Acta Odontologica Scandinavica, 2013; 71: 1038–1042

informa healthcare

ORIGINAL ARTICLE

Investigating the effect of incorporating nanosilver/nanohydroxyapatite particles on the shear bond strength of orthodontic adhesives

AZAM AKHAVAN[1], AHMAD SODAGAR[2], FARAMARZ MOJTAHEDZADEH[2] & KOSAR SODAGAR[3]

[1]*Radiation Applications Research School, Nuclear Science and Technology Research Institute, Tehran, Iran,* [2]*Dental Research Center, Department of Orthodontics, Tehran University of Medical Sciences, Tehran, Iran, and* [3]*Dentist, Private Practice, Tehran, Iran*

Abstract

Introduction: Development of clinically acceptable orthodontic adhesives with additional anti-microbial and remineralizing features could be undertaken only if their mechanical properties have also been considered. The purpose of this study was to investigate the effect of incorporating Silver and Hydroxyapatite (HA) nanoparticles on the shear bond strength (SBS) of an orthodontic adhesive. ***Methods:*** Silver and HA nanoparticles were prepared and inspected by scanning electron microscopy and EDAX analysis. The nanoparticles were added to the primer of Transbond XT in 1%, 5% and 10% silver concentrations. Each compound (along with a control) was used for bonding stainless steel brackets to 12 human premolars (48 in total) and the SBS of all samples, along with their ARI scores were measured. ***Results:*** The SBS of the control, 1%, 5% and 10% nanoparticle groups were 12.06 ± 5.48, 20.66 ± 5.72, 10.77 ± 8.16 and 5.40 ± 2.00 MPa, respectively. A significant difference existed between all study groups ($p < 0.05$), except for the control–5% and 5%–10% study groups ($p = 0.99$ and $p = 0.35$). There was no statistically significant difference in distribution of ARI scores across the study groups ($p = 0.44$). ***Conclusions:*** Incorporation of silver/HA nanoparticles containing 5% and 1% silver maintains and increases the SBS of orthodontic adhesives, respectively, whereas increasing the amount of particles to 10% has an undesirable effect when compared to the control group.

Key Words: *nanoparticles, silver, hydroxyapatite, orthodontics, bond strength*

Introduction

Decades since their introduction into the field of orthodontics, composite resin adhesives undoubtedly remain the first choice of most orthodontists for bonding brackets. However, despite their popularity, some of their shortcomings have not been totally eliminated yet. The remaining exposed composite surface, which is specifically located around the bracket margin, makes it a locus for plaque accumulation and further promotes decalcification of the adjacent enamel [1]. This issue, along with the fact that routine oral hygiene procedures become difficult to perform in orthodontic patients, necessitates research in this field.

In order to overcome these inconsistencies, different processing and manipulations in conventional (commercial) composite adhesives are made, leading to the development of what has been called 'experimental composite adhesives (ECA)' [2]. As an example, one group of efforts has been the incorporation of different antimicrobial agents in the adhesives, of which fluoride and chlorhexidine could be considered two of the most common [3,4]. However, disregarding their questionable antimicrobial efficiency, one drawback to incorporating such antimicrobial substances has been claimed to be undesirable and clinically unacceptable mechanical properties of the resulting adhesive [3,5–7].

With the emergence of nanotechnology and the different behavior expressed by nanoparticles, attempts have been made to take advantage of this concept in orthodontic bonding. It can be said that nanoparticles used in ECAs for antimicrobial purposes have mainly been silver nanoparticles [8]. However, studies have also been reported that have used

Correspondence: Dr Ahmad Sodagar, Department of Orthodontics, School of Dentistry, Tehran University of Medical Sciences, North Kargar St. Tehran 1439955991, Iran. Tel: +982188351162. E-mail: sodagara@tums.ac.ir

(Received 25 March 2012; revised 8 June 2012; accepted 24 September 2012)

ISSN 0001-6357 print/ISSN 1502-3850 online
DOI: 10.3109/00016357.2012.741699

hydroxyapatite (HA) nanoparticles, which in theory can be proposed to enhance remineralization of the enamel surface located around the bracket and consequently aid in the prevention of dental caries.

However, as mentioned previously, attempts for producing ECAs with the intention of increasing protective properties preferably should not cause adverse effects on the mechanical properties. Different nanoparticles have been incorporated and different results have been reported in terms of mechanical properties [9]. One important characteristic of orthodontic adhesives is to withstand loads, which is commonly evaluated by orthodontic shear bond strength (SBS) studies.

To our knowledge, there is no study that has investigated the mechanical properties of ECAs' which contain both silver and HA nanoparticles. Therefore, the purpose of the current study was to evaluate the effect of incorporating different amounts of silver/HA nanoparticles on the shear bond strength of orthodontic adhesives.

Materials and methods

Materials

In order to produce the ECA for this study, Transbond XT Primer (3M Unitek, Monrovia, CA, LOT No. N238085), was selected as the base. HA powder (Aldrich Co.) with a particle size of less than 200 nm and a specific surface area of more than 97% was used.

Preparation of silver/HA nanoparticles

Doped HA with various concentrations of silver nanoparticles (1%, 5%, 10%) were prepared by gamma irradiation as a clean physical method. The obtained products were characterized by scanning electron microscopy (SEM) and EDAX analysis recorded on the Philips XL30.

Specimen preparation

Forty-eight intact mandibular premolars were collected and stored in a physiologic saline solution for this study. After cleaning the enamel surface with a prophylaxis brush, the labial enamel surface was etched for 30 s with a 37% phosphoric acid gel rinsed with a water syringe for 60 s and dried thoroughly at least for 10 s until the expected chalky white appearance was observed. Next, the teeth were randomly assigned to four study groups, wherein each group the following primers were applied: (1) Conventional Transbond XT primer (control group), (2) Transbond XT primers containing equal amounts of silver/HA nanoparticles (5%) but with a different concentration of silver, 1%, 5% and 10%. The primer was cured using a halogen lamp – light curing device (3M Unitek, Monrovia, CA) for 10 s. In the next step, 0.018 inch slot standard edgewise stainless steel premolar brackets were placed in the center of the labial enamel surface using the Transbond XT adhesive paste (LOT No. N238051), pressure was applied to the bracket in order to seat the bracket on the enamel surface and excess adhesive was removed with the aid of a hand scaler. Finally the adhesive was cured with the same halogen light-curing unit, by means of 10 s exposures from each side of the bracket (a total of 40 s for each specimen). The bracket placement procedure for all samples was performed by one of the investigators (A.S) in order to obtain uniform specimens. Samples were embedded in self-polymerizing acrylic in the next stage. Special molds were designed for this purpose, comprised of a 0.016 × 0.022 inch stainless steel wire acting as the jig, which the bonded brackets were tied to with ligature wire. After securing the wire in its precise position, self-polymerizing acrylic (Meliodent, Batch No. 20402F-1) was prepared and poured into the molds up to the crowns of the teeth (Figure 1).

All samples were next thermocycled in a thermocycling machine for 1500 cycles between 5–55°; 15 s bath times for each temperature and a 10 s interval).

Shear bond strength procedure

Shear bond strength testing was performed with a universal testing machine (Zwick OZ50, Germany), the shearing force was applied with the aid of a chisel shape blade having a 0.6 mm thick edge. An occlusogingival force with a cross-head speed of 0.5 mm/min was applied to the specimens until failure occurs and the maximum loading force was recorded in Newtons. These figures were divided by a nominal

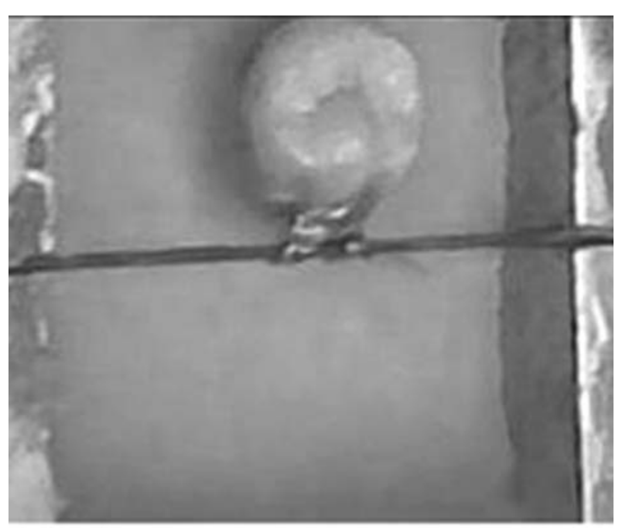

Figure 1. Sample specimen mounted in specified jig and embedded in self-curing acrylic.

bracket base surface area of 12.62 mm^2, in order to convert the values into Megapascals (MPa).

Finally, the specimens were examined under a stereomicroscope with a 10× magnification in order to determine the adhesive remnant index (ARI) as introduced by Artun and Bergland [10].

Data analysis was performed with the aid of version 16 of the SPSS software (SPSS, IL). In addition to the descriptive statistics, one-way analysis of variance (ANOVA) and Tamhane were also considered.

Results

To determine the size and distribution of the silver/HA nanoparticles, SEM images were taken. As shown in Figure 2, a uniform particle size without any significant aggregation is observed for three kinds of samples. The EDAX spectra also confirmed the presence of elemental compounds (Ca, P, Ag) in silver/HA nanoparticles without any impurity peaks (Figure 3).

The descriptive data for the shear bond strength of the four study groups are shown in Table I. The highest mean SBS was observed in the 1% group and the lowest in the 10% group. One-sample Kolmogorov-Smirnov tests showed a normal distribution among all groups.

The Analysis of Variance test among the four study groups showed a significant difference (p = 0.00). Post-hoc Tamhane tests were performed between each pair of groups. Levels of significance are presented in Table II. Significant statistical differences were observed between most of the study groups except between the control–5% and the 5%–10% groups. A significant difference between the 1% and 10% existed. The results of the Kruskal-Wallis analysis for comparing ARI results among groups demonstrated a statistically non-significant difference for remaining adhesive scores (p = 0.44).

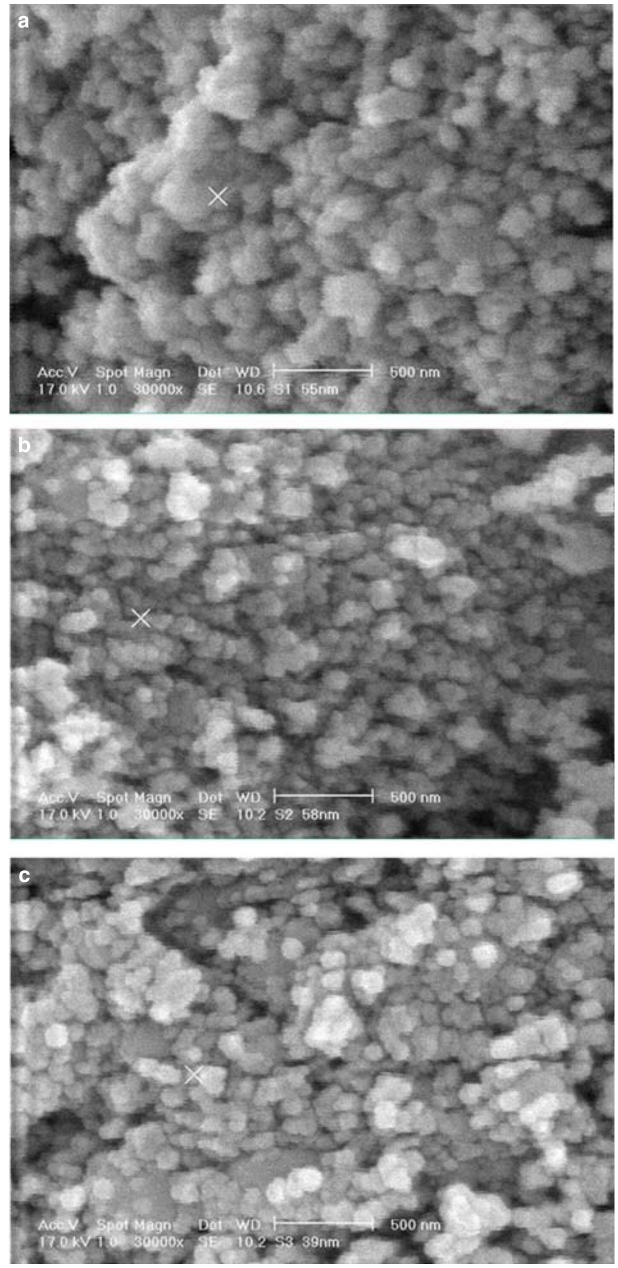

Figure 2. SEM images of silver/HA nanoparticles containing (a) 1%, (b) 5%, (c) 10% silver.

Discussion

As incorporation of nanoparticles into other orthodontic material (e.g. acrylic resins) has shown significant effects in terms of antimicrobial and mechanical properties [11,12], it was also decided to investigate such interventions on the properties of orthodontic adhesives.

The results of the current study show that incorporation of silver/HA nanoparticles containing 1% silver significantly increase the orthodontic shear bond strength of Transbond XT as an orthodontic adhesive. Increasing the amount of silver to 5% reduces the bond strength to a level comparable to the control group. Also, finally, further increasing the amount of silver nanoparticles to the 10% level demonstrates a decline in SBS.

The initial increase in shear bond strength of Transbond XT may be attributed to the silver/HA nanofiller consistent with previous reports [13]. It has been proposed that nanofillers can improve adhesion at the interface between the restorative material and the tooth structure through increasing mechanical strength of the adhesive layer and providing structural reinforcement. In fact, the nanofillers are stress absorbing and have the role of an elastic layer between dental composite and enamel. However, the amount of nanofiller and distribution of particles are the critical parameters which should be optimized in experiments.

The further decrease in bond strength associated with the addition of more nanoparticles could be due to an agglomeration of particles, creating defect points and interfering with the curing process of the

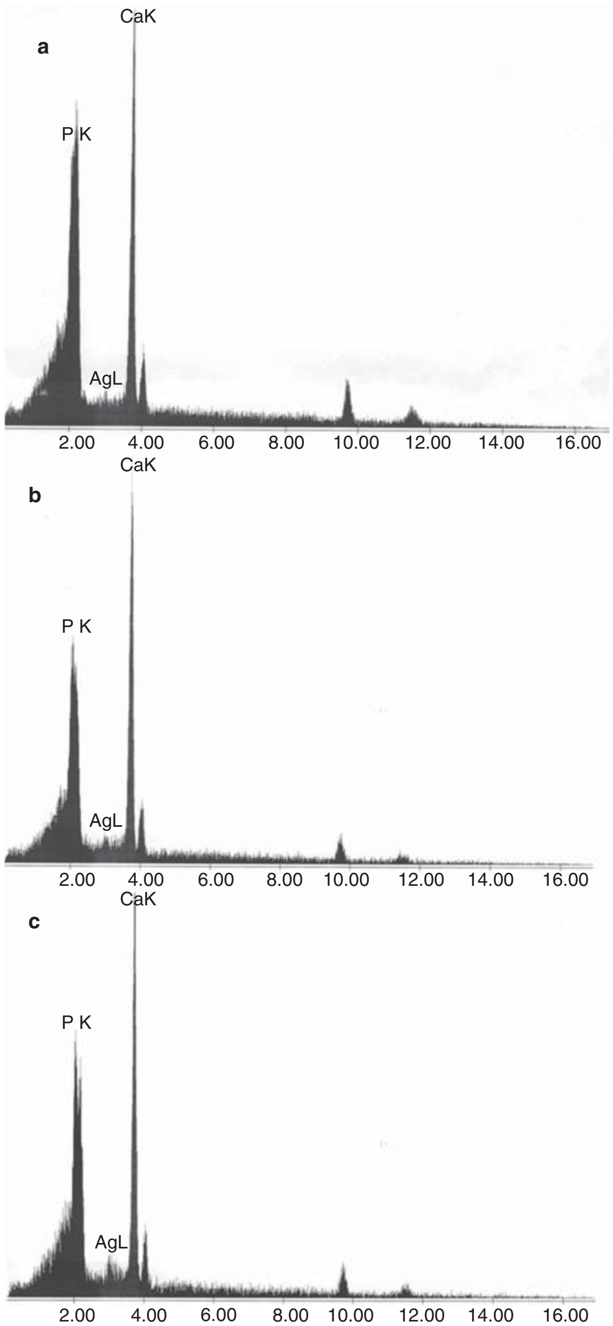

Figure 3. EDAX spectra of silver/HA nanoparticles containing (a) 1%, (b) 5%, (c) 10% silver.

adhesive. This pattern of a primary increase in bond strength followed by a further decline has also been seen in other studies [14].

Many investigations dealing with the orthodontic SBS of adhesives compare the results of their studies with previously published articles, claiming that a value between 6–8 MPa is a clinically acceptable figure, adequate to withstand occasional masticatory loads and at the same time low enough to prevent damage to the enamel [15]. However, elsewhere, other authors have criticized this rule to be highly influenced by the loading design [16]. Therefore, in order to avoid misjudgements, it appears more logical to discuss the results of this study only within its own boundaries.

Table I. Descriptive data of the shear bond strength of the study groups.

Group	*n*	Mean	SD	Min	Max
Control	12.06	5.48	10	3.91	24.26
Nano 1%	20.66	5.72	10	11.53	27.48
Nano 5%	10.77	8.16	10	2.85	27.01
Nano 10%	5.40	2.00	10	2.04	8.03

Based on this concept and giving consideration to the opinions of some who propose Transbond XT as the 'gold standard' orthodontic bonding adhesive and since the results obtained from the 5% study group is comparable to the 'gold standard', it could be proposed that incorporation of 5% of nanoparticles keeps the bond strength at the same 'clinically' acceptable level. An increase in the amount of bond strength, which was observed in the 1% study group could be considered an improvement, as long as this increase does not jeopardize the enamel surface. However, since there was no significant difference between the groups in terms of remaining adhesive patterns, it can be assumed that the addition of 1% silver/HA nanoparticles could improve the clinical performance of Transbond XT.

Incorporating nanoparticles into the bonding agent and not into the adhesive body itself could be considered a more logical approach and somehow beneficial from a clinical viewpoint, since it is the bonding agent which comes into direct contact with the enamel surface; the target area for preventive attempts.

Future studies are undoubtedly required to investigate the biological effects of adding such amounts of nanoparticles.

Conclusion

It was observed that the addition of silver/HA nanofiller to the orthodontic adhesives affects the shear bond strength of the products. The incorporation of 5% and 1% silver/HA nanoparticles maintains and increases the orthodontic shear bond strength of orthodontic adhesives, respectively, whereas increasing the amount of particles to 10% has an undesirable effect on shear bond strength when compared to the control group. Further research is required in order to

Table II. *P*-values of Tamhane tests between each two study group.

Group	Control	1%	5%	10%
Control	—	0.018	0.999	0.024
Nano 1%	0.018	—	0.038	0.000
Nano 5%	0.999	0.038	—	0.356
Nano 10%	0.024	0.000	0.356	—

approve such experimental composite adhesives as clinically acceptable.

Acknowledgments

The authors would like to thank Dr Mohammad Javad Kharazi Fard for his contribution in performing the statistical analysis of this study.

Declaration of interest: The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

References

[1] Lim BS, Lee SJ, Lee JW, Ahn SJ. Quantitative analysis of adhesion of cariogenic streptococci to orthodontic raw materials. Am J Orthod Dentofacial Orthop 2008;133:882–8.
[2] Weitman RT, Eames WB. Plaque accumulation on composite surfaces after various finishing procedures. J Am Dent Assoc 1975;91:101–6.
[3] Jedrychowski JR, Caputo AA, Kerper S. Antibacterial and mechanical properties of restorative materials combined with chlorhexidines. J Oral Rehabil 1983;10:373–81.
[4] Cohen WJ, Wiltshire WA, Dawes C, Lavelle CL. Long-term *in vitro* fluoride release and re-release from orthodontic bonding materials containing fluoride. Am J Orthod Dentofacial Orthop 2003;124:571–6.
[5] Ashcraft DB, Staley RN, Jakobsen JR. Fluoride release and shear bond strengths of three light-cured glass ionomer cements. Am J Orthod Dentofacial Orthop 1997;111:260–5.
[6] Paschos E, Kurochkina N, Huth KC, Hansson CS, Rudzki-Janson I. Failure rate of brackets bonded with antimicrobial and fluoride-releasing, self-etching primer and the effect on prevention of enamel demineralization. Am J Orthod Dentofacial Orthop 2009;135:613–20.
[7] Bulut B, Türkün M, Türkün LS, Isiksal E. Evaluation of the shear bond strength of 3 curing bracket bonding systems combined with an antibacterial adhesive. Am J Orthod Dentofacial Orthop 2007;132:77–83.
[8] Hernandez-Sierra JF, Ruiz F, Pena DC, Martinez-Gutierrez F, Martinez AE, Guillen Ade J, et al. The antimicrobial sensitivity of Streptococcus mutans to nanoparticles of silver, zinc oxide, and gold. Nanomedicine 2008;4:237–40.
[9] Xia Y, Zhang F, Xie H, Gu N. Nanoparticle-reinforced resin-based dental composites. J Dent 2008;36:450–5.
[10] Artun J, Bergland S. Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pretreatment. Am J Orthod 1984;85:333–40.
[11] Sodagar A, Kassaee MZ, Akhavan A, Javadi N, Arab S, Kharazifard MJ. Effect of silver nano particles on flexural strength of acrylic resins. J Prosthodont Res 2012;56:120–4.
[12] Kassaee MZ, Akhavan A, Sheikh N, Sodagar A. Antibacterial effects of a new dental acrylic resin containing silver nanoparticles. J Appl Polym Sci 2008;110:1699–703.
[13] Kim JS, Cho BH, Lee IB, Um CM, Lim BS, Oh MH, et al. Effect of the hydrophilic nanofiller loading on the mechanical properties and the micro tensile bond strength of an ethanol-based one-bottle dentin adhesive. J Biomed Mater Res B Appl Biomater 2005;72:284–91.
[14] Sadat-Shojai M, Atai M, Nodehi A, Khanlar LN. Hydroxyapatite nanorods as novel fillers for improving the properties of dental adhesives: synthesis and application. Dent Mater 2010;26:471–82.
[15] Reynolds IR. A review of direct orthodontic bonding. Br J Orthod 1975;2:171–8.
[16] Mojtahedzadeh F. The problem with normal orthodontic shear bond strength values. World J Orthod 2010;11:210.