Shear bond strength of orthodontic brackets after adding silver nanoparticles to a nano-bond adhesive at different thermal cycles and cyclic loading- An *in vitro* study

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Abstract

OBJECTIVE: To evaluate the effects of silver nano particles (AgNPs) added on Nano-Bond adhesive system (NBA) and its effect on shear bond strength (SBS) of orthodontic brackets attached to enamel at different thermal cycles and cyclic loading.

MATERIALS AND METHODS: Forty extracted premolar teeth for orthodontic reasons were divided randomly into two main groups (20 teeth in each group). Group A: the brackets were bonded by NBA without additives. Group B: the brackets were bonded by NBA containing AgNPs with concentration 0.05%. Every group was further subdivided into 4 subgroups according to teeth subjected or not subjected to thermocycling and cyclic loading. SBS was tested using Lloyd universal testing machine. One-way analysis of variance (ANOVA) was used for testing the significance between the means of tested groups.

RESULTS: Shear bond strength of Nano-Bond adhesive system with AgNPs showed significantly highest mean SBS (20.25 MPa) than Nano-Bond adhesive system without additives (15.64 MPa, *P* = 0.001). The SBS increased in Group B with AgNPs compared to Group A in all the conditions tested. Group A1 and B1 with zero thermal cycling and cyclic loading exhibited highest mean SBS.

CONCLUSIONS: These results indicate addition of AgNPs significantly increased the shear bond strength of Nano-Bond adhesive system.

Keywords:

AgNPs, cyclic loading, nano-bond adhesive, orthodontic brackets, thermal cycling

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Introduction

Decades since their entrance into the world of orthodontics, the first choice of many orthodontists for binding brackets remains without question composite resin adhesives.[1,2] One of the crises the orthodontists may face during treatment is bracket failure. Bracket failure occurs in 0.6% to 28.3% of cases in which light- or chemical-cured composite resins are used, usually due to improper force or poor bonding technique, exposure to thermal fatigue and cyclic loading intra-orally, and development of caries around the bonded teeth.[3-5] However, the addition of nano bonding agents can improve the adhesion properties of composite resins.[6] Recently, multiple bonding agents have been developed, including antibacterial adhesives to inhibit the bacterial biofilm accumulation at the bracket margins.[7,8] Antibacterial adhesives are promising to combat bacteria and reduce recurrent caries at the tooth-restoration margins.[9] Use of

silver nano particles (AgNPs) as antibacterial adhesives gained popularity in recent times due to its nano size, increased surface area, excellent antimicrobial properties, and low antibacterial resistance.[10,11] However, the recent systematic review of in-vitro studies showed addition of antimicrobial agents to orthodontic adhesive system does not influence the bond strength of enamel.[9] The present in-vitro study was conducted with an aim to evaluate the shear bond strength of the attachment between brackets and enamel with and without incorporation of AgNPs to a nano bond adhesive system after subjecting to different thermo cycling and cyclic loading.

Materials and Methods

Tooth preparation and study subgroups

The study involved 40 caries-free human maxillary premolar teeth that had been freshly extracted as part of courses of orthodontic treatment. The teeth were cleaned using an ultrasonic scaler and stored in distilled water at 37°C.

The teeth were randomly divided into two groups of 20 (A and B). Nano-Bond adhesive (Pentron Clinical Technologies LLC, Orange, CA, USA) without additives was used for Group A (the control group) while the Nano-Bond adhesive with 0.05% AgNPs was used for Group B. Both groups were divided into four subgroups, each containing five teeth. The teeth were subjected to subgroup-specific thermal and loading stress, as shown in Table 1. The bracket bonding procedure was based on acid etching the enamel, following the procedure developed by Buonocore in 1955,[12] using phosphoric acid gel at a concentration of 37% (Eco-Etch, IvoclarVivadent AG, Schaan, Liechtenstein). The enamel was then rinsed with water and dried with oil-free stream for five s. Primer was applied to the etched surface using an applicator brush the excess primer was removed using a dry applicator brush, but the surface was left with a very wet appearance. The enamel was then light-cured for 10 s using a 430-490 nm light-emitting diode (LED; BG Light Ltd, Plovdiv, Bulgaria).

Application of nano-bond adhesive and brackets

The Nano-Bond adhesive was applied to the entire enamel surface in accordance with the manufacturer's instructions. Dry air was then gently applied for 15 s to disperse the material to produce a thin, uniform, shiny surface. The adhesive was subsequently cured for 10 s using the LED. The base of each metal orthodontic bracket (0.022 x 0.028 inch; Global Orthodontics LLC, McLean, VA, USA) was filled with allowable composite resin (Flow-It ALC, Pentron Clinical Technologies LLC) and firmly pressed onto the surface of the tooth. The excess resin was removed and the resin was cured for 40 s (i.e. 20 s for the mesial and distal sections,

respectively) using the LED (placing the LED as close as possible to the tooth). A LI-189 Quantum/Radiometer/Photometer (LI-COR Biosciences Inc., Lincoln, NE, USA) was used to ensure that the light intensity was constant throughout the polymerization process. The teeth were then stored at 37°C in distilled water for 24 h before thermocycling and cyclic loading.

Thermocycling

The thermocycling procedure involved placing the teeth in a water reservoir [Figure 1] and subjecting them to 5°C for 30 s and then to 55°C for 30 s. The number of thermal cycles that the teeth in each subgroup were subjected to is shown in Table 1.

Cyclic loading

The number of loading cycles that the teeth in each subgroup were subjected to is shown in Table 1 (100,000, 200,000, and 400,000 cycles were used to simulate six-months, one-year, and two-years of physiological occlusal stress, respectively). AnLRX plus II SERIES Materials Testing Machine (Lloyd Instruments Ltd., Fareham, UK) was used to apply a load to the middle of each tooth surface at an angle of 45° to the tooth's long axis using a steel rod with a diameter of 0.8 mm. The process involved load cell of 5 kN, a force of 90 N, and a load profile in the form of a sine wave with a rate of 1 Hz (which is equivalent to the normal masticatory

Table 1: Study subgroups according to thermo cycling and loading methods

Group	Subgroup	Number of cycles	
		Thermal	**Loading**
A	A1	0	0
	A2	500	100,000
	A3	1000	200,000
	A4	2000	400,000
B	B1	0	0
	B2	500	100,000
	B3	1000	200,000
	B4	2000	400,000

Group A: the brackets were bonded by NBA without additives. Group B: the brackets were bonded by NBA containing AgNPs with concentration 0.05%

Figure 1: Thermocycling sample

cycle rate of 0.8–1.0/s). The data were recorded using NEXYGEN-MT version 4.5.1 software (Lloyd Instruments Ltd.).

Shear bond strength testing

The shear bond strength testing was carried out using the LRX Plus II. A specially designed upper attachment knife-edge was used. Prior to testing, the teeth were embedded in chemically cured modelling acrylic (Palavit G, Heraeus Kulzer GmbH, Wertheim, Germany) in plastic cylinders to allow secure, standardized placement during the tests. The plastic cylinders were mounted on the lower attachment and the teeth were positioned to ensure that the application point and direction of the force were consistent. The load was applied perpendicular to the interface between the tooth and bracket [Figure 2], using a cross-head speed of 0.5 mm/min until deboning occurred. The loads at bracket failure were recorded. The shear bond strength (kg/cm^2) was calculated using the following formula: $\sigma_s = P/\pi \cdot r^2$, where σ_s are the shear bond strength (kg/cm^2), P is the shear load (kg), π equals 3.14, and r is the tooth radius (cm). The shear bond strength was converted to MPa by multiplying σ_s by 0.09807.

Statistical analysis

The data were analysed using one-way analysis of variance (ANOVA), an omnibus test. Post-hoc Tukey's tests were then used to find which between-group differences in the means were statistically significant. $P \leq 0.05$ was considered statistically significant. All the tests were two sided.

Results

Table 2 shows the comparisons of the mean shear bond strength (SBS) between Nano-Bond adhesive with no additives and Nano-Bond adhesive containing 0.05% AgNPs. Group B1 had a significantly greater mean SBS (20.25 MPa) compared to group A1 (15.64 MPa; $P = 0.001$). Group B2 had a significantly greater mean SBS (16.75 MPa) compared to group A2 (12.75 MPa; $P = 0.001$). Group B3 had a significantly greater mean SBS (12.96 MPa) compared to group A3 (9.26 MPa; $P = 0.001$). Lastly, group B4 had a significantly greater mean SBS (9.21 MPa) compared to group A4 (5.98 MPa; $P = 0.001$). Thus, in each comparison, the mean SBS was greater for the adhesive containing 0.05% AgNPs. The mean SBS decreased with increasing thermal cycling and cyclic loading in both the groups.

Discussion

Bracket deboning due to masticatory forces, poor bonding technique, bracket bases with low retentive capacity, and small bracket bases (selected for aesthetic
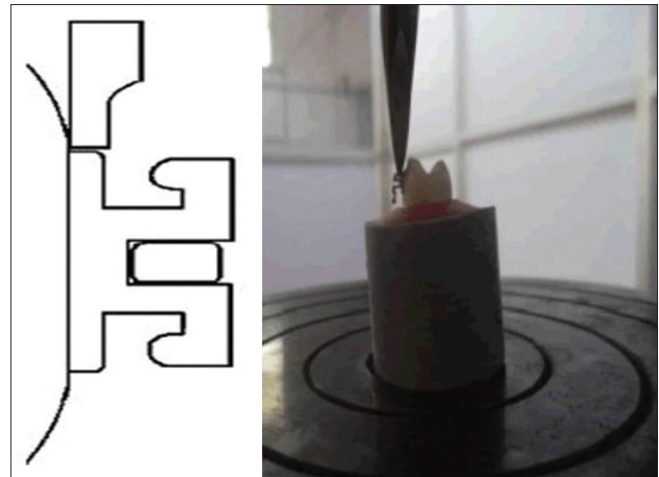

Figure 2: Diagram and photo of force application during the shear bond strength tests

Table 2: Comparison of shear bond strengths (MPa) between different nano adhesive groups

Subgroups compared	Mean (SD)		ANOVA F	ANOVA P
	Group A	Group B		
A1 and B1	15.64[b] (0.96)	20.25[a] (0.56)	17.382	0.001*
A2 and B2	12.75[b] (0.43)	16.75[a] (0.87)	13.237	0.001*
A3 and B3	9.26[b] (0.54)	12.96[a] (0.71)	15.218	0.001*
A4 and B4	5.98[b] (0.08)	9.21[a] (0.16)	15.321	0.001*
ANOVA F	7.214	8.237		
ANOVA P	0.032	0.041		
Tukey post hoc	A 1>A4, A3	B1>B4, B3		

[a/b]$P \leq 0.001$ according to post-hoc Tukey's tests, ANOVA: Analysis of variance; SD: Standard deviation, Group A: The brackets were bonded by NBA without additives. Group B: The brackets were bonded by NBA containing AgNPs with concentration 0.05%, A1, B1=zero thermal or loading cycles, A2, B2=500 thermal and 100,000 loading cycles; A3, B3=1000 thermal and 200,000 loading cycles; A4, B4=2000 thermal and 400,000 loading cycles

reasons) is a common issue in orthodontics, and it can increase the duration and cost of treatment.[3-5] Several solutions have been proposed to minimize bracket deboning, including aluminium oxide sandblasting of the bracket base and the use of primers.[6,13,14] In addition, a study has shown that adding nano bond adhesive resin improves the mechanical properties and the marginal and internal seal of composite restorations.[15] This occurs because of changes in the stress dynamics at the interface between the enamel and resin, which reduces crack initiation and propagation.

This study measured the strength of the attachment between brackets and enamel using shear bond strength tests. These tests are widely used to evaluate the performance of orthodontic bonding systems, and a variety of techniques have been developed for this purpose.[16] The tests are extensively used because of their relative simplicity compared to tensile bond strength tests (which require the specimens to be carefully aligned in the testing machine in order to avoid detrimental stress

distributions.[17,18] However, the lack of standardization of the shear bond strength tests and the large distribution of results has often prevented firm conclusions from being drawn from the results of previous studies.[9]

The present study tested the effects of adding 0.05% AgNPs to adhesive on shear bond strength because AgNPs is a useful addition to adhesives as a result of its potent antibacterial properties.[10,11] The smaller particle size and larger surface area-to-volume ratio of AgNPs enable more Ag^+ to be released at a low filler level, thereby reducing the Ag concentration required for efficacy.[9-11] The results demonstrated that the shear bond strength of Nano-Bond adhesive increased significantly when 0.05% AgNPs was added. This is likely to be due to the inherent characteristics of the silver nano particles, as they are able to form strong ionic bonds. The use of low concentrations of AgNPs in bonding agents improves their thermal and mechanical properties, as well as their bio stability and antibacterial properties, which can reduce the interfacial stress concentration within the adhesive-resin complex.[10] The researchers in previous studies used AgNPs concentration of 1%,[19] 0.3%,[20] and 0.11% to 0.33%[21] which was higher compared to the present study, however the authors concluded higher concentration of AgNPs leads to reduced shear bond strength due to agglomeration of NPs inside the primer, which in turn create defects points and interfere with the curing process.[19-21] The recent meta-analysis by Khursheed-Alam *et al*.[22] showed higher concentration of AgNPs reduced SBS of orthodontic adhesives.

In the present study the specimens were subjected to different levels of thermal cycling and cyclic loading. Physiological changes in intraoral temperature can affect the mechanical properties of the adhesive layer, leading to thermal stresses in the adhesive layer, teeth surface, and brackets.[23] Thermocycling is the *in vitro* process of subjecting restorations and teeth to temperature extremes to simulate the thermal stress conditions in the oral cavity.[24] Thermocycling with cyclic loading simulates the physiological occlusal stress conditions in the oral cavity.[25] The present study result showed decreased shear bond strength in both the groups with increasing thermal cycles and cyclic loading. The result are in agreement with previous studies which showed decreased bond strength between orthodontic brackets and enamel surface with increased thermal cycling and cyclic loading.[23,24]

In the present study, the specimens were acid etched using phosphoric acid gel at a concentration of 37%. Acid etching cleans the enamel surface and increases its wettability. The acid also removes calcium salts from the enamel and causes selective dissolution of enamel rods, leading to an increase in the size and number of micro

pores, into which low-viscosity resins can easily flow. When resin is applied to an acid-etched enamel surface, it can penetrate into the microspores, thus producing 'resin tags' (finger-like projections) that increase the bond strength and reduce marginal discoloration.[26]

Conclusion

The study showed that adding 0.05% AgNPs to Nano-Bond adhesive increased shear bond strength in different thermal cycling and cyclic loading. Further studies are needed to investigate the effects of AgNPs on physical properties other than the shear bond strength, and the effects of low concentration of AgNPs on oral microbes.

Conflicts of interest

There are no conflicts of interest.

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