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Assessment of enamel demineralization, shear bond strength, and failure mode following the use of orthodontic adhesive containing silver nanoparticles: an *in-vitro* study

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Objective: This study evaluated enamel demineralization, shear bond strength, and failure modes associated with orthodontic adhesives containing silver nanoparticles (AgNPs).

Methods: Forty-eight extracted human premolar teeth were selected and evenly divided into two groups based on the intended assessments, with 28 specimens in each group: Part I focused on enamel demineralization depth, and Part II addressed shear bond strength and failure modes. Specimens were prepared, mounted, and bonded using two adhesive systems: Group A used a conventional adhesive system without AgNPs, while Group B employed a conventional adhesive system mixed with AgNPs. In Part I, specimens underwent artificial demineralization at pH 4.5°C and 37°C for 7 days, followed by buccolingual sectioning. The depth of demineralization was measured using a scanning electron microscope and analyzed with ImageJ software. In Part II, shear bond strength was assessed using a universal testing machine, followed by failure mode evaluation using a stereomicroscope.

Results: Results showed that Group A exhibited a significantly greater demineralization depth compared to Group B ($p = 0.000$). However, no significant difference was observed between overall location levels ($p = 0.093$). Additionally, Group B demonstrated significantly higher shear bond strength ($p = 0.000$) and a more uniform distribution of failure modes compared to Group A.

Conclusions: These findings suggest that incorporating silver nanoparticles into orthodontic adhesives reduces demineralization depth and enhances shear bond strength compared to conventional adhesive systems. In contrast,

conventional adhesives were associated with a higher incidence of adhesive mode failure.

KEYWORDS

orthodontic adhesive, silver nanoparticles, shear bond strength, mode of failure, enamel demineralization

1 Introduction

Enamel demineralization [i.e., white spot lesions (WSLs)] can occur during orthodontic therapy, often within the first month after the insertion of orthodontic appliance. WSLs can be detected in up to 96% of orthodontic patients (Tukfekci et al., 2011; Bishara and Ostby, 2008; Borzabadi-Farahani et al., 2014; Chapman et al., 2010; Ren et al., 2014). Maintaining proper oral hygiene becomes challenging when orthodontic appliances are present, which can lead to an increase in the colonization of bacteria such as *Lactobacillus acidophilus* and *Streptococcus mutans*. This bacterial growth can lower the pH of the mouth to a critical level of 5.5, promoting the demineralization process and potentially resulting in the formation of WSLs (Khoroushi and Kachuie, 2017).

Maintaining proper oral hygiene is the most effective preventive measure to control biofilm formation and reduce the likelihood of WSL formation (Migliorati et al., 2015). The use of topical fluoride varnish and the inclusion of antibacterial agents in mouthwashes and toothpaste, can further enhance these preventive measures, however, individual compliance with these practices is crucial for their effectiveness (Lopatiene et al., 2016; Cosma LL et al., 2019). More advanced preventive measures may include the use of bioactive glass and Casein Phosphopeptide Amorphous Calcium Phosphate (CPP-ACP) (Taha et al., 2017; Cochrane and Reynolds, 2012; Beerens et al., 2018; De Almeida et al., 2018).

Another common challenge that orthodontists encounter during treatment is bracket detachment, which can occur in approximately 0.6%–28.3% of cases involving light-cured or chemical-cured composite resins (Al-Thomali, 2022). Shear bond strength (SBS) is defined as the maximum stress a material can sustain in a shear loading condition before failing (El Mourad, 2018). This measurement is crucial in orthodontics, as it ensures that brackets remain securely attached to tooth enamel throughout the treatment process.

The success of orthodontic treatments greatly depends on how well adhesives can endure mechanical forces. Failures of these adhesives are typically categorized into three types: adhesive, cohesive, and mixed (Kumar et al., 2005). These failure modes not only influence the durability and effectiveness of orthodontic treatments but also involved in developing new adhesives with greater mechanical strength and better bonding qualities.

The incorporation of nanotechnology into bonding agents has significantly improved adhesive performance and transformed the field. These advanced materials utilize smaller particle sizes, which not only enhance adhesive strength but also provide antibacterial properties, greatly reducing the risk of microbial buildup around brackets (Jung et al., 2008). Metallic nanoparticles are particularly beneficial due to their small size and high surface-to-volume ratio, allowing them to release metal ions that interact

with bacterial membranes, resulting in antibacterial effects (Song and Ge 2019; Vimbela et al., 2017).

The integration of silver nanoparticles (AgNPs) into orthodontic adhesive systems provides both antibacterial and antifungal properties. This is primarily attributed to the reactive oxygen species present on the surface of silver nanoparticles, as well as the release of free silver ions that induce cell death and inhibit the growth of *Streptococcus mutans* (Borzabadi-Farahani et al., 2014; Wang et al., 2016; Tran and Le, 2013). By enhancing the interaction between the adhesive and microorganisms, AgNPs effectively prevent bacterial proliferation at the junction of orthodontic adhesive resins, thereby reducing the likelihood of lesion development (Yassaei et al., 2020; Degrazia et al., 2016; Eslamian et al., 2020).

The shear bond strength of adhesives is affected by the amount of filler they contain. Increasing the filler content in adhesives shows improved bonding between the enamel and the bracket (Faltermeier et al., 2007). Previous studies have examined the impact of silver nanoparticles (AgNP) on adhesive bonding and failure, yielding contradictory findings regarding shear bond strength. Some research indicates that adhesives containing AgNP may decrease shear bond strength (Eslamian et al., 2020; Reddy et al., 2016), while other studies suggest that adding AgNP can enhance bond strength by increasing the filler content (Al-Thomali, 2022; Faltermeier et al., 2007).

There are significant differences in the modes of failure observed. Sánchez-Tito and Tay (2024) found that 1% AgNP adhesive caused primary failure at the adhesive-enamel interface; in contrast, failures in the 0.05% AgNP adhesive and the conventional adhesive group predominantly took place at the bracket-adhesive interface (Sánchez-Tito and Tay, 2024). Additionally, previous research has found no significant differences in the distribution of the Adhesive Remnant Index (ARI) failure pattern between the modified adhesive and the control group, suggesting a similar SBS effect on the mode of failure (Eslamian et al., 2020).

To the best of our knowledge, there is a significant gap in understanding how incorporating silver nanoparticles (AgNPs) into orthodontic adhesives influences enamel demineralization and essential bonding properties, such as shear bond strength (SBS) and failure modes. Understanding the impact of AgNPs on these factors is crucial for optimizing adhesive performance and enhancing patients' overall experience and satisfaction with orthodontic treatments. Therefore, it is essential to thoroughly investigate the role of silver nanoparticles in orthodontic adhesive formulations. This study aimed to evaluate the effects of AgNPs-containing orthodontic adhesives on enamel demineralization, SBS, and failure modes. Additionally, this study examined the impact of incorporating AgNPs on the microhardness and surface roughness of the experimental adhesive material. The null hypothesis proposed that there would be no significant differences in enamel demineralization, SBS, or failure

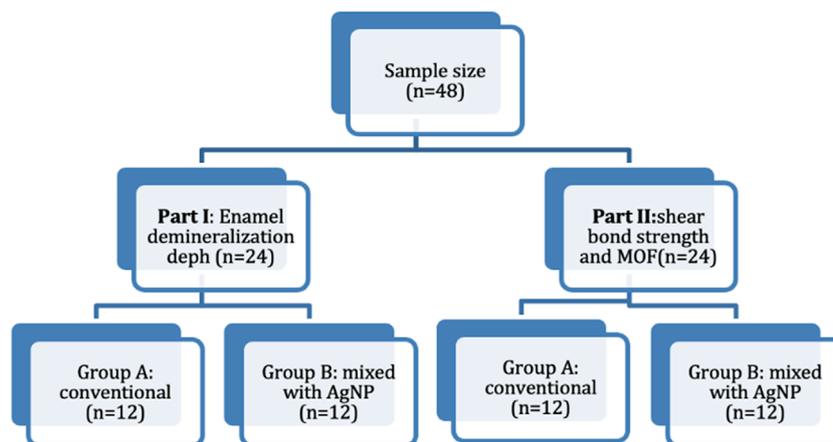


FIGURE 1
Flow chart presenting the study design.

mechanisms between orthodontic adhesives with and without silver nanoparticles.

2 Materials and methods

This study was approved by the Institutional Review Board for Health Sciences Colleges Research on Human Subjects, College of Medicine, King Saud University, Riyadh, Saudi Arabia, and the College of Dentistry Research Center, Riyadh, Saudi Arabia (E-23-8,233).

2.1 Specimen selection and preparation

The sample size was determined using the G*Power calculator (version 3.1.9.6, Franz Faul, Universitt Kiel, Kiel, Germany). A total sample size of 48 specimens was sufficient at (0.05) type I error rate, 0.15 effect size, and 80% power. The study utilized 48 extracted human premolars for orthodontic purposes were collected, with an inclusion criterion of sound teeth without any crack, restoration, or caries.

All specimens were disinfected in a 10% formalin solution for 1 week at room temperature. They were then mounted on acrylic (methyl methacrylate self-cure resin), ensuring that the buccal surface remained exposed for the bonding of orthodontic brackets.

The specimens were simply randomized and evenly divided based on the conducted assessments; Part I: enamel demineralization depth ($n = 24$) and Part II: shear bond strength and mode of failure ($n = 24$). Each part was further categorized according to the adhesive system, Group A included a conventional adhesive system without AgNPs ($n = 12$), and Group B comprised a conventional adhesive system mixed with AgNPs ($n = 12$). The study grouping is illustrated in Figure 1.

2.2 Silver nanoparticles (AgNP) adhesive preparation

Transbond XT orthodontic bonding agent (Transbond XT, 3M Unitek, CA, United States) was mixed with silver nanoparticles (readymade Ag, 99.99% 50–80 nm, w/-0.2% PVP), Stock#: US1018, CAS#:7,440–22-4, US Research Nanomaterials, Inc., Houston, TX, United States). The mixture was prepared using a ratio of 2% AgNP to 98% Transbond XT. It was processed using a mixer at 3500 RPM for 5 min in a dark room to prevent premature photoactivation of the light-sensitive bonding agent.

2.3 Specimen bonding

- Conventional adhesive system (Group A):

The mid-buccal surfaces of the specimens were etched with 37% phosphoric acid (Unitek Etching Gel, 3M, Monrovia, United States) for 20 s, then rinsed with water. After rinsing, the surfaces were air-dried until a chalky, white surface formed. A bonding agent (Transbond, 3M Unitek, CA, United States) was applied using a disposable brush, followed by a gentle airbrust for 2 s, and light-curing for 15 s, according to the manufacturer's instructions.

Metal brackets (3M™ Victory Series™ Low Profile Bracket System, Roth 022, with hooks United States), were then placed using a light-cured adhesive (Transbond XT, 3M Unitek, CA, United States) were used. The brackets were positioned and pressed at the center of the mid-buccal surface, parallel to the long axis of the tooth, using a bracket positioning gauge following the manufacturer's instructions. A lightemitting diode (LED) curing, was then applied for 40 s.

- Conventional adhesive system mixed with AgNP (Group B):

The bonding procedure for this group followed the same steps as Group A but employed the silver nanoparticles-infused adhesive (Transbond XT, 3M Unitek, CA, United States).

2.4 Enamel demineralization depth assessment—Part I

Part I specimens were immersed in 40 mL of an artificial demineralization solution of pH 4.5 at a temperature of 37°C (JSR JSCI-150T General Incubator, Gongju, Korea) for 7 days (Zawawi and Almosa 2023; Buzalaf et al., 2010, Moura et al., 2004,]. The solution was prepared in a 500 mL Erlenmeyer flask, which contained filled with 200 mL of deionized water, 0.166 g of Calcium chloride dihydrate, 0.204 g of disodium phosphate, 25 mL of glacial acetic acid, and 0.154 g of ammonium acetate.

After immersion, brackets were removed using bracket removal pliers (Unitek Debonding Instrument 804–175; 3M, St. Paul, MN, United States), and any excess resin was cleared using a large non-cutting carbide bur with a slow-speed handpiece (FX23 Contra Angle, NSK, Kanuma, Tochigi, Japan).

A low-speed double-sided diamond disk was used for sectioning the teeth buccolingually along the right and left margins of the bracket under continuous water irrigation (0.6-mm ISOMET 2000 Precisions Saw; Buehler, Lake Bluff, IL, United States). The specimens were then subjected to ultrasonic cleaning using a Sonicer machine (Botous Dental, WA, United States).

To enhance image quality, the specimens were gold-coated (1,600 Fine Coat Ion Sputter; JFC, Tokyo, Japan).³¹ The areas of enamel demineralization in each section were captured using a scanning electron microscope (JEOL 6060 LV Scanning Electron Microscope; JEOL) at an accelerating voltage of 15 kV and magnification of $\times 100$. The depth of enamel demineralization was measured in micrometers using ImageJ software (SMile View™, JEOL Ltd., Tokyo, Japan).

2.5 Shear bond strength assessment and mode of failure evaluation—Part II

All specimens designated for Part II were assessed for shear bond strength in megapascals (MPa) using a universal testing machine (Instron, Illinois Tool Works Inc., Norwood, MA, United States) at a speed of 1 mm per minute (mm/min). The mode of failure was examined using a stereomicroscope (SM80, Swift microscope, Carlsbad, CA, United States) and categorized as adhesive, cohesive, or mixed failures.

Adhesive failure mode occurs at the interface between the adhesive and the tooth or bracket, indicating potential issues such as insufficient bond strength or improper tooth etching. In contrast, cohesive failure mode takes place within the adhesive material or the bracket itself, reflecting the inherent properties of the materials used. Mixed failure mode arises when both adhesive and cohesive factors are involved, suggesting an interplay between the bonding methods and the material characteristics.

2.6 Specimen preparation for microhardness and surface roughness tests

The study evaluated microhardness and surface roughness using twenty adhesive resin discs (10 specimens with Transbond XT orthodontic bonding agent (Transbond XT, 3M Unitek, CA, United

States) and 10 specimens with same adhesive mixed with 2% AgNPs). The discs were fabricated using a stainless steel mold, filled incrementally, and covered with a Mylar strip and glass slide. The samples were light-cured using an LED curing unit for 20 s. After polymerization, the discs were polished under running water to maintain consistency and achieve a uniform, smooth surface. This process was used to ensure accurate and reproducible measurements of surface roughness and microhardness.

2.7 Microhardness test

Microhardness testing was conducted using a INNOVATEST microhardness tester (INNOVATEST Europe BV, Bargharenweg 140, 6222 AA Maastricht, Netherlands). To ensure consistent and precise measurement, each specimen was secured in place using a custom polyvinyl siloxane (PVS) putty index (Express™ XT Putty Soft VPS, 3M™, Riyadh, Saudi Arabia), which stabilized the samples throughout the testing process. Microhardness was assessed using a Vickers diamond indenter, applying a load of 100 g for a dwell time of 30 s at each testing point. For every disc, three indentations were performed on the polished surface, spaced 200 μm apart to avoid interference between impressions. The Vickers microhardness (VMH) value for each specimen was calculated as the average of the three measurements, providing a reliable estimate of surface hardness. This protocol was adapted with modifications from previously published methodologies (Al-Angari et al., 2021; Sánchez-Tito and Tay, 2024).

2.8 Surface roughness

Surface roughness (Ra) measurements were carried out using a three-dimensional (3D) optical profilometer (Contour-GT-X®, Bruker Nano Surfaces Division, San Jose, CA, United States), which provides high-resolution, non-contact surface topography analysis. The parameter selected to quantify surface texture was the arithmetic average roughness, expressed in micrometers (μm). This parameter reflects the average vertical deviation of the surface profile from a central mean line, offering a reliable assessment of surface smoothness or roughness. Measurements were performed on all specimens from both experimental groups, with and without silver nanoparticles (AgNPs), to allow for comparative analysis. For each disc, three independent measurements were taken from different locations across the polished surface to account for any surface variability. The mean of these three readings was calculated and recorded as the final Ra value for each specimen, ensuring accuracy and reproducibility in the surface roughness assessment.

2.9 Statistical analyses

The data were analyzed using SPSS software (version 26; IBM, Armonk, NY, United States) at a level of significant $p < 0.05$. Mean values and standard deviations were calculated to describe each output. The normality and equality of variance were tested using Shapiro-Wilk and Levene's tests. Two-way ANOVA was conducted to test the interaction between the different adhesive groups and

TABLE 1 Demineralization depth descriptive statistics and Two-way ANOVA between the adhesive systems and the overall location levels.

Group	Mean \pm SD	Std. Error	p-value
Silver	4.957 \pm 1.457	0.221	0.000
Conventional	6.618 \pm 1.202	0.221	
Demineralization depth descriptive statistics and Two-way ANOVA between the overall location levels			
Location	Mean	Std. Error	p-value
Coronal	5.767	0.271	0.993
Middle	5.783	0.271	
Cervical	5.811	0.271	

location levels of the enamel demineralization outcome. One-way ANOVA was employed to compare the demineralization depth of different locations within the same group. Independent t-test was used to compare the shear bond strength, mode of failure between the different adhesive groups and for the comparison of the mean values of the VMH and Ra values of the test specimens.

3 Results

3.1 Enamel demineralization depth

Normality and equality of variance were satisfied ($p = 0.566$). Group A demonstrated the highest mean values for demineralization depth across various sections: Coronal (6.99 ± 1.22), Middle (6.16 ± 0.97), and Cervical (6.69 ± 1.32). In contrast, Group B exhibited the lowest values in these sections: Coronal (4.54 ± 1.18), Middle (5.40 ± 1.60), and Cervical (4.92 ± 1.53).

The two-way ANOVA test indicated a significant interaction in demineralization depth between the two groups. Specifically, Group A exhibited a statistically significantly deeper demineralization than Group B ($p < 0.05$) (Table 1) (Figure 2A). Figure 3 displays SEM images that capture the depth of demineralization for both adhesive systems. There was no significant difference in overall location levels within the same group (Group A: $p = 0.236$, and Group B: $p = 0.361$) (Table 2). However, when comparing different adhesive systems within the same section, Group B showed shallower demineralization in the coronal and cervical sections compared to Group A ($p < 0.05$) (Table 2) (Figure 2B). In contrast, the middle section did not show a significant difference between the two groups ($p = 0.173$) (Table 2) (Figure 2B).

3.2 Shear bond strength and mode of failure

Normality and equality of variance were satisfied ($p > 0.05$). Group B showed higher mean values compared to Group A, with means of 20.23 ± 1.65 and 17.155 ± 0.94 , respectively. The

independent t-test indicated that the shear bond strength in Group B was significantly greater than that in Group A ($p < 0.05$) (Table 3) (Figure 4).

The overall distribution of the mode of failure revealed that 50% of failures were attributed to adhesive failure, 20.8% to cohesive failure, and 29.2% to mixed failure. In Group B the mode of failure was evenly distributed with 33% for adhesive failure, 33% for cohesive failure, and 33% for mixed failure. In contrast, Group A exhibited a different distribution: 66.7% of failures were due to adhesive failure, 8.3% to cohesive failure, and 25% to mixed failure (Table 4). An image of adhesive failure is shown in Figures 5–A, while Figures 5–B displays cohesive failure.

3.3 Microhardness analysis

The results of the microhardness analysis (Table 5) revealed that the incorporation of AgNPs had a significant impact on the VMH of the adhesive specimens. The mean VMH value for the group without AgNPs was 2.94 ± 3.48 MPa, whereas the group with AgNPs exhibited a higher mean value of 3.11 ± 1.99 MPa. Statistical analysis using an independent samples test demonstrated that this increase in microhardness was significant ($p = 0.000$), indicating that the addition of AgNPs effectively enhanced the surface hardness of the adhesive material.

3.4 Surface Roughness evaluation

In terms of Ra, the specimens containing AgNPs showed a slight increase in the mean arithmetic roughness (Ra) compared to those without (Table 5). The mean Ra value for the AgNPs-containing group was 0.19 ± 0.02 μm , while the non-AgNPs group had a slightly lower value of 0.18 ± 0.03 μm . Despite this observed difference, statistical analysis indicated that the increase in surface roughness was not statistically significant ($p = 0.534$). These findings suggest that while AgNPs addition significantly improves microhardness, it does not markedly alter the surface roughness of the adhesive material.

4 Discussion

Enamel demineralization is a common consequence of orthodontic treatment, which can negatively impact individual aesthetics (Althomali YM, 2017; Benkaddour A et al., 2014). It is essential to implement effective strategies to prevent this issue. Silver nanoparticles (AgNPs) have garnered attention in dentistry and orthodontics due to their distinctive properties and potential benefits. Several studies have indicated that AgNPs possess antibacterial properties, which can help reduce bacterial adhesion and the development of demineralization (Eslamian L et al., 2020; Hernandez-Gomora et al., 2017; Zakarzewski et al., 2021). It is essential to ensure that the addition of silver nanoparticles does not compromise the mechanical properties of orthodontic materials, particularly shear bond strength and the mode of failure. Therefore, this study aimed to assess the enamel demineralization, shear bond

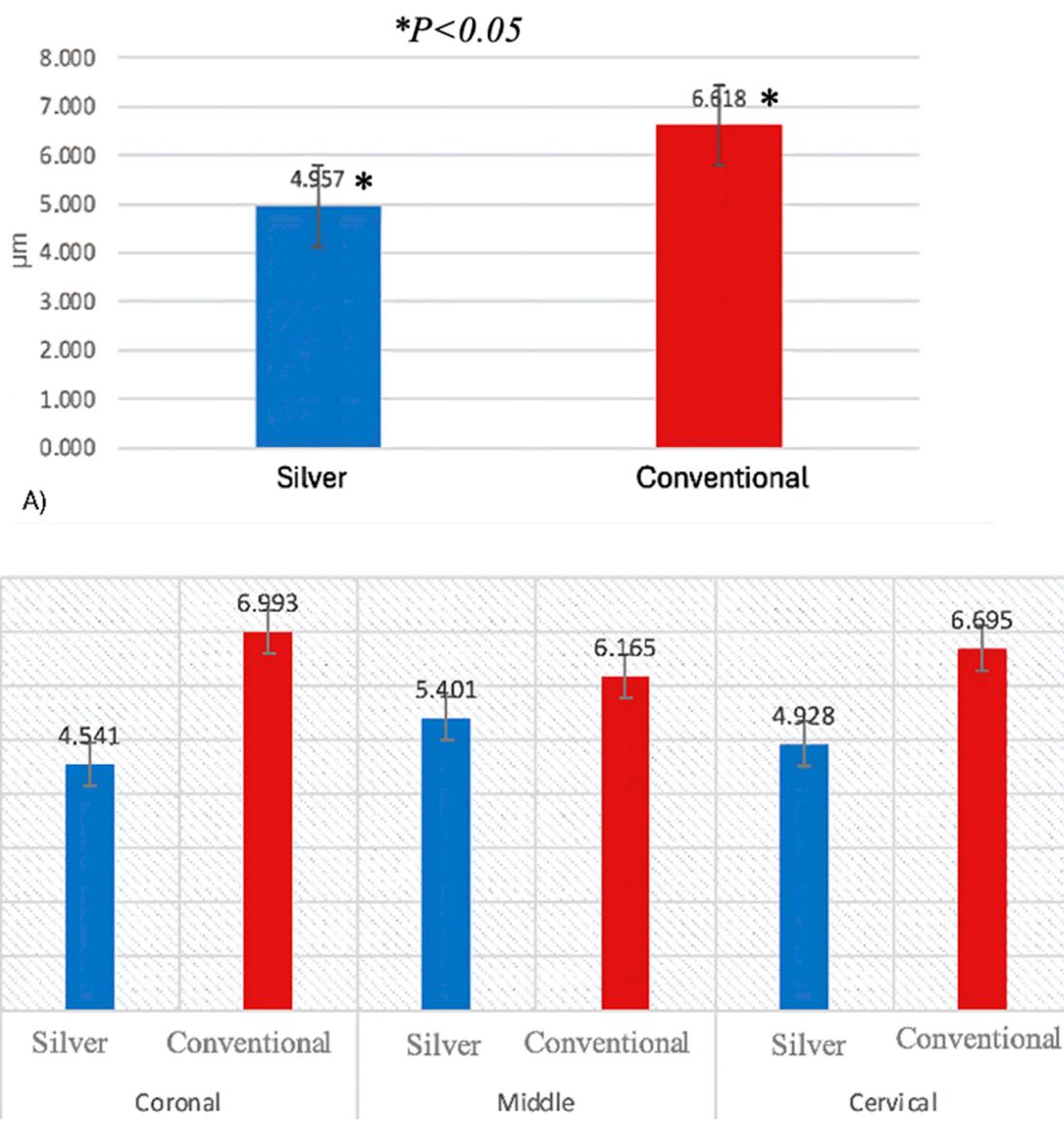


FIGURE 2 (A) Demineralization depth means according to adhesive system. (B) Demineralization depth means according to location levels.

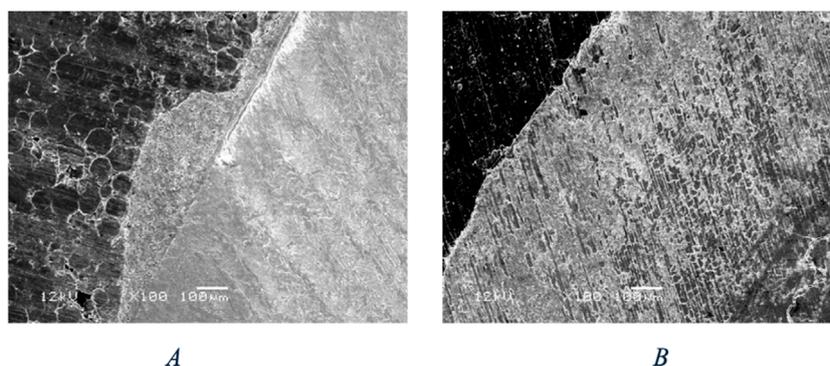


FIGURE 3 Enamel Demineralization depth under SEM. (A) Conventional adhesive (B) Silver nanoparticles-infused adhesive.

TABLE 2 Demineralization depth descriptive statistics and One-way ANOVA comparison at different location levels within the same groups.

Group	Location	Mean ± SD	P-value
Silver	Coronal	4.541 ± 1.186	0.361
	Middle	5.401 ± 1.609	
	Cervical	4.928 ± 1.535	
conventional	Coronal	6.993 ± 1.229	0.236
	Middle	6.165 ± 0.974	
	Cervical	6.695 ± 1.323	

Demineralization depth descriptive statistics and t-test comparison of different location levels between adhesive groups

Location	Group	Mean ± SD	P-value
Coronal	Silver	4.541 ± 1.186	0.000
	conventional	6.993 ± 1.229	
Middle	Silver	5.401 ± 1.609	0.173
	conventional	6.165 ± 0.974	
Cervical	Silver	4.928 ± 1.535	0.000
	conventional	6.695 ± 1.323	

TABLE 3 Shear bond strength descriptive statistics and t-test mean comparison.

Group	Mean ± SD	p-value
Silver	20.239 ± 1.6574	0.0000
Conventional	17.155 ± 0.9484	

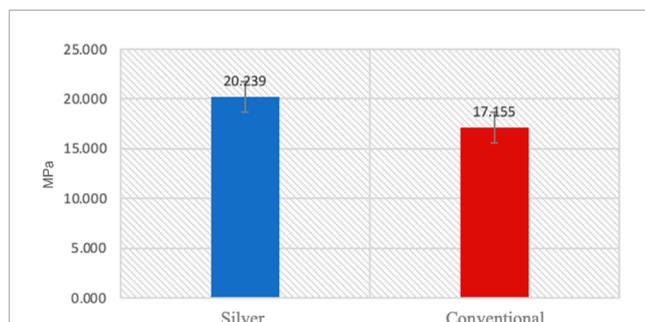


FIGURE 4 Shear bond strength mean according to adhesive system.

strength, and failure modes associated with orthodontic adhesives that contain silver nanoparticles (AgNPs).

Previous studies have assessed the SBS and antibacterial behavior of adhesives containing AgNPs (Eslamian et al., 2020; Al-Thomali 2022; Reddy et al., 2016; Blocher et al., 2015). However, this study is unique in that it simultaneously investigates enamel demineralization, SBS, and modes of failure within a single setting. Additionally, it conducted quantitative assessments using SEM to measure enamel lesion depth, providing a more accurate evaluation of demineralization compared to the qualitative methods used in earlier studies. The adhesive’s preparation process allowed for high-speed standardized mixing, ensuring the uniform dispersion of nanoparticles within the adhesive. This uniformity can enhance the mechanical properties and lead to more consistent failure modes. By employing this integrated approach with high methodological rigor, we gain a broader and more clinically relevant understanding of AgNPs in the context of orthodontic adhesives, thereby advancing the field significantly.

The null hypothesis regarding enamel demineralization was rejected, as the study found that silver nanoparticle adhesive resulted in significantly shallower enamel demineralization compared to conventional adhesive systems. These results are consistent with previous research indicating that silver nanoparticles can prevent enamel demineralization by reducing the acid production generated by biofilms (Eslamian L et al., 2020; Zakarzewski et al., 2021; Allaker, 2010; Espinosa-Cristóbal LF et al., 2009; Espinosa-Cristóbal LF et al., 2018; Espinosa-Cristóbal LF et al., 2012; Chou CW et al., 2006). The antibacterial activity of silver nanoparticles against *Streptococcus mutans* is attributed to their small size and large surface area. Furthermore, the advantages of silver nanoparticles are enhanced by their ability to counterbalance the conventional adhesive nature, which tends to promote the colonization and adherence of microorganisms (Degrazia FW et al., 2016).

This study revealed that the shallow demineralization caused by the silver nanoparticle adhesive was noticeable at the coronal and cervical levels, in contrast to conventional adhesive systems. However, no significant difference in demineralization depth was observed at the middle level between the two types of adhesives. The current literature does not offer a clear understanding of the relationship between the location and the depth of demineralization, which remains to be explored. This finding may be attributed to the fact that the middle part is mostly covered by brackets and is least exposed to the artificial demineralization solution. The artificial demineralization and its limited duration may not accurately reflect the long-term effects of orthodontic adhesives. Additionally, varying the duration of demineralization could either lead to ineffective results or cause excessive erosion of the specimens due to the nature of the chemicals used in the artificial demineralization process. This limitation highlights the need for further studies to evaluate different artificial demineralization protocols that better mimic the clinical long-term effects of orthodontic adhesives.

The null hypothesis regarding shear bond strength was rejected, as a significant increase in shear bond strength was observed in the group using silver nanoparticle adhesive compared to those using conventional adhesives. This enhanced shear bond strength may lead to a reduced risk of leakage, which in turn would result in less leaching of the demineralization solution in an

TABLE 4 Mode of failure distribution.

Test Groups	Count (N) and % within Group	Mode of Failure			Total
		Adhesive	Cohesive	Mixed	
Conventional	N	8	1	3	12
	%	66.6	8.3	25	100
Silver	N	4	4	4	12
	%	33.3	33.3	33.3	100
Overall	N	12	5	7	24
	%	50.0	20.8	29.2	100

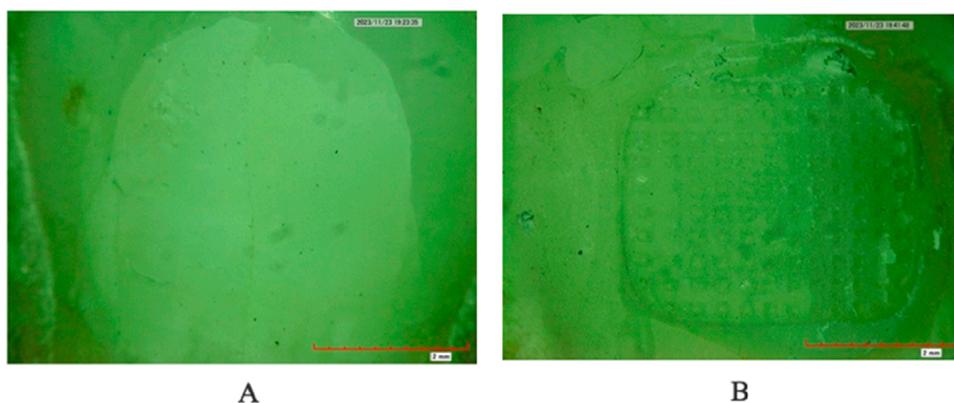


FIGURE 5 Mode of failures captured images. (A) Adhesive failure, (B) cohesive failure.

TABLE 5 Group statistics and comparison of the mean values of the Vickers Micro-Hardness (VMH) and Surface Roughness (Ra) with Independent Samples T-Test for the specimens with or without Silver Nanoparticles (AgNPs).

Test parameters	Specimen	N	Mean	Std. Deviation	Std. Error mean	*Sig. (2-Tailed) P Value
VMH (MPa)	Without AgNPs	10	2.942	3.487	1.102	0.000
	With AgNPs	10	3.114	1.995	0.631	
Ra (µm)	Without AgNPs	10	0.183	0.032	0.010	0.534
	With AgNPs	10	0.191	0.024	0.007	

*P-value was significant at P ≤ 0.05.

in-vitro setting. Consequently, this contributes to the shallower enamel demineralization observed, particularly at the coronal and cervical levels. Al-Thomali (2022) reported that AgNPs significantly increased SBS when a concentration of 0.05% AgNPs was used, which is lower than the concentration used in the present study. In contrast, Eslamian et al. (2020) found that while AgNPs resulted in a reduced SBS, the strength remained above the recommended range of 5.9–7.8 MPa, with their study utilizing 0.3% AgNPs. Furthermore,

another study revealed that conventional adhesives outperform AgNPs in terms of SBS (Reddy et al., 2016). However, another study found no significant impact of AgNPs on SBS (Blocher et al., 2015).

The small particle size and high surface area of AgNPs effectively enhance Van der Waals interactions between positively charged nanoparticles (Hernández-Gómora AE et al., 2017). This helps strengthen the structure of adhesive biomaterials, reduces stress concentrations at the bonding interface by acting as a

stress absorber, enhances mechanical properties, and improves the adhesion layer between dental materials and the enamel surface (Chou CW, et al., 2006; Graig RG & Powers JM, 2002). However, the amount and distribution of the nanofiller particles affect the ultimate adhesive properties; the wide variation of w/w AgNPs incorporation ratio could explain the contradictory findings of AgNPs use on SBS in the literature.

The null hypothesis concerning failure modes was rejected. The AgNP-infused adhesive demonstrated a more balanced distribution of failure modes, whereas the conventional adhesive system showed a higher incidence of adhesive failures. Previous studies reported that cohesive failure modes caused surface damage to teeth, with damage rates of 12.9% and 3.6% resulting from the adhesive resin undergoing cohesive resin failure. (Su MZ et al., 2012; Paolone et al., 2023). Another evaluated failure modes of ceramic brackets bonded to enamel found that most brackets failed at the bracket-adhesive interface (Yetkiner and Ozcan, 2014). Studying failure modes is clinically significant, as it helps identify factors related to complete bracket separation, the amount of remaining adhesive, and the integrity of the enamel, all of which contribute to successful bonding procedures (Borzabadi-Farahani et al., 2014; Su et al., 2012; Jakavice et al., 2023). In orthodontics, it is preferable to have adhesive failure occur between the adhesive layer and the enamel surface. This situation helps maintain proper bracket attachment and ensures safer debonding (Yahya NA & Shekh AM, 2019). Cohesive failure that occurs between the adhesive layer and the back surface of the bracket can lead to a higher incidence of enamel damage. The mode of failure can be influenced by several factors, including the type of surface preparation, the adhesive system used, the material of the bracket, and amount of SBS (Paolone et al., 2023; Chen CS et al., 2008). While a higher bond strength may be desirable, a very high SBS can result in cohesive failures during debonding.

The main concern with AgNPs is their potential cytotoxicity due to oxidative stress and silver ion release. AgNPs can directly generate free radicals through their active surface and produce hydroxyl radicals when dissolved in acidic endo/lysosomes (Chen HY et al., 2007; He W et al., 2012). The impact of AgNPs on shear bond strength (SBS) and demineralization varies with concentration, underscoring the need for comparative studies to optimize their safety and effectiveness. Cost is another limitation, as AgNP-containing adhesives remain expensive. Preparation methods also influence adhesive quality, curing, stress distribution, and SBS (Chou CW, et al., 2006). The mixture preparation method used in this study offers uniform AgNP distribution, enhancing mechanical strength, reducing shrinkage, and minimizing defects, as demonstrated by high SBS values. Exploring alternative preparation methods, such as heat preparation, may further enhance mechanical properties by increasing AgNP temperatures (Metin-Gursoy G et al., 2017). Additionally, discrepancies in SBS results across studies may result from inconsistent artificial aging protocols, such as water aging at 37°C or thermocycling (Chou CW, et al., 2006; Graig and Powers, 2002). The type of bracket material also affects bonding. AlThomali (2017) found that a 0.05% AgNP adhesive achieved higher SBS for metal brackets compared to ceramic brackets, although conventional adhesives were not tested. Further studies should investigate bonding interactions between different bracket materials and AgNP adhesives.

It is essential to acknowledge the differences between laboratory conditions and the clinical environment, as findings from *in vitro* studies may not fully translate to clinical settings. Therefore, antibiofilm analysis and also clinical trials are necessary to better understand the practical implications of these results. While silver nanoparticles (AgNPs) have shown promise in preventing enamel demineralization and enhancing shear bond strength (SBS), further research is required to address several unresolved issues. These include evaluating the potential cytotoxicity of AgNPs, their long-term stability within the adhesive matrix, and the overall clinical performance and durability of AgNP-containing orthodontic adhesives.

Incorporating AgNPs into adhesive systems has shown significant effects on microhardness and surface roughness, indicating both mechanical enhancement and potential surface modifications. Chou et al. (2006) noted that embedding AgNPs into polyurethane matrices improved thermal stability and mechanical properties positively (Chou CW, et al., 2006). This supports the hypothesis that AgNPs strengthens the structural network, which likely contributes to increased microhardness in dental adhesives. Similarly, Hernández-Gómora et al. (2017) reported that AgNPs biosynthesized onto orthodontic elastomeric modules enhanced mechanical resistance while also exhibiting antimicrobial activity (Hernández-Gómora AE et al., 2017). The findings of this study demonstrate that the incorporation of silver nanoparticles (AgNPs) into the adhesive resin formulation significantly enhanced its microhardness without causing a statistically significant increase in surface roughness. The observed improvement in VMH may be attributed to the reinforcing effect of AgNPs, which are known to improve the mechanical properties of polymer-based materials by increasing filler content, reducing polymer chain mobility, and promoting better stress distribution within the matrix. This enhancement in surface hardness could potentially lead to greater resistance to wear and mechanical degradation in clinical applications, particularly in stress-bearing areas.

Conversely, although the Ra values showed a slight increase in the presence of AgNPs, this difference was not statistically significant. This suggests that the addition of AgNPs at the tested concentration does not adversely affect the smoothness of the adhesive surface, an important consideration for maintaining aesthetic quality and minimizing plaque accumulation in oral environments. Taken together, these results suggest that AgNP incorporation could be a promising strategy to enhance the mechanical performance of dental adhesives, particularly when increased hardness is desirable. Moreover, the fact that surface roughness remained unaffected supports the potential clinical viability of such formulations, as smooth surface characteristics are essential for both esthetics and biofilm resistance (Holban AM et al., 2021).

The chemical interaction between resin and AgNPs has the potential to induce minor surface chemistry and phase dispersion changes, which would influence the mechanical performance and antimicrobial activity of the adhesive (Chou et al., 2006; Degrazia et al., 2016). Thus, it is essential for future studies to evaluate the effects of AgNPs on the structural, chemical, and phase transition characteristics of adhesive materials. Utilizing techniques such as Fourier Transform Infrared

Spectroscopy (FTIR), and X-ray diffraction will be important in these evaluations, to have a better understanding on the changes that AgNPs makes on orthodontic adhesives. Furthermore, future clinical studies that would cover a wider range of variables and more comprehensive assessments is needed.

5 Conclusion

- Orthodontic adhesives infused with silver nanoparticles (AgNPs) demonstrate promising advantages over conventional adhesives, including reduced enamel demineralization and enhanced shear bond strength. However, conventional adhesives show a higher incidence of adhesive mode failure, which may offer safer bracket removal.
- Incorporating AgNPs presents a valuable approach to minimizing enamel demineralization during orthodontic treatment. Nevertheless, further research is essential to determine the optimal AgNP concentration that balances antibacterial efficacy and mechanical stability over the long term.
- The addition of AgNPs significantly improved the microhardness of dental adhesives without affecting surface roughness. These results suggest that AgNPs may enhance the mechanical properties of adhesives while maintaining clinical surface quality.
- Despite their potential benefits, the economic feasibility of AgNP-containing adhesives remains a critical barrier to widespread adoption. Addressing this challenge will be crucial to making this innovative solution more accessible and practical for clinical use.

Data availability statement

The raw data supporting the conclusions of this article will be made available by the authors, without undue reservation.

Ethics statement

This study was approved by the Institutional Review Board for Health Sciences Colleges Research on Human Subjects, College of Medicine, King Saud University, Riyadh, Saudi Arabia, and the College of Dentistry Research Center, Riyadh, Saudi Arabia (E-23-8233).

Author contributions

NiA: Writing - original draft, Conceptualization, Validation, Project administration, Data curation, Supervision. AhA:

Methodology, Data curation, Writing - original draft, Investigation. AIA: Software, Visualization, Formal Analysis, Data curation, Writing - original draft, Resources, Funding acquisition, Conceptualization, Project administration, Investigation, Supervision, Validation, Methodology. NdA: Writing - original draft, Investigation, Methodology, Data curation. AbA: Investigation, Data curation, Methodology, Writing - original draft. LA: Data curation, Writing - original draft, Methodology. RZ: Investigation, Writing - original draft, Methodology. SH: Formal Analysis, Software, Funding acquisition, Writing - review and editing.

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Conflict of interest

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