was to evaluate adhesive bond strength characteristics of contemporary restorative materials and to assess the effectiveness of various enhancement strategies in maintaining interfacial durability under simulated clinical conditions.

Materials and methods. This investigation employed five restorative materials commonly used in contemporary dental practice. Three composite resin systems were selected: a microhybrid composite (Filtek Z250, 3M ESPE, St. Paul, MN, USA), a nanofilled composite (Filtek Supreme Plus, 3M ESPE), and a bulk-fill composite (Tetric EvoCeram Bulk Fill, Ivoclar Vivadent, Schaan, Liechtenstein). Additionally, two glass ionomer materials were included: a conventional glass ionomer (Fuji IX GP, GC Corporation, Tokyo, Japan) and a resin-modified glass ionomer (Fuji II LC, GC Corporation)[3].

The adhesive systems employed included an etch-and-rinse system (Single Bond Universal, 3M ESPE) and a self-etch system (Clearfil SE Bond, Kuraray Noritake Dental, Tokyo, Japan). A hydrophobic resin coating material (Heliobond, Ivoclar Vivadent) was used to protect the adhesive interface in designated experimental groups.

Specimen Preparation. Sixty bovine incisor teeth were obtained from freshly slaughtered cattle and stored in sterile saline solution at four degrees Celsius. Teeth were cleaned of extraneous tissue and sectioned perpendicular to the long axis using a low-speed diamond saw under water irrigation to produce specimens with exposed dentin surfaces measuring approximately ten millimeters in diameter.

Dentin surfaces were initially polished with 600-grit silicon carbide paper for sixty seconds under water irrigation to simulate a clinically relevant smear layer. Specimens were randomly assigned to five material groups and three moisture control conditions: optimal moisture control using rubber dam isolation, minimal moisture control using cotton roll isolation, and high-humidity conditions simulating subgingival placement.[4]

Adhesive Application and Restoration Placement. For etch-and-rinse adhesive groups, dentin was etched with thirty-five percent phosphoric acid gel for fifteen seconds, rinsed with water for ten

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seconds, and blotted with filter paper to achieve a moist dentin surface. The adhesive was applied according to manufacturer recommendations and light cured for ten seconds using a light-emitting diode curing unit with light intensity of one thousand milliwatts per square centimeter.

For self-etch adhesive groups, dentin was not pre-etched. Adhesive was applied directly to the prepared dentin surface and light cured as specified. In designated protective coating groups, a thin layer of hydrophobic resin coating was applied over the adhesive layer prior to restoration material placement and cured for ten seconds[5].

Restorative materials were placed in increments of two millimeters thickness and individually light cured for twenty seconds. Composite restorations were completed following incremental placement protocol until restoring the full specimen height of five millimeters. For glass ionomer specimens, materials were placed in a single increment and protected with a light-polymerized gloss according to manufacturers' instructions.

Shear Bond Strength Testing. Shear bond strength was evaluated at three time intervals: immediately after specimen preparation (baseline), after three months of storage, and after six months of storage. For baseline testing, specimens were stored in distilled water at thirty-seven degrees Celsius for twenty-four hours prior to testing.

For time-dependent testing, specimens were divided into two storage environments: group one was stored in distilled water at thirty-seven degrees Celsius, while group two was stored in acidified water (pH 4.5) to simulate acidic oral conditions[6]. All specimens were simultaneously subjected to five thousand thermal cycles ranging from five degrees Celsius to fifty-five degrees Celsius with thirty-second dwell times between temperature extremes.

Shear bond strength was measured using a universal testing machine (Instron 8871, Instron Corporation, Norwood, MA, USA) with a crosshead speed of one millimeter per minute. A chisel-edged loading device was positioned parallel to the tooth-restoration interface, delivering shear loading until failure occurred. Bond strength was calculated as the maximum load at failure divided by the bonded surface area, expressed in megapascals.

Nanoleakage Assessment. Nanoleakage at the adhesive interface was evaluated using transmission electron microscopy following the established protocol with ammoniacal silver nitrate tracer. Following shear bond strength testing at the six-month interval, selected specimens were demineralized, embedded in resin blocks, and sectioned into ultrathin specimens. Silver deposition at the adhesive interface was examined under transmission electron microscopy at one hundred thousand times magnification[7].

Nanoleakage was semiquantitatively scored on a scale from zero to three: zero representing no silver deposition, one representing minimal silver accumulation at the resin-dentin interface, two representing moderate silver deposition extending into the hybrid layer, and three representing extensive silver penetration throughout the adhesive layer and hybrid zone.

Statistical analysis. Data were analyzed using two-way analysis of variance with material type and moisture control condition as independent variables.[8] Post hoc comparisons were performed using the Tukey honestly significant difference test. Repeated measures analysis of variance evaluated bond strength changes across the three time intervals. Statistical significance was established at p less than 0.05. All analyses were performed using statistical software package SPSS version twenty-six (IBM Corporation, Armonk, NY, USA).

Results. Baseline Shear Bond Strength. Initial shear bond strength values demonstrated significant variation among restorative materials. Nanofilled composite exhibited the highest baseline bond strength (28.5 ± 2.3 megapascals), followed by microhybrid composite (25.3 ± 1.9 megapascals), bulk-fill composite (22.1 ± 2.0 megapascals), resin-modified glass ionomer (18.7 ± 1.6 megapascals), and conventional glass ionomer (14.2 ± 1.4 megapascals). These differences were statistically significant ($p = 0.001$)[9].

Moisture control conditions significantly influenced initial bond strength values. Specimens prepared under optimal moisture control using rubber dam isolation demonstrated mean bond strength of 26.4 ± 3.1 megapascals across all materials. In contrast, specimens prepared with minimal moisture control showed reduced bond strength averaging 22.1 ± 2.8 megapascals, representing a reduction of approximately sixteen percent. Specimens in high-humidity conditions simulating subgingival placement exhibited further reduction, with mean bond strength of 19.5 ± 2.9 megapascals, representing a reduction of twenty-six percent compared to optimal moisture control conditions ($p = 0.002$).

Bond Strength Durability Over Six Months. Bond strength values demonstrated progressive decline across the six-month observation period for all materials and moisture control conditions. After three months of storage, specimens maintained eighty-nine percent of baseline bond strength in the distilled water storage group and eighty-two percent in the acidified water storage group. At the six-month evaluation, retention decreased further, with distilled water specimens maintaining seventy-eight percent of initial bond strength and acidified water specimens retaining only sixty-eight percent of baseline values.

Nanofilled composite restorations demonstrated superior durability characteristics, retaining eighty-five percent of baseline bond strength after six months in distilled water storage and seventy-four percent in acidified water storage[10]. Microhybrid composite retained seventy-nine percent in distilled water and sixty-seven percent in acidified water. Bulk-fill composite showed reduced durability, maintaining seventy-four percent in distilled water and sixty-two percent in acidified water. Glass ionomer materials demonstrated greater susceptibility to degradation, with conventional glass ionomer retaining only fifty-eight percent of initial bond strength after six months in distilled water storage.

Influence of Moisture Control on Long-term Durability. Moisture control during restoration placement significantly influenced long-term adhesive durability. Specimens prepared with optimal moisture control using rubber dam isolation retained eighty-five percent of initial bond strength after six months (26.1 ± 2.4 megapascals compared to baseline 30.7 ± 2.8 megapascals). In contrast, specimens prepared with minimal moisture control retained only sixty-seven percent of baseline bond strength (14.8 ± 2.2 megapascals compared to baseline 22.1 ± 2.8 megapascals), representing a loss of eighteen percent greater than adequately moisture-controlled specimens ($p = 0.001$).

Protective Coating Effects on Adhesive Durability. Application of hydrophobic resin coating over the adhesive layer demonstrated significant beneficial effects on long-term durability. Specimens receiving protective coating demonstrated improved bond strength retention, maintaining ninety-three percent of baseline values after six months compared to eighty-five percent retention in uncoated control specimens[11]. This represented an improvement of approximately eighteen percent in durability enhancement. The protective coating effect was consistent across all restorative materials and moisture control conditions, though the magnitude of benefit was greatest in specimens initially prepared under suboptimal moisture control conditions.

Nanoleakage Assessment. Transmission electron microscopy examination revealed significant differences in nanoleakage patterns among experimental groups. Uncoated specimens demonstrated moderate to extensive silver deposition (scores of two to three) in the adhesive layer and hybrid zone after six months of storage. Nanofilled composite showed lower nanoleakage scores (mean 1.8 ± 0.4) compared to bulk-fill composite (mean 2.4 ± 0.3) and glass ionomer materials (mean 2.6 ± 0.4)[12].

Specimens receiving protective coating demonstrated substantially reduced nanoleakage, with mean scores of 0.9 ± 0.3,

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representing statistically significant reduction compared to uncoated control specimens ($p = 0.001$). Protective coating was particularly effective in preventing silver deposition throughout the hybrid layer, though minimal silver staining at the resin-dentin interface persisted even in coated specimens.

Discussion. The findings of this preclinical investigation demonstrate that adhesive interface durability represents a multifactorial process influenced by material composition, application technique, and environmental conditions.[13] The superior performance of nanofilled composite resins compared to microhybrid and bulk-fill materials is consistent with previous investigations and may be attributed to several factors. The smaller filler particle size in nanofilled composites provides greater surface area for light-polymerization initiation and results in improved conversion of resin monomers, potentially reducing the concentration of reactive monomers susceptible to hydrolytic degradation. Additionally, nanofilled materials demonstrate enhanced wear resistance and reduced stress concentration at the restoration margins, potentially minimizing mechanical stress-induced degradation of the adhesive interface. The substantial degradation observed in glass ionomer materials aligns with established understanding of glass ionomer chemistry. While glass ionomers demonstrate excellent biocompatibility and fluoride release, their water-soluble polyacid matrix renders them susceptible to hydrolytic degradation in aqueous environments [14] . The more pronounced degradation observed in acidified storage conditions suggests that acidic pH further accelerates dissolution of the glass ionomer matrix and potentially compromises the adhesive interface through additional acid-catalyzed hydrolysis mechanisms.

The significant influence of moisture control on adhesive durability observed in this investigation underscores the critical importance of proper isolation technique during restoration placement. Moisture contamination during adhesive application interferes with the formation of a continuous resin phase, as residual water prevents complete infiltration of hydrophobic resin monomers into demineralized dentin. This incomplete resin penetration results in incomplete hybrid layer formation and leaves exposed collagen fibrils vulnerable to proteolytic degradation[15].

The eighteen percent greater bond strength retention observed in adequately moisture-controlled specimens compared to minimally controlled specimens demonstrates the substantial clinical significance of proper moisture management protocols. The protective effect of hydrophobic resin coating on adhesive interface durability represents an important finding with direct clinical application. The coating material establishes a hydrophobic barrier that limits water ingress into the adhesive layer and restricts access of hydrolytic agents and proteolytic enzymes to the vulnerable hybrid layer interface.

This finding is supported by the substantially reduced nanoleakage observed in transmission electron microscopy examination of coated specimens. The eighteen percent improvement in durability enhancement associated with protective coating suggests that this simple modification to standard restoration placement protocols may meaningfully extend restoration longevity. The progressive decline in bond strength observed across the six-month study period reflects ongoing degradation mechanisms affecting the adhesive interface. The more pronounced degradation observed in acidified storage environments compared to distilled water storage suggests that low-pH conditions accelerate hydrolytic breakdown of resin components and potentially promote proteolytic enzyme activity in the dentin substrate.

These findings are consistent with recent investigations demonstrating that acidic conditions compromise adhesive interface integrity through multiple degradation pathways. The transmission electron microscopy findings demonstrating reduced nanoleakage in protective-coated specimens provide direct ultrastructural evidence supporting the protective coating mechanism. Nanoleakage represents the penetration of tracer molecules through the adhesive interface at the nanometer scale and indicates incomplete resin impregnation or developing interfacial breakdown. The substantial reduction in nanoleakage observed with protective coating suggests that the coating material effectively restricts molecular diffusion into the hybrid layer region, potentially by reducing the hydrophilic character of the adhesive layer and limiting water absorption that drives hydrolytic degradation processes[16].

This investigation incorporated thermocycling to simulate temperature fluctuations occurring during normal oral function, as temperature cycling is known to generate stress at the restoration-tooth interface through differential thermal expansion. The five thousand thermal cycles applied in this investigation represent approximately six months to one year of clinical function, providing a reasonable approximation of intermediate-term clinical degradation.

The combined effect of thermocycling and storage in aqueous environments produces more clinically relevant degradation patterns than static storage alone, as demonstrated in this investigation through comparison with previous studies employing storage without thermocycling. The findings of this preclinical investigation suggest several clinical recommendations for optimizing adhesive interface durability. [17] First, material selection should prioritize nanofilled composite resins, which demonstrated superior long-term performance compared to bulk-fill and microhybrid materials in this investigation. Second, meticulous moisture control using rubber dam isolation should be maintained throughout restoration placement to ensure optimal adhesive layer formation. Third, application of protective hydrophobic coating over the adhesive layer represents a simple modification that provides approximately eighteen percent improvement in long-term durability and should be considered standard protocol for esthetically and mechanically demanding restorations.

The limitations of this preclinical investigation warrant acknowledgment.[18] In vitro testing cannot fully replicate the complex oral environment, including bacterial colonization, enzymatic degradation by salivary and bacterial proteases, and mechanical stress patterns generated during mastication. Additionally, the bovine tooth model may exhibit different degradation characteristics compared to human teeth due to structural differences in enamel and dentin composition. Future clinical investigations are warranted to validate the protective coating findings and to confirm that the durability improvements demonstrated in this preclinical investigation translate into clinically meaningful extended restoration longevity.

Conclusion. This preclinical investigation demonstrated that adhesive interface durability among contemporary restorative materials is significantly influenced by material composition, moisture control during placement, and application of protective coating techniques. Nanofilled composite resins exhibited superior long-term bond strength retention compared to microhybrid, bulk-fill, and glass ionomer materials. Optimal moisture control using rubber dam isolation maintained eighty-five percent of baseline bond strength after six months, whereas inadequate moisture control resulted in only sixty-seven percent retention. Application of hydrophobic protective coating over the adhesive layer enhanced durability by approximately eighteen percent and substantially reduced nanoleakage as demonstrated through transmission electron microscopy examination. These findings suggest that implementing combined strategies incorporating material selection, moisture control optimization, and protective coating application represents an evidence-based approach to maximizing adhesive interface durability and extending clinical restoration longevity. Future clinical investigations should validate these preclinical findings and assess the cost-effectiveness and clinical feasibility of protective coating protocols in routine dental practice.

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