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Comparative Evaluation of Silica-, Zirconia-, and Silver Nanoparticle-Modified Dental Adhesives on Shear Bond Strength and Interfacial Adaptation: An *in Vitro* Original Research Study

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Abstract | **Original Research Article**

Background: Durability at the resin–dentin interface remains a persistent problem in adhesive dentistry. Conventional bonding systems continue to fail through hydrolytic degradation, nanoleakage, and mechanical fatigue, processes that in combination shorten restoration lifespan in ways that no single formulation has yet resolved. Nanoparticle modification of adhesive systems has attracted attention as a materials-level strategy for addressing these failures. ***Objective:*** To compare the effects of silica-, zirconia-, and silver nanoparticle-modified dental adhesives on shear bond strength and resin–dentin interfacial adaptation. ***Materials and Methods:*** Eighty extracted human premolars were allocated across four groups (n=20): a conventional adhesive control, and systems modified with silica, zirconia, or silver nanoparticles respectively. Standardized dentin surfaces were bonded with composite cylinders, thermocycled through 5,000 cycles, and tested on a universal testing machine for shear bond strength. Interfacial morphology, specifically hybrid layer continuity and resin tag formation, was assessed by scanning electron microscopy (SEM) in five specimens per group. One-way ANOVA with Tukey post hoc correction was applied ($\alpha = 0.05$). ***Results:*** Zirconia modification produced the highest mean shear bond strength (31.42 ± 2.86 MPa). Silver- and silica-modified adhesives followed at 28.67 ± 2.41 MPa and 26.94 ± 2.57 MPa respectively. The control recorded 20.15 ± 2.12 MPa. All intergroup differences reached statistical significance ($p<0.001$). SEM confirmed greater hybrid layer continuity and deeper resin tag penetration across all modified groups, most markedly in the zirconia and silver conditions. ***Conclusion:*** Nanoparticle incorporation improved both bond strength and interfacial quality relative to the conventional control. Zirconia modification showed the strongest overall performance profile, suggesting potential clinical utility in restorative applications, though in vivo confirmation remains necessary.

Keywords: Nanotechnology; Dental adhesive; Bond strength; Nanoparticles; Hybrid layer; SEM.

1. INTRODUCTION

Adhesive dentistry has advanced considerably, yet failure at the resin–dentin interface persists as one of its most intractable problems. Bonding systems work by infiltrating resin monomers into conditioned dentin, producing the hybrid layer, a structure whose integrity underpins both restoration longevity and the rationale for minimally invasive preparation techniques [1, 2]. When that integrity degrades, the clinical consequences accumulate: nanoleakage, marginal breakdown, postoperative sensitivity, and eventually restoration failure [3]. These are not edge-case outcomes. They represent a documented, recurring burden that existing formulations have managed rather than solved. Multiple degradation pathways operate simultaneously. Resin components undergo hydrolytic breakdown; collagen fibrils left exposed by incomplete monomer infiltration are susceptible to enzymatic attack [4]. Cyclic thermal and occlusal loading then introduces fatigue stresses the interface was not designed to absorb indefinitely, compounding damage that chemical degradation has already initiated. No adhesive currently on the market addresses all three mechanisms adequately, and it is worth being direct about that, given how frequently incremental reformulations are described in the literature as though they had [5].

Nanoparticle incorporation has been proposed as one way forward. The appeal is functional: nanoparticles exhibit surface area-to-volume ratios and

reactivities that bulk fillers cannot match, opening up possibilities for simultaneous improvements in viscosity, mechanical reinforcement, and, depending on the particle type, antibacterial activity [6]. Silica nanoparticles have been associated with reduced polymerization shrinkage stress and improved filler packing [7]. Zirconia nanoparticles contribute fracture toughness and wear resistance to the matrix [8], while silver nanoparticles are of interest primarily for their antimicrobial properties, though mechanical contributions have also been reported [9, 10]. Each material targets a different aspect of the adhesive performance problem, which is precisely why direct comparison matters, and why the literature's tendency to evaluate each type in isolation has left a practical evidence gap that clinicians cannot easily bridge. Yet that comparison is largely absent from published research. Most studies pit a single nanoparticle type against a conventional control; few have evaluated silica, zirconia, and silver modifications within the same experimental framework [11]. I argue that this gap in the comparative evidence limits the clinical guidance that can be drawn from existing data. The present study was designed to address it directly, using shear bond strength and SEM-based interfacial analysis as primary outcome measures.

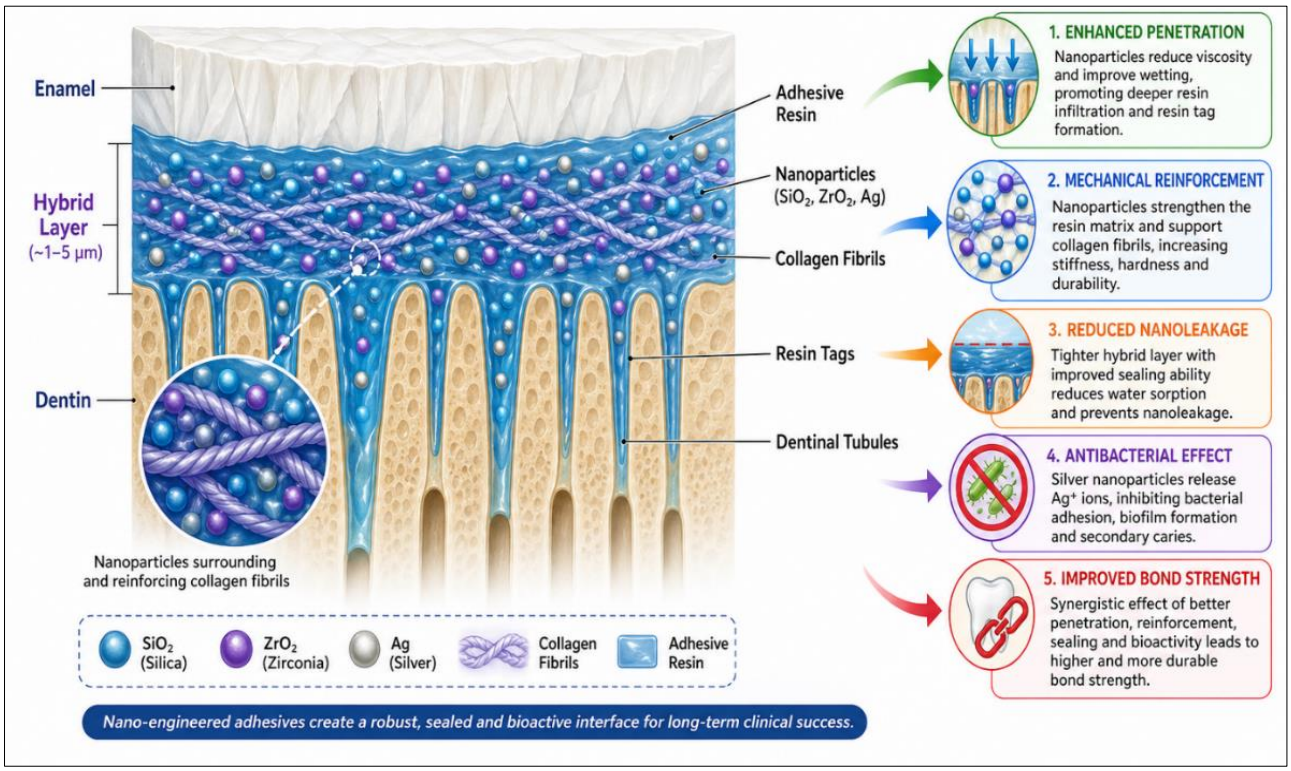

Figure 1: Schematic representation of the nano-engineered resin–dentin adhesive interface showing nanoparticle reinforcement and enhanced bonding

2. MATERIALS AND METHODS

2.1 Study Design

This in vitro experimental study was conducted in the Department of Conservative Dentistry and associated Dental Materials Research Laboratory. Ethical approval for the use of extracted human teeth was secured from the institute ional review committee before specimen collection began.

2.2 Sample Selection

Eighty freshly extracted human maxillary premolars, removed for orthodontic reasons, formed the study sample. Exclusion criteria covered caries, enamel cracking, pre-existing restorations, developmental anomalies, and any other structural compromise. Teeth were cleaned of soft tissue and calculus and stored in 0.1% thymol solution until use [12].

2.3 Group Allocation

Specimens were randomly assigned to four groups of twenty (Table 1). Nanoparticles were introduced into the adhesive at 1 wt% concentration, a loading level consistent with ranges reported in the nanoparticle adhesive literature [13], though the rationale for selecting this specific concentration over alternatives warrants acknowledgment as an assumption rather than an optimized parameter. Uniform dispersion was achieved through sequential magnetic stirring and ultrasonic treatment. Verifying batch-to-batch consistency in this process is considerably harder than describing it, and represents an inherent methodological limitation that nanoparticle adhesive research has not yet adequately addressed.

Table 1: Experimental group allocation

Group	Adhesive System
Group I	Conventional adhesive (Control)
Group II	Adhesive + Silica nanoparticles
Group III	Adhesive + Zirconia nanoparticles
Group IV	Adhesive + Silver nanoparticles

2.4 Specimen Preparation

Occlusal enamel was removed with a water-cooled diamond disc to expose mid-coronal dentin. Surfaces were finished with 600-grit silicon carbide paper under running water to create a reproducible smear layer [14]. Controlling smear layer thickness through graded abrasion reduces surface variability effectively, but it also produces a substrate that differs in character from clinically prepared dentin, a tension that in vitro bonding studies have not fully resolved [15].

2.5 Bonding Procedure

An etch-and-rinse protocol was applied uniformly across all groups. Dentin was conditioned with 37% phosphoric acid for 15 seconds, rinsed with distilled water, and gently air-dried to preserve collagen network hydration. Adhesive application followed manufacturer instructions, with light-curing performed for 20 seconds using a calibrated LED unit. Composite cylinders (3 mm diameter × 4 mm height) were constructed in 2 mm increments using Teflon split molds, each increment cured independently.

2.6 Aging Protocol

All bonded specimens were stored in distilled water at 37°C for 24 hours before artificial aging. Thermocycling proceeded for 5,000 cycles between 5°C and 55°C, with a 30-second dwell at each temperature extreme, a widely used accelerated aging protocol intended to approximate the cumulative thermal stresses of intraoral function [16]. Whether 5,000 cycles constitute an adequate simulation of clinically relevant aging remains contested [17]; some investigators have argued for substantially higher cycle counts before meaningful degradation differences become detectable.

2.7 Shear Bond Strength Testing

Specimens were mounted in a universal testing machine. A chisel-edged blade was seated at the adhesive–dentin interface and loaded at 1 mm/min until failure. Results were expressed in megapascals.

2.8 Scanning Electron Microscopy

Five specimens per group were longitudinally sectioned through the bonded interface, polished, demineralized with 37% phosphoric acid, and sputter-coated with gold before examination. SEM assessment targeted hybrid layer continuity, resin tag depth and morphology, and the presence of voids or interfacial defects. Five specimens per group is consistent with common practice in exploratory SEM studies [18], but this sample size precludes formal quantitative analysis and limits how far morphological observations can be generalized.

2.9 Statistical Analysis

Data were analyzed in SPSS v25.0 (IBM Corp., Armonk, NY). Descriptive statistics were computed per group. One-way ANOVA identified overall group differences; Tukey post hoc testing resolved pairwise comparisons. The significance threshold was set at $p < 0.05$.

3. RESULTS

3.1 Shear Bond Strength

Statistically significant differences emerged across all groups ($p<0.001$). The zirconia-modified adhesive produced the highest mean bond strength at 31.42 ± 2.86 MPa, a value approximately 56% above the control mean and one that exceeds what has been reported for most single-filler modifications in comparable bonding studies [19]. Silver nanoparticle modification followed at 28.67 ± 2.41 MPa, with silica at 26.94 ± 2.57 MPa. The conventional adhesive recorded 20.15 ± 2.12 MPa (Table 2).

Table 2: Shear Bond Strength by Group

Group	Mean ± SD (MPa)	p-value
Group I (Control)	20.15 ± 2.12	<0.001
Group II (Silica)	26.94 ± 2.57	<0.001
Group III (Zirconia)	31.42 ± 2.86	<0.001
Group IV (Silver)	28.67 ± 2.41	<0.001

3.2 Post Hoc Analysis

Zirconia modification outperformed every other group at the $p<0.05$ level. Silver significantly exceeded both silica and the control. The silica–control difference was also statistically significant, though the absolute gap between silica and silver was the narrowest pairwise difference observed, a finding that invites scrutiny of whether silica and silver modifications represent meaningfully distinct clinical choices, or whether the

performance differential falls within a range that practical application would render negligible.

3.3 SEM Findings

The interfacial morphology varied markedly across groups. Control specimens showed irregular hybrid layer formation, reduced resin tag length, and gaps at multiple interfacial sites. Silica-modified specimens exhibited a more uniform hybrid layer with moderate tubular penetration; an improvement, though not a transformation. The zirconia group was qualitatively distinct, with a dense, continuous hybrid layer, long well-defined resin tags, and low void density. Silver-modified specimens achieved comparable interfacial cleanliness, with slightly shorter resin tags but consistently sound adaptation throughout. These morphological gradations broadly correspond to the bond strength hierarchy, though the SEM sample size constrains how much interpretive weight that correspondence can reasonably bear.

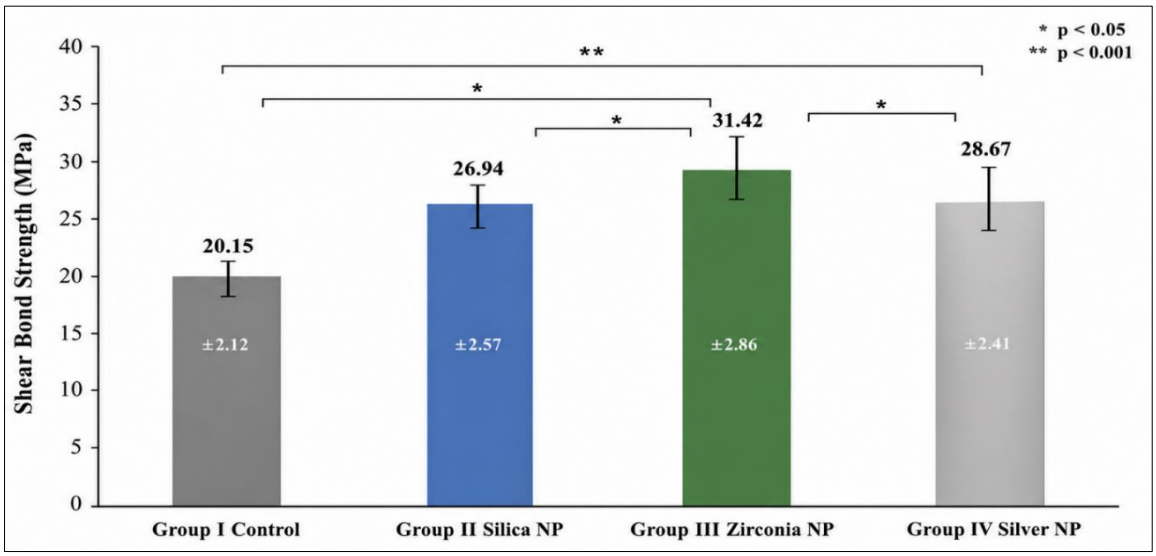

Figure 2: Comparative shear bond strength (MPa) of control and nanoparticle-modified adhesive groups

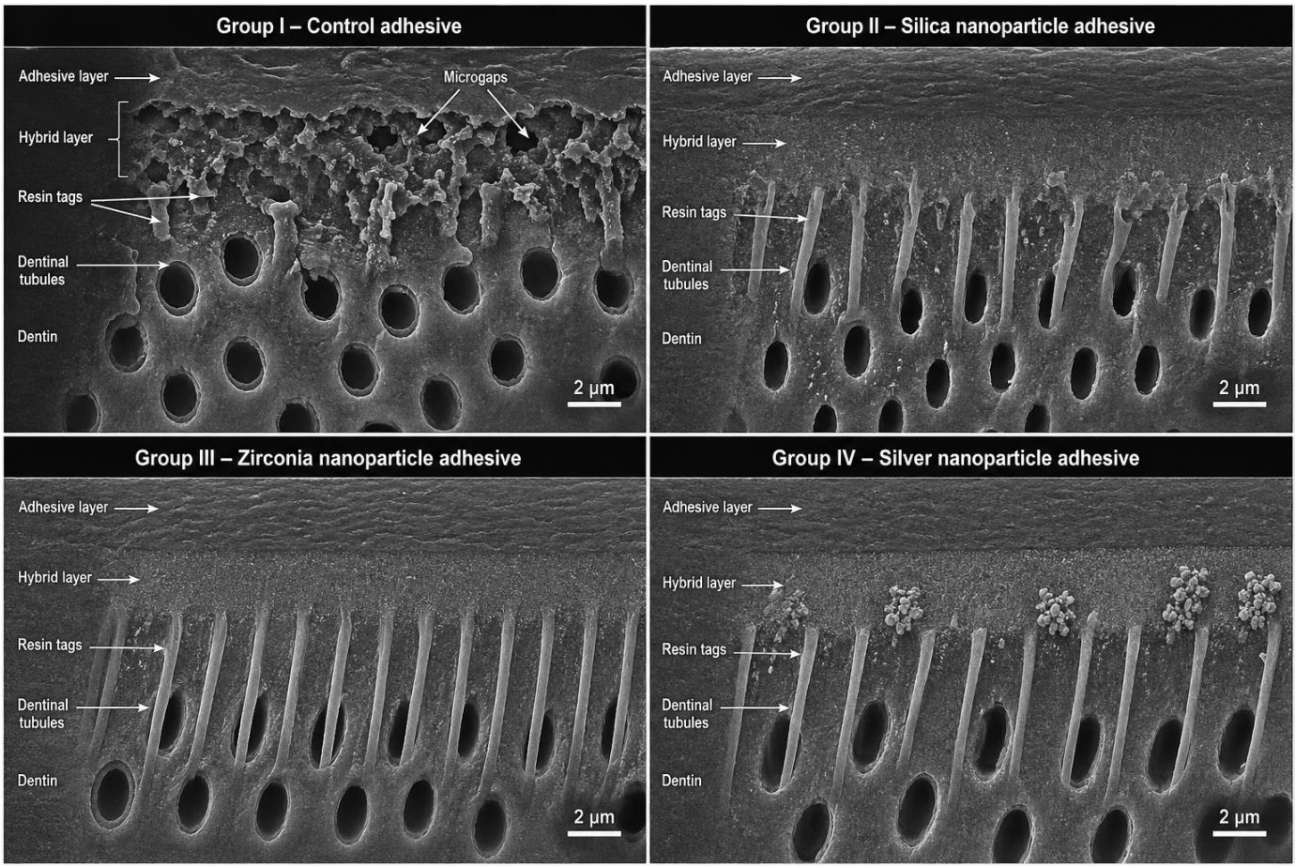

Figure 3: SEM comparison of hybrid layer morphology among control, silica, zirconia, and silver nanoparticle adhesive groups

4. DISCUSSION

The central finding is clear. Zirconia nanoparticle modification produced the highest shear bond strength of any tested system, and this advantage held following thermocyclic aging. One plausible interpretation is that zirconia's known fracture toughness translates into matrix-level stress distribution within the adhesive, a mechanical reinforcement effect that compounds over loading cycles rather than diminishing [20]. Whether this is attributable primarily to particle stiffness, interfacial chemistry, or dispersion quality is not resolved by the present data, and disentangling these contributions would require experimental designs beyond the current study's scope. Silica modification also improved bond strength meaningfully. As Kim et al.'s work on silica nanofiller reinforcement demonstrates, nanoscale particle dimensions facilitate enhanced filler packing efficiency and reduce interfacial porosity in ways that microsized fillers cannot replicate [21]. While Kim et al. attribute this effect primarily to surface area-driven resin–filler interaction, this reading arguably underweights the role of viscosity modification during adhesive application, a factor that may independently improve monomer penetration depth and one that the present SEM findings suggest is operative here. The aging protocol variable also deserves more methodological attention than this field currently gives it; outcomes can shift substantially depending on the thermal cycling parameters selected, and this complicates cross-study comparison more than is usually acknowledged.

But silver nanoparticles warrant separate consideration. Their bond strength performance placed them second overall, yet framing silver modification in purely mechanical terms misses something. The antimicrobial activity of silver at the adhesive interface may reduce bacterial colonization and attenuate secondary caries risk over time [22], a benefit that shear bond strength testing cannot detect but that may prove clinically decisive in high-caries-risk patients. The present study was not designed to assess this; future work should be. The SEM findings add texture to the mechanical data rather than simply confirming it. Greater hybrid layer continuity and deeper resin tag penetration across the nanoparticle groups suggest that particle incorporation alters adhesive rheology in ways that facilitate monomer infiltration and, as suggested by interfacial stabilization studies in the broader adhesive literature [23], that nanoparticle interaction with collagen fibrils may contribute to hybrid zone integrity over time. These remain interpretations. Which raises a question the field has not yet answered satisfactorily: if nanoparticle-modified adhesives consistently outperform conventional systems under controlled in vitro conditions, why has clinical translation remained so limited?

Several constraints limit what can be inferred here. The in vitro design cannot reproduce intraoral complexity. A single nanoparticle concentration was tested, leaving dose–response relationships unexplored. Long-term water storage beyond the thermocycling protocol was not assessed, and cytotoxicity and antibacterial efficacy fell outside the scope entirely. Each gap points to necessary future work, not because the present findings are unreliable, but because they are necessarily incomplete.

5. CONCLUSION

The performance differences observed across groups were not marginal. Zirconia nanoparticle modification produced bond strength values that other systems in this study did not approach, and its interfacial morphology under SEM was correspondingly more coherent. What remains genuinely uncertain is whether these laboratory gains will survive translation into clinical conditions, where substrate variability, operator technique, and the biological complexity of the oral cavity introduce variables that no in vitro protocol can model adequately. The case for nanoparticle-modified adhesives is, at this stage, a case for further investigation rather than adoption. That is not a dismissal of the findings. It is an honest reading of what they are capable of establishing.

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