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Research Article

Effect of nano–zinc oxide and nano-chitosan particles on the shear bond strength of dental composites used as orthodontic adhesive

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ABSTRACT

Background: This study aimed to evaluate the effect of the combination of zinc oxide nanoparticles (NPs) and chitosan NPs on the shear bond strength (SBS) of composites used for orthodontic bonding.
Methods: Four groups of composites ($n = 10$), containing 0%, 1%, 5%, and 10% w/w NP fillers, respectively, were used to bond brackets to the surfaces of 40 intact bovine incisors. After 1000 rounds of thermal cycling at 5°C–55°C, all specimens were mounted in acrylic blocks. The SBS was tested using a universal testing machine, and the adhesive remnant index scores were registered using a stereomicroscope. Data were statistically analyzed using a 1-way ANOVA and the Kruskal–Wallis test.
Results: The highest value of mean SBS was found in the control group, and the lowest value was found in the group with composite containing 10% NPs. The adhesive remnant index did not differ significantly among the groups ($P = 0.823$).
Conclusions: Incorporation of 1% and 5% zinc oxide and chitosan NPs had no effect on the SBS of composite, and the obtained SBS values were similar to that of the control group.

1. Introduction

Bonding is the most commonly used technique to attach brackets to tooth surfaces, owing to its having high aesthetic appeal and being simple to use. However, plaque accumulation around the attachments, formation of white spot lesions, and bond failure are among the drawbacks of this technique, prolonging treatment, at higher cost and with more chair time [1–4]. An ideal bond should be able to resist forces applied during treatment and maintain an enamel surface that is unharmed after debonding [5–10]. Recently introduced dental adhesives and composite resins are highly reliable, with higher bond strength and less microleakage, which explain their frequent use in orthodontics [11–13].

Nanotechnology has greatly advanced in the area of composite resin production. Filler volume can be increased if the particle size is smaller; consequently, the polymerization shrinkage decreases, yielding better mechanical properties, such as higher compressive and tensile strength. At present, nano-composites and nano-ionomers are accepted for clinical use as bonding agents [11].

Metal oxides such as alumina, zirconia, and silica are frequently used to enhance the bond strength of restorative materials. Rodríguez et al. reported a significant decrease in the shear bond strength (SBS) of orthodontic adhesive as a result of incorporation of silver nanoparticles (NPs); however, the strength was still above that required for orthodontic treatment [14]. In another study, the SBS of orthodontic adhesive decreased as the percentage of nanosilver/nanohydroxyapatite increased [15]. Poosti et al. reported that the antimicrobial activity of orthodontic adhesives containing titanium dioxide (TiO_2) NPs improved, without deterioration of their SBS [16]. Another study showed that incorporating zinc oxide (ZnO) NPs into composite resin improved the physical properties, such as the flexural modulus and compressive strength [17]. Barcellos et al. reported that with addition of ZnO NPs to an adhesive system, the bonding to dentin was preserved after 6 months, without compromising the mechanical features [18]. ZnO NPs also exhibit antimicrobial activity and prevent the development of biofilm [19,20]. NPs are believed to penetrate the bacteria cell wall because they are smaller and exert their antibacterial activity more effectively [2,21]. Chitosan also has applications in many fields of indus-

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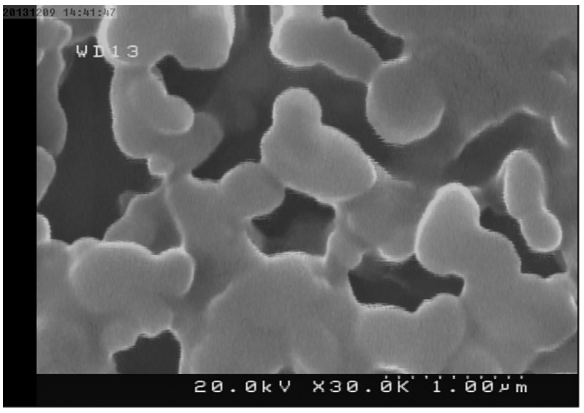

Fig. 1. Nano chitosan under scanning electron microscope (magnification: 30.0 KX).

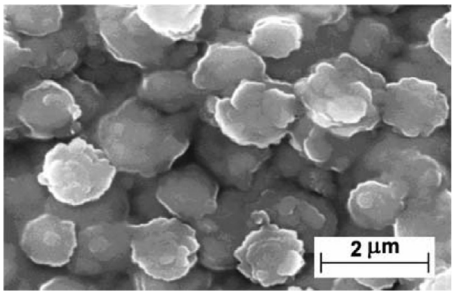

Fig. 2. Nano zinc oxide under scanning electron microscope (magnification: 80.0 KX).

try and can be prepared in nano- and micro-forms. The antibacterial activity of chitosan NPs has been demonstrated for a wide range of bacteria. In addition, it has been reported that adding chitosan to composites enhances their antibacterial properties without compromising their SBS [22]. The structure of this particular NP allows incorporation of other compounds, making it a bionanocarrier, and enhances the benefit of the favorable synergistic effects of several materials [14,23,24].

As the antimicrobial properties of these 2 NPs was investigated previously [25], the current study was performed to assess the effect on SBS of incorporation of various percentages (wt%) of ZnO and chitosan NPs into orthodontic adhesive, in order to determine which adhesive concentration provides proper bond strength for enabling orthodontic treatment. The null hypothesis was that these concentrations would make no difference in the SBS, and no adverse effects would be observed.

2. Methods and materials

2.1. Nanocomposite preparation

For preparation of chitosan NPs, low–molecular weight (1–3 KDa) chitosan (ACROS Organic, New Jersey, USA) was vigorously mixed with water, 1% acetic acid, and tripolyphosphate and centrifuged for 30 minutes. After rinsing the deposit, it was frozen and then crushed to prepare the powder for use [26] (Fig. 1). Also, 38-nm ZnO NPs were prepared from Zn(OH)2 using a trisodiumcitrate-assisted hydrothermal process [27] (Fig. 2).

2.2. Preparation of specimens

To weigh the required amounts of NPs and composites, a laboratory scale (U.S. Solid, ND, USA) with a precision of 0.0001 G was used. Next, 576 mg of Transbond XT (3M Unitek, Monrovia, CA) composite was blended with 64 mg nano powder (containing 50/50 w/w NPs) to achieve 640 mg of 10% NP–containing composite. Then, 200 mg of original composite was blended with 200 mg of 10% w/w blended composite to obtain a composite with 5% NPs. Then, 40 mg of 10% w/w composite was mixed with 360 mg of plain composite to obtain a 1% w/w NP composite. The process of blending was done with a spatula on a glass slab in a dimly lit environment.

Forty bovine incisors without cracks, fractures, or decay were kept in a 0.5% chloramine solution at 4°C for 1 week. Then the samples were randomly assigned into 1 of 4 groups (n = 10)- 1 control group and 3 groups of bonded teeth with adhesives containing 1%, 5%, and 10% NPs, respectively. A prophylaxis brush was used to clean the teeth followed by rinsing and drying. The buccal surfaces were etched with 37% phosphoric acid gel (Ultra etch, Ultradent, South Jordan, UT) for 30 seconds and were rinsed for 15 seconds and dried with air free of oil and moisture until their surface appeared white and chalky [16]. A thin coat of Transbond XT Primer (3M Unitek, Monrovia, CA, USA) was applied uniformly to the etched surfaces and cured with a light-curing unit (Woodpecker LED Curing light, Guilin Woodpecker Medical Instrument Co., Ltd.; Guangxi, China), for 10 seconds. Transbond XT adhesive (3M Unitek) was added to the base of the upper central incisor brackets (American Orthodontics, Sheboygan, WI). The brackets were part of the standard edgewise system and had a 0.018 x 0.028-inch slot and a 12.62-mm^2 base area. They were placed on the middle third of the tooth crowns, aligned with the longitudinal axis of the crowns, and after removal of excess adhesive by a scaler, a 40 seconds of curing cycle was applied to each sample (10 seconds for each surface—mesial, distal, incisal, and gingival) [28].

To simulate the conditions of the oral environment, all samples were subjected to thermocycling (TC/300, Vafaei Industrial Factory, Tehran, Iran) of 1000 cycles within 24 hours. In each round, the samples were immersed in a 5°C water bath for 15 seconds, taken out of the bath for 10 seconds, and then immersed in 55°C water bath for 15 seconds [29–31]. Then the teeth were fixed to a stainless steel rectangular wire with elastomeric O - rings and mounted in the middle of 2.5-cm square metal molds. Eventually, the molds were packed with an auto-polymerizing acrylic resin Acropars (Marlic Co. Tehran, Iran) .

2.3. Evaluation of SBS

To measure the SBS, a universal testing machine (Zwick/Roell, Ulm, Germany) was used. The teeth were placed in a position such that the base of the bracket was parallel to the machine force direction. The 0.6 mm–thick metal machine blade applied the force in an inciso-gingival direction, with a crosshead speed of 0.5 mm/min [32] until the bracket is debonded. The recorded value in Newtons (N) was divided by the bracket base area (mm^2) to calculate the SBS in MegaPascal (MPa) units .

2.4. Evaluation of adhesive failure

To check the amount of remaining adhesive on the bracket base after debonding, a stereomicroscope (SMZ800, Nikon, Tokyo, Japan) was used with x10 magnification. The following system was used to determine the adhesive remnant index (ARI): 0 = absence of adhesive on bracket base; 1 = <25% composite on bracket base; 2 = 25%–50% composite on bracket base; 3 = 50%–75% composite on bracket base; 4 = 75%–100% composite on bracket base (Fig. 3) [22].

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Table 1
Results of One-way ANOVA test.

	Sum of squares	*df*	Mean square	F	Significance level
Between groups	452.577	3	150.859	5.66	0.003
Within groups	959.063	36	26.641	–	–

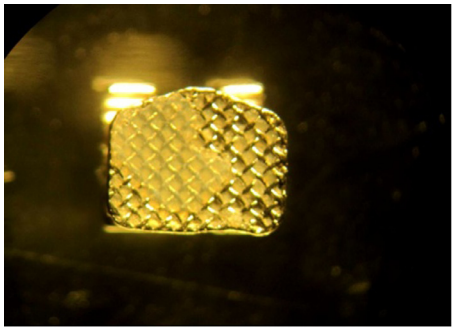

Fig. 3. Adhesive remaining on bracket base after debonding.

Table 2
Descriptive statistics of shear bond strength (SBS) in the 4 studied groups ($n = 10$).

% of NPs	Mean strength (Mpa)	SD
0	33.7	6.50
1	31.4	4.93
5	27.8	5.90
10	24.7	2.40

NP, nanoparticle.

Table 3
Comparison of adhesive remnant index (ARI) scores among groups.

% of NPs	Score 0	Score 1	Score 2	Score 3	Score 4
0	0 (0)	0 (0)	3 (30)	4 (40)	3 (30)
1	1 (10)	0 (0)	3 (30)	3 (30)	3 (30)
5	0 (0)	1 (10)	4 (40)	2 (20)	3 (30)
10	0 (0)	1 (10)	4 (40)	3 (30)	2 (20)
Total	1 (2.5)	2 (5)	14 (35)	12 (30)	11 (27.5)

NP, nanoparticle.
Values are n (%), unless otherwise indicated.

To analyze the results, a One-way ANOVA test was used, followed by a post hoc Tukey's Honest Significant Difference (HSD) test. The ARI scores were analyzed using the Kruskal–Wallis test.

3. Results

Data distribution was normal using the One-Sample Kolmogorov-Smirnov Test ($P > 0.005$). According to the One-way ANOVA results, there was a statistically significant difference among the SBS of the 4 groups ($F = 5.66$; $P = 0.003$; Table 1). Descriptive statistics showed that the control group exhibited the highest value of mean SBS (33.7 ± 6.50 Mpa); the lowest value (24.7 ± 2.40 Mpa) found was for the 10%-NPs group (Table 2). No statistically significant difference was revealed among the ARIs of the groups, using the Kruskal–Wallis test ($P = 0.823$;) (Table 3).

4. Discussion

The dental literature is rich with evidence that the antimicrobial properties of composites containing chitosan or ZnO NPs decrease plaque accumulation and lower the incidence of caries [19,24,25,33,34]. However, the synergistic effects of these 2 NPs, and the consequent changes in the mechanical and physical properties of composites following their incorporation, have yet to be fully elucidated.

The aim of current study was to assess the alterations in SBS following the addition of chitosan and ZnO NPs to composite resin for orthodontic purposes. The null hypothesis was that these tested concentrations would not alter the SBS and no adverse effect would be observed. The results demonstrated that the SBS of composites containing ≤5% NPs was not significantly different from that of the conventional control Transbond XT composite, and the obtained SBS value was within the acceptable range of 6–8 Mpa [35]. Thus, it might be advisable to use 1–5 wt% of NPs to ensure that the consequent mechanical alterations are within the acceptable range. However, this may not apply for the 10% NP group, in which the SBS was lowest. This finding may be due to the disruption in the homogeneity of the composite caused by blending with this amount of NPs. Given that this percentage of NPs may also be toxic, it is not recommended for use in the clinical setting. On the other hand, Mirhashemi et al. [25] reported that a ZnO/chitosan NPs mixture conferred an antibacterial property to adhesive; the strength of this property was significantly higher for the 10 wt% NPs group in the biofilm formation test. This group also showed a significant reduction in bacterial counts, compared with unmodified counterparts, and was the only group that exhibited significant inhibition in the DAD (Disc agar diffusion) test. The only remarkable finding in the eluted component test was on day 30, in which the 10%-NP discs inhibited *Lactobacillus acidophilus* bacteria.

The results of this study were in line with what Sodagar et al., who reported on evaluating the effect of propolis NPs incorporation in 1%, 2%, 5%, and 10% wt. concentrations [36]. They demonstrated that addition of propolis NPs up to 5% maintained the SBS in an acceptable range, whereas the 10% significantly reduced the SBS .

Sodagar et al. also experimented with the addition of 1%, 5%, and 10% Cur [37] and TiO_2 [22] in 2 separate studies and reported that the mean SBS of adhesive with 1% and 5% NPs was still acceptable. The 10% concentration was not recommended for clinical use.

Similar results were reported by Pourhajibagher et al. [38] after assessing the effect of incorporating a mixture of Cur and ZnO at concentrations of 1.2%, 2.5%, 5%, 7.5%, and 10%. They reported that a decrease in SBS values was positively associated with the increase in Cur and ZnO NPs concentration. But because the 7.5% group had both the highest concentration of NPs and the highest SBS value, they recommended it as an appropriate orthodontic adhesive.

Assery et al., however, found an increased mean value of SBS following incorporation of 1% and 3% TiO_2 to an experimental Bis-GMA (bisphenol A-glycidyl methacrylate) free resin composite [39]. The highest adhesion strength was found in the group who had 1% TiO_2, and the lowest strength level was found in the control group. The variation in results may be attributed to the different NPs and adhesive used, and to operator expertise.

Poosti et al. [16] reported no significant difference between the SBS of the composite containing 1% TiO_2 and that in the unmodified group. However, the process of incorporating the NPs into the composite was performed using a high-speed mixer, whereas in

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our study, this process was performed using a spatula; this difference presumably resulted in different particle dispersion and bond strength values. Moreover, instead of thermocycling, which has been shown to reduce bond strength [29–31], they stored the specimens at 37°C for 24 hours. In addition, operator-related factors should not be disregarded.

Another study by Akhavan et al. concluded that composite containing 1% nano-silver and nano-hydroxyapatite had a higher SBS than the control group, whereas the bond strength decreased in the 5% and 10% groups. It is to be mentioned that the study by Akhavan et al. performed thermocycling in their study as well [15]. However, differences in the NPs used, incorporation of NPs into the composite (addition of NPs to the primer), the choice of tooth, and operator-related factors should all be taken into account when comparing the results of this study with the present one.

Yaseen et al. [40] investigated addition of 3% nano-cinnamon and found no negative effects on SBS. Likewise, both tested concentrations of QPEI (quaternary polyethyleneimine) (1% and 1.5%) showed no significant difference in SBS, compared with conventional orthodontic adhesives in a control group [41]. Eslamian et al. [42] also reported that SBS decreased following addition of 0.3% silver NPs, but it was still above the recommended acceptable range.

The ARI index used to evaluate the amount of adhesive remaining showed insignificant differences among the 4 groups, and the highest levels belonged to groups two and three, indicating that bond failure often occurs at the tooth–composite interface. These findings are in accordance with the results of Poosti et al. [16], and Akhavan et al. [15].

The current study assessed the orthodontic adhesive SBS in an in-vitro situation. However, as we know, there are some factors, such as moisture, temperature, and plaque accumulation in the oral cavity, that make the interpretation and generalization of these results difficult. Besides, a diversity of forces—including shear, tensile, and compressive—are applied to orthodontic appliances in vivo [43].

5. Conclusions

Incorporation of up to 5% ZnO and chitosan NPs kept the SBS value of composite resin similar to that of a control group. The 10% NP group showed significantly lower SBS, making this addition an unacceptable choice for use in the clinical setting.

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Competing interest

Authors have completed and submitted the ICMJE Form for Disclosure of potential conflicts of interest. None declared.

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