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Original Article

Effect of the addition of silver nanoparticles on the mechanical properties of an orthodontic adhesive

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ABSTRACT

Objectives: This study evaluated the effects of adding silver nanoparticles on the shear bond strength, microhardness, and surface roughness of orthodontic adhesives.

Material and Methods: Fifty upper premolars were randomly allocated to five groups ($n = 10$). Orthodontic brackets were bonded with silver nanoparticle (AgNP)-modified adhesives (1 %, 0.5 %, 0.1 %, 0.05 %), and conventional adhesive was used as a control. The shear bond strength was recorded using a universal testing machine, and the adhesive remnant index was evaluated using a stereomicroscope. Ten discs of each adhesive were subjected to the microhardness and surface roughness tests. The Vickers microhardness values were measured under a constant load of 100 g for 30 s using a microhardness tester. The samples were analyzed using a surface profilometer, and the arithmetic average roughness was used as the measurement parameter. Data were analyzed using one-way analysis of variance and chi-square tests. A significance level of 5 % was considered significant.

Results: AgNP concentration > 0.1 % significantly reduced the shear bond strength ($p < 0.05$). At higher AgNP concentration, the bonding failure pattern occurred mainly at the bracket-resin interface. The Vickers microhardness increased with increasing concentration, and significant differences were observed between the group with 1 % AgNP and the other groups ($p < 0.05$). The average roughness values were similar between the groups with AgNP concentrations > 0.1 % ($p > 0.05$).

Conclusion: The incorporation of AgNP into an orthodontic adhesive has the potential to decrease the shear bond strength while increasing the microhardness and surface roughness.

1. Introduction

The inclusion of antibacterial agents in orthodontic adhesives has recently been proposed to control bacterial colonization and prevent the formation of white spot lesions (Altmann et al., 2016; de Almeida et al., 2018; Poosti et al., 2013). In particular, silver nanoparticles (AgNP) have exhibited significant antibacterial activity against cariogenic bacteria (Sánchez-Tito and Tay, 2021; Yassaei et al., 2020). Additionally, the anti-adherence properties of AgNP have been tested, and they are effective in inhibiting the adhesion of *S. mutans* on the surface of orthodontic materials (Nafarrate-Valdez, et al., 2022).

Despite the important antibacterial and anti-adherence properties of AgNP, some studies have reported that addition of AgNP to orthodontic adhesives may be associated with a reduction in the bond strength required to keep the brackets adhered to the enamel surface. However, there are still contradictory results regarding this issue. Orthodontic adhesive modified by the addition of 1 % AgNP reportedly reduces the shear bond strength (SBS) compared to the unmodified adhesive (Reddy et al., 2013). The SBS of orthodontic adhesives may decrease after the incorporation of 0.3 % AgNP (Eslamian et al., 2020). However, the addition of a combination of AgNP and hydroxyapatite at concentrations of 5 % and 1 %, respectively, to an orthodontic adhesive maintained and increased SBS values (Akhavan et al., 2013).

The amount of filler in the adhesive matrix may influence SBS because a greater amount of filler provides greater bond strength (Faltermeier et al., 2007). Additionally, the amount of filler is directly correlated with the microhardness value observed in orthodontic adhesives (Vilchis et al., 2008). The wear of adhesives or resins in the oral cavity may be due to natural causes or as a result of tooth brushing, which increases surface roughness and consequently allows the

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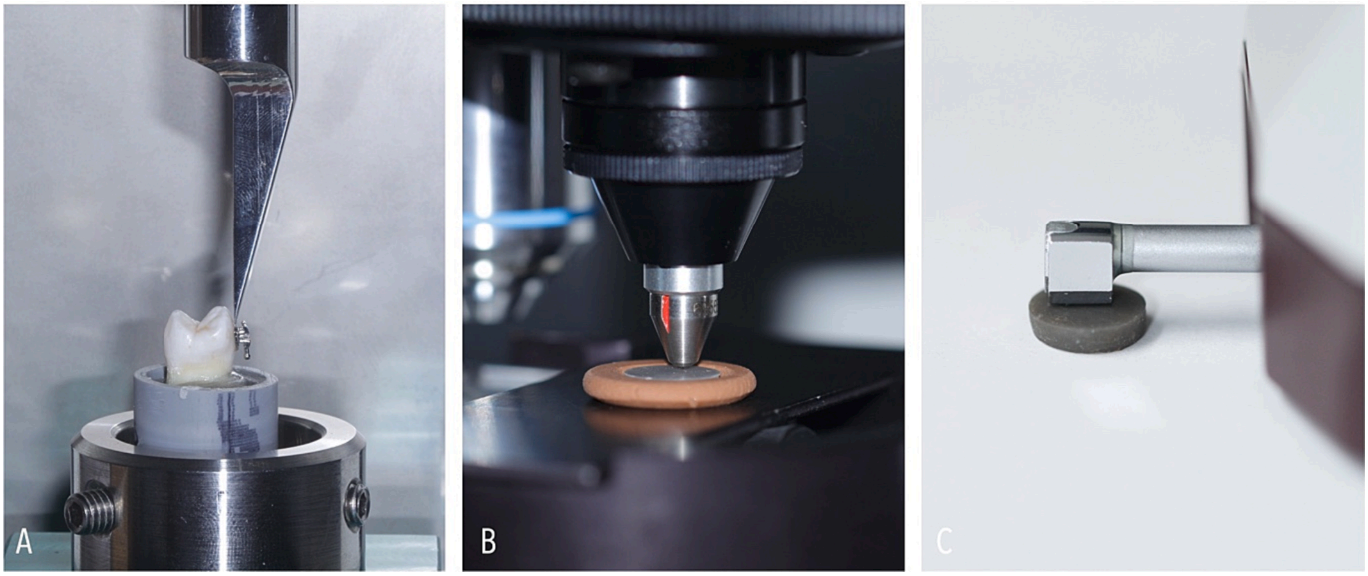

Fig. 1. Details of the tests carried out: (A) shear strength, (B) Microhardness, (C) Surface roughness.

colonization of cariogenic bacteria (Ahn et al., 2010; O'Kane et al., 1993).

Considering the interest in the use of AgNP in orthodontic adhesives and the controversial results regarding their effect on the mechanical properties, the objective of this study was to evaluate the effect of the addition of AgNP on the mechanical properties of orthodontic adhesives. The null hypothesis was that adhesives with different concentrations of AgNP present SBS, microhardness, and roughness values similar to those of the unmodified adhesive.

2. Material and methods

2.1. Ethical consideration and sample size calculation

This study was approved by the Institutional Ethics Committee (no. 310–15-19). Sample size calculation was conducted using the software G*Power 3.1.3 (Heinrich Heine Universität, Düsseldorf, Germany), employing a one-way analysis of variance (ANOVA) test with fixed effects. A medium effect size $\eta_p^2 = 0.06$ (Cohen, 1992), an $\alpha = 0.05$, and a power of 0.8 were adopted. The estimated sample size was 10 for each group.

2.2. Preparation of the experimental adhesive

Four experimental adhesives were prepared by mixing the AgNP (average particle size: 20 nm; US Research Nanomaterials Inc. Houston, Tx, USA) with Transbond XT (3 M Unitek, Monrovia, California, USA). Experimental adhesive (3 g) was prepared by adding AgNP at concentrations of 1 %, 0.5 %, 0.1 %, and 0.05 % (wt/wt). The mixture was prepared using a glass slab and plastic spatula for 2 min in a dark environment to avoid inadvertent polymerization of the adhesive.

2.3. Specimen preparation

Fifty upper premolars with sound enamel were collected for the SBS testing. The teeth were embedded in PVC cylinders (20 mm inner diameter × 15 mm height) containing a self-curing acrylic resin (VIPI®, Pirassununga, SP, Brazil), with the buccal surface of the teeth perpendicular to the base of the cylinder. The enamel was etched with 37 % phosphoric acid gel (Unitek Etching Gel; 3 M, Monrovia, CA, USA) for 30 s (Sodagar et al., 2016), rinsed with water for 60 s, and dried with oil-free compressed air. The specimens were randomly divided into five groups according to the concentration of AgNP.

The primer (Transbond XT Primer; 3 M Unitek, Monrovia, CA, USA) was applied and photopolymerized for 20 s (Elipar DeepCure-L unit; 3 M Unitek, Monrovia, CA, USA). The experimental adhesive was applied to the base of premolar metal brackets (Gemini, 3 M Unitek, Monrovia, CA, USA). The brackets were positioned at the center of the crown, and a constant force of 250 gf was applied for 20 s to ensure uniform adhesive thickness (Poosti et al., 2013). Excess adhesive was removed using a scale and polymerized for 40 s (10 s for each side of the bracket) (Altmann et al., 2017). The specimens were stored in distilled water for 24 h at 37 °C prior to the SBS test.

For microhardness and surface roughness tests, 50 adhesive discs (diameter: 8 mm; thickness: 2 mm) were prepared. The samples were then photopolymerized for 20 s. The discs were polished using a sequence of #600, #800, #1000, and #1500 grit silicon carbide papers.

2.4. Shear bond strength test

The specimens were placed on a universal testing machine (Odeme; OM 150, São Carlos, Brazil), and a knife-edge chisel was positioned at the bracket-tooth interface at a crosshead speed of 1 mm/min (Eslamian et al., 2020) (Fig. 1A). The results were recorded in kgf and converted into newtons (N). SBS was presented in megapascals (MPa), dividing the values in Newtons by the area of the bracket base (10.62 mm^2), as reported in a previous study (Turk et al., 2007).

2.5. Adhesive remnant index

The teeth were assessed under 20 × magnification (AmScope$^{\text{TM}}$, ZM-3B/T, United Scope LLC, California, USA), and the adhesive remnant index (ARI) score was recorded (Artun and Bergland, 1984). Briefly, the ARI scores were classified as follows: 0 = no resin remaining on the tooth surface, 1 = less than half of the resin remaining on the tooth surface, 2 = more than half of the resin remaining on the tooth, and 3 = the entire resin remaining on the tooth with a distinct impression of the bracket mesh.

2.6. Microhardness test

The microhardness was evaluated using an LM248AT machine (LECO, St. Joseph, MI, USA). Three indentations were made 200 µm apart. The samples were pressed onto a thin layer of plasticizing material

Table 1
One-way ANOVA results for shear bond strength, surface microhardness, and surface roughness.

Group	SBS	VMH	Ra
1 % AgNP	16.40 ± 0.74^{a}	122.5 ± 6.83^{a}	0.429 ± 0.014^{a}
0.5 % AgNP	16.92 ± 0.97^{ab}	92 ± 2.58^{b}	0.412 ± 0.096^{ab}
0.1 % AgNP	17.32 ± 0.88^{ab}	74.1 ± 3.72 ^{cd}	0.422 ± 0.016^{ab}
0.05 % AgNP	17.72 ± 0.75^{b}	68.9 ± 2.28^{de}	0.409 ± 0.013^{bc}
0 % AgNP	17.85 ± 0.76^{b}	67.3 ± 2.83^{e}	0.399 ± 0.017^{c}

Values are presented as the mean ± standard deviation. Different superscript letters in a row indicate significant differences, as determined by multiple comparisons using the Bonferroni test ($P < 0.05$).
SBS, shear bond strength; VMH, Vickers microhardness; Ra, average roughness; ANOVA, one-way analysis of variance.

(Plastalina; Van Aken®, North Charleston, SC, USA) during the observation stage. Indentations were made using a Vickers diamond indenter with a load of 100 g for 30 s (Fig. 1B). The measurements were averaged to obtain Vickers microhardness (VMH) values for each sample (Al-Angari et al., 2021).

2.7. Surface roughness

The samples were analyzed using a surface profilometer Surftest SJ-210 (Mitutoyo, Kawasaki, Japan) (Fig. 1C). The arithmetic average roughness (Ra) was used as a measurement parameter. Five measurements were performed on the disc surface, and the mean values were calculated. A sampling length of 0.8 mm, a cut-off of 0.25 mm, and a measurement speed of 0.25 mm/s were applied (Porojan et al., 2021).

2.8. Statistical analysis

Data were analyzed using Stata software package (version 17.0; StataCorp, College Station, TX, USA). For the descriptive analysis, the mean, standard deviation, and frequency measures were used. Normal distribution and homogeneity of variances were confirmed using the Shapiro–Wilk and Bartlett tests, respectively. ANOVA followed by the Bonferroni post-hoc test was used to compare parameter values between groups. The chi-square test for homogeneity was used to assess the differences in ARI scores. A significance level of 5 % was set for all statistical tests.

3. Results

Table 1 summarizes the results for SBS, VMH, and Ra. One-way ANOVA showed a significant difference in the SBS between all groups ($p < 0.05$). The addition of AgNP to the orthodontic adhesive decreased SBS in all groups. No significant differences were observed in the reduction of SBS among the experimental groups with AgNP concentrations of 1 %, 0.5 %, and 0.1 %. The ARI scores in the group with 1 % AgNP were mainly distributed between scores of 0 and 1; thus, the pattern of bonding failure occurred mainly in the bracket-resin interface. ARI scores of 1 and 2 were more frequent in the 0.5 % and 0.1 % groups, respectively. For the 0.05 % AgNP and control groups, the most frequent ARI score was 2 (Fig. 2). The chi-square test showed significant differences in ARI scores among the groups ($p < 0.05$) (Table 2). Representative fracture failure patterns are shown in Fig. 3. VMH increased as the concentration of AgNP increased, and significant differences were found between the 1 % group and the other concentrations ($p < 0.05$) (Table 1). In contrast, the Ra values were similar

Table 2
Comparison of adhesive remnant index (ARI) between groups.

Group	ARI Scores				P-value*
	0	1	2	3	
1 % AgNP	5	4	1	0	<0.001
0.5 % AgNP	1	5	4	0	
0.1 % AgNP	0	4	6	0	
0.05 % AgNP	0	0	8	2	
0 % AgNP	0	0	7	3	

* Chi-square test for homogeneity.

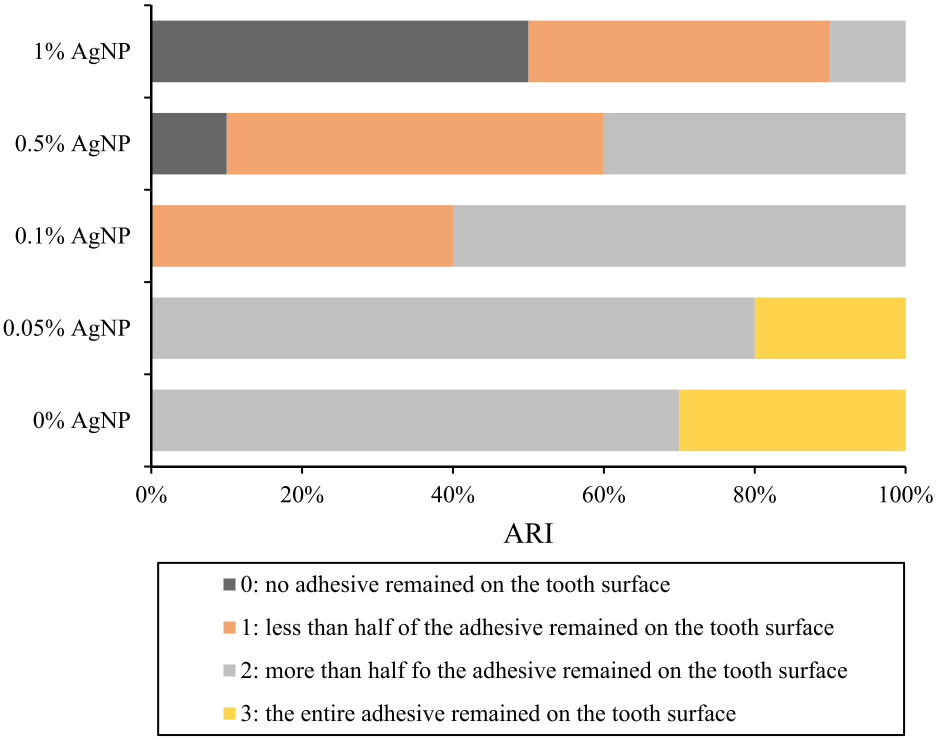

Fig. 2. Frequency and distribution of adhesive remnant index (ARI).

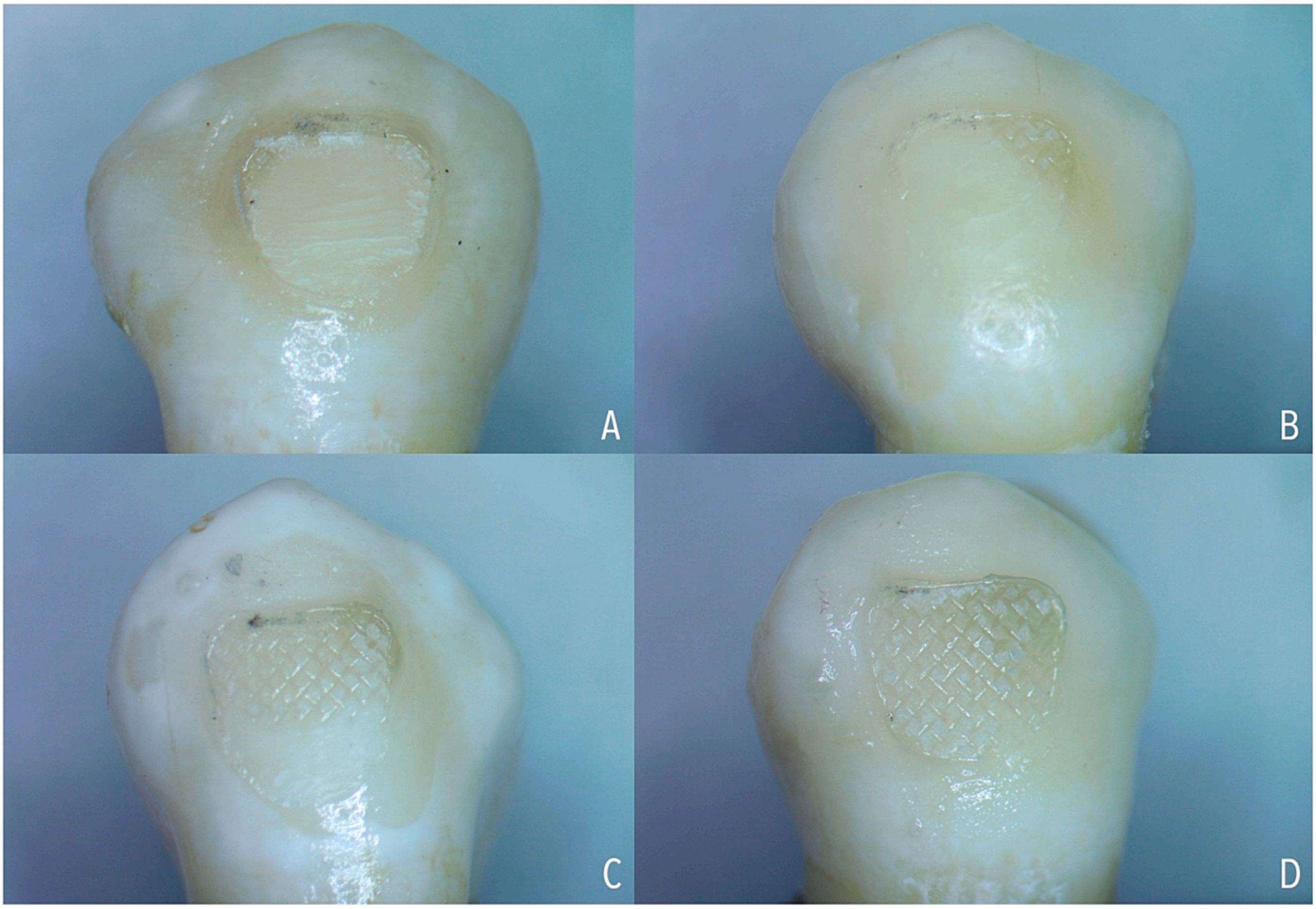

Fig. 3. Representative sample according to IRA. **A.** IRA 0 = no resin remaining on the tooth surface. **B**. IRA 1 = less than half of the resin remaining on the tooth surface. **C.** IRA 2 = more than half of the resin remaining on the tooth. **D.** IRA 3 = entire resin remaining on the tooth surface with a distinctive impression of the bracket mesh.

between groups with AgNP concentrations greater than 0.1 % ($p > 0.05$) (Table 1).

4. Discussion

The high prevalence of white spot lesions is an important concern in orthodontic treatment (de Almeida et al., 2018). Several studies have proposed the inclusion of AgNP in the adhesive matrix to reduce colonization by cariogenic bacteria (Poosti et al., 2013;2; Sánchez-Tito and Tay, 2021; Yassaei et al., 2020). A recent study showed that AgNP not only have a high capacity to inhibit bacterial growth but also play a crucial role in preventing the adherence of these bacteria to orthodontic materials (Nafarrate-Valdez, et al., 2022).

Addition of AgNP in the adhesive should not affect its mechanical properties, which is important from a clinical point of view. However, orthodontic adhesives modified with AgNP exhibit diverse effects in terms of the physical and mechanical properties (Reddy et al., 2013; Reynolds and von Fraunhofer, 1976).

Our results showed that the addition of AgNP to the orthodontic adhesive decreased the average of SBS in the experimental groups. Incorporating 1 % AgNP, with a size of 21 ± 5 nm, into orthodontic adhesive significantly decreases the SBS (7.55 ± 1.29 MPa) when compared to a control group (9.43 ± 3.03 MPa) (Reddy et al., 2013). The addition of 0.3 % AgNP (50 nm) in an orthodontic adhesive significantly decreases the SBS values (10.51 ± 7.15 MPa) compared to the control group (17.72 ± 10.55 MPa) (Eslamian et al., 2020). Although the addition of AgNP led to a reduction in SBS values, it is important to note that in all cases, these values remained within the clinically acceptable range, typically described as falling between 6 and 8 MPa (Reynolds and von Fraunhofer, 1976). However, these values are recommended for clinical conditions; therefore, they may differ from the findings of the *in vitro* studies (Eslamian et al., 2020).

Additionally, the results showed significant differences in the bonding failure patterns. In the case of the 1 % group, the main failure pattern was between the adhesive and enamel interface, whereas in the case of the 0.05 % group and the control group, failure occurred mainly between the bracket and adhesive interface. A previous study found no differences in the distribution of the ARI failure pattern between the modified adhesive and control, with a score of 2 being more frequent, where more than half of the resin remained on the tooth surface (Eslamian et al., 2020). The failure pattern of AgNP-modified adhesives can prevent enamel damage, and failure between the adhesive and enamel can increase the probability of damage to the enamel surface (Degrazia et al., 2016).

The microhardness values increased significantly with increasing concentration of AgNP in the adhesive. The evaluation of the ultrastructure of six light-curing orthodontic adhesives revealed the microhardness of Transbond XT as 59.1 ± 3.0 VMH (Vilchis et al., 2015). The filler content in the adhesive matrix increased the strength, reduced dimensional changes, and facilitated material handling. The size and type of the filling can also influence these properties (Faltermeier et al., 2007). A linear relationship between the adhesive wear and filler particle content exists, where an increased filler level is correlated with increased wear resistance (Faltermeier et al., 2006). In this study, the incorporation of AgNP, with an average size of 20 nm, increased the filler content of the adhesive, which explains the increase in microhardness values reported as the AgNP concentration increased.

The evaluation of surface roughness showed that in the groups with AgNP concentrations greater than 0.1 %, the roughness increased with respect to the values observed for the control group. O'Kane et al. (1993) reported that the surface roughness of the Transbond adhesive showed Ra values of 0.392 ± 0.256 (O'Kane et al., 1993). Transbond adhesives contain a large amount of filler (82 %) with a small particle size (3 μ) (O'Kane et al., 1993). In this regard, a smaller particle size in adhesives is associated with a smoother surface (Van Noort and Davis, 1984). Incorporating the AgNP into the adhesive matrix increased the amount of filler, which could explain why higher surface roughness values were observed.

The present study had some limitations. These findings must be interpreted considering that *in vitro* studies are incapable of fully reproducing conditions and variables related to the oral environment. To the best of our knowledge, no studies have established protocols for incorporating the correct percentage of antibacterial agents into orthodontic adhesives. Further studies should be conducted to evaluate the incorporation of AgNP into orthodontic adhesives of different sizes and amounts of filling, as it is known that these factors can influence the mechanical properties of orthodontic adhesives.

5. Conclusion

The incorporation of AgNP into an orthodontic adhesive can potentially decrease the SBS while increasing the microhardness and surface roughness values.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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