



ORIGINAL ARTICLE

Evaluation the properties of orthodontic adhesive incorporated with nano-hydroxyapatite particles

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KEYWORDS

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Shear bond strength

Abstract Objective: This research was designed to study the effects of calcium hydroxyapatite nanoparticle incorporation on polymerization as well as the shear bond strength for Heliosit adhesive.

Materials and methods: Calcium hydroxyapatite nanoparticles were prepared from natural products using the sol–gel method, and were inspected using a transmission electron microscope. The nanoparticles were added to the conventional orthodontic adhesive at 2% wt and 4% wt concentrations. The degree of conversion for each test group was measured using a Fourier transform infrared spectroscopy device. Each adhesive group was used for bonding metal brackets to the pre-molar buccal enamel surface. The shear bond strength of all samples was measured.

Results: A significant difference was found among all the study groups ($p \leq 0.05$) in terms of the degree of conversion and shear bond strength. The 2% wt nanoparticle group showed the highest values for both variables. The lowest value was recorded within the 4% wt nanoparticle group in comparison to the control group.

Conclusions: Calcium hydroxyapatite nanoparticle incorporation with a conventional Heliosit adhesive resin to a limited concentration has improved the mechanical properties of orthodontic adhesive.

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1. Introduction

Composite adhesive is the first choice for bonding orthodontic brackets (Sodagar et al., 2017). Demineralization around the

bracket continues to be a concern point for orthodontists (Twomley et al., 2019; Alamri et al., 2020).

As nanotechnology has progressed, many researchers have tried to improve adhesive properties using nanoparticles (NPs) (Akhavan et al., 2013; Altmann et al., 2017; Elsharkawy, 2018; Callister et al., 2020). Nanotechnology can restrain dental caries in two main ways. The first employs the application of nanomaterials with antibacterial properties, such as titanium dioxide (TiO₂) and zinc oxide nanoparticles (ZnO). The second involves utilizing materials with fluoride and calcium release ability, such as calcium fluoride (CaF₂) and calcium hydroxyapatite (Ca₁₀(PO₄)₆(OH)₂) (Ahmadian et al., 2018; Scribante

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et al., 2020). Other nanomaterials with remineralizing performances such as phosphorylated chitosan amorphous calcium phosphate (Pchi-ACP) (Ahmadian et al., 2018) and silver nanoparticles (AgNPs) (Lee et al., 2017; Callister et al., 2020).

Calcium hydroxyapatite nanoparticles have been used widely in medicine and dentistry (Priyadarsini et al., 2018; Nobre et al., 2020). Calcium hydroxyapatite nanoparticles can efficiently fill the micropores present in the enamel by releasing inorganic ions (Sari et al., 2014). Ca^{2+} and PO_4^{3-} diffuse through interprismatic spaces and are converted into hydroxyapatite crystals during the remineralization process (Elasser et al., 2018; Juntavee et al., 2018). Furthermore, these nanosized particles have optimal antibacterial effects due to their high solubility and rapid ion release in addition to their capacity for active oxygen species production (Seyedmajidi et al., 2018).

Calcium hydroxyapatite nanoparticles are either commercially available or synthesized by utilizing natural sources. Several techniques for calcium hydroxyapatite nanoparticle synthesis have been established (Majhool et al., 2019), such as sol-gel reactions (Chen et al., 2011; Agha & Taqa, 2019).

The introduction of orthodontic adhesives within remineralizing features should be considered only if their physical properties are clinically accepted. In 2007, Bishara et al. (2007) studied the shear bond strength for nanohybrid restorative materials and traditional adhesive materials. They reported that the traditional adhesive has higher values compared with the nanorestorative material. Another study that tried to explore the effect of adding nanoparticles to the adhesive was Akhavan et al. (2013) who found that the adhesive containing 5% and 1% silver/calcium hydroxyapatite nanoparticles had the highest shear bond strength, whereas the lowest values were recorded with 10% nanoparticle addition when compared to the control group.

This paper was designed to evaluate the effects of incorporating calcium hydroxyapatite nanoparticles on the degree of conversion (DC) and shear bond strength (SBS) of Heliosit orthodontic adhesives.

The first hypothesis for this paper was that there is no statistically significant difference in the degree of monomer conversion among the control adhesive group and those reinforced with nanocalcium hydroxyapatite particles. The second hypothesis was that there is no statistically significant difference among the shear bond strength for metallic brackets bounded with a conventional orthodontic adhesives and those bounded with a calcium hydroxyapatite nanocomposite.

2. Materials and methods

For this research, ethical approval was provided by the Authorized Committee of College of Dentistry, University of Mosul, Mosul, Iraq on 27/1/2020.

2.1. Preparation of hydroxyapatite nanoparticles by the sol-gel method

Calcium hydroxyapatite nanoparticles were prepared from chicken eggshells, and white eggshells were cleaned to remove the inner lining. The shell was then crushed using a manual mortar (Okhli, India), and the coarse practices were put in an oven at 900 C° for one hour. Snow-white cake was the

end result. Due to the heating, eggshell calcium carbonate CaCO_3 converted to carbon dioxide and calcium oxide (CO_2 - CaO). A solution of 0.6 M H_3PO_4 (phosphoric acid) was slowly added under continuous mixing to the aqueous suspension of CaO (molar ratio). The product was filtered and washed with distilled water and then dried and sterilized in an oven at 110 C° (Agha & Taqa, 2019).

2.2. Transmission electron microscope (TEM)

The crystalline evaluation of calcium hydroxyapatite nanoparticle size and shape was performed using a transmission electron microscope (Philips CM10, Germany). Powder scattered on film was reinforced by copper grids.

2.3. Nanocomposite preparation

Heliosit/ Ivoclar Vivadent, Liechtenstein, Germany adhesive was tested in this paper. Heliosit adhesive is translucent with a low inorganic filler content.

Compositions of Heliosit adhesive	Percentage
silica dioxide	14% wt
monomer matrix of urethane dimethacrylate, Bis-GMA and decandiol dimethacrylate	85% wt
catalysts and stabilizers	1% wt

Two different concentrations of 2% wt and 4% wt were prepared in weight to weight ratios. The definite weight of the calcium hydroxyapatite nanoparticles and adhesive was measured using an electrical sensitive balance (AZZOTA, USA). A plastic spatula was used for mixing in a semi dark room until the soaking of nanoparticles with the adhesive was completed and uniform colour was obtained.

Adhesive tube net weight	Calcium hydroxyapatite nanoparticles concentration	Calcium hydroxyapatite nanoparticles weight for each adhesive tube
2.5 g	2% wt	50 mg
	4% wt	100 mg

2.4. Degree of conversion test sample preparation

Forty five resin discs, fifteen for each test group (control group, group with 2% wt nanoparticle, group with 4% wt nanoparticle), were prepared and standardized by a single operator. A transparent cellulose ring with a 5 mm diameter and a 2 mm thickness was used (Pithon et al., 2010). A plastic spatula was used to settle the adhesive, and a glass slide was placed on top of the ring. Then, a weight of 300 gm was placed on the glass slide (Pithon et al., 2010). The light cure device was fixed on a rod to keep the distance between the light cure device and adhesive to 2 mm (Jain et al., 2013). The material was photopolymerized for 40 s (Pithon et al., 2010). The

LED light cure apparatus was used with a lamp intensity of approximately 1200–1500 mw/cm² (LED Light Curing Device, Coxo Medical, China) standardized with a radiometer (Woodpecker, China).

After curing, the samples were sealed in a dark bottle for monomer conversion evaluation, which was carried out after 24 h (Pithon et al., 2010), using a Fourier transform infrared spectroscopy (FTIR) device (Alpha Bruker, Germany). The following equation was used to evaluate DC (Agha & Taqa, 2019):

$$DC = 1 - ((a/b)/(c/d)) \times 100$$

a = C = C peak area (after curing), b = C-C peak area (after curing).

c = C = C peak area (before curing) d = C-C peak area (before curing).

C = C with an absorption rate (1638 cm⁻¹). C-C approximately (1608 cm⁻¹) (see Fig. 2.)

For intra-examiner calibration, the researcher repeated the FTIR analysis for 5 samples, and a Paired *t*-test was implemented to compare two reading results. We found that there was no significant difference at the level of $p \leq 0.05$ between the two reading results for DC. For inter-examiner calibration, a paired *t*-test was used to compare the current researcher values and other expert researcher values, and we found that there was no significant difference at the level of $p \leq 0.05$ between the two researcher values.

2.5. Shear bond strength test

2.5.1. Study sample

Sixty human maxillary first premolars extracted for orthodontic treatment were collected from private clinics. The inclusion criteria for the study sample were: non-carious teeth, free of restorations and enamel defects, such as hypoplasia, or visible cracks. The extracted teeth were kept in distilled water (Cerone et al., 2019).

The collected teeth were randomly divided into three equal groups:

Group 1: (Control group) Teeth for conventional Heliosit adhesive bonding.

Group 2: Teeth for a modified adhesive (2% wt nanoparticles) bonding.

Group 3. Teeth for a modified adhesive (4% wt nanoparticles) bonding.

2.5.2. Study sample preparation

Each tooth was fixed vertically, such that the tooth root was embedded to the cemento-enamel junction in a cold-cure acrylic (BMS Dental, Italy) block. Teeth were cleansed and polished using non-fluoridated pumice stone (Qualy, Dental, England) and a rubber cup (China) for 10 seconds using a slow speed handpiece (NSK, Japan) and were then washed with water and dried (Cerone et al., 2019). A 37% phosphoric acid gel (Ivoclar Vivadent, Germany) was applied for 15 s, rinsed for 30 s and dried by air jet for 20 s until a chalky white appearance was visible on the enamel (Uysal et al., 2010). A stainless steel bracket (Standard Edgewise, Dentaaurum, Germany) with mesh with a total surface area of (9 cm²) was used. The adhesive resin was applied on the bracket mesh, the bracket was placed on the labial enamel surface, and all excess was removed from around the bracket. The light-

curing unit was fixed on a rod (Pithon et al., 2010) to standardize the distance between the light cure device and the bracket base (2 mm) (Jain et al., 2013). The mesial and distal sides of the bracket were cured for 20 s for each side (total time 40 s) (Uysal et al., 2010). After bonding the brackets, specimens were stored in distilled water at 37 °C for 24 h (Vargas et al., 2017).

2.5.3. Measuring shear bond strength

This test was carried out with the aid of a universal testing machine (Sans, China). A chisel shaped blade with a cross-head speed of (0.5 mm/min) (Uysal et al. 2010; Akhavan et al. 2013) was applied to the occluso-gingival interface until failure occurred, and the maximum loading force was verified in Newton. The shear bracket bond strength in MPa equals the force required to remove the bracket divided by the surface area of the bracket base (Akhavan et al., 2013).

2.6. Statistical analysis

Statistical significance was analysed using Soft Gather SPSS V19. Statistical analyses were implemented using one-way analysis of variance (ANOVA) and Tukey's post hoc test.

3. Results

3.1. Transmission electron microscopy (TEM)

The calcium hydroxyapatite nanoparticle shape is spherical, with sizes that range from 30 to 70 nm at 19,000 to 64,000 magnification one-to-one (see Fig. 1).

3.2. Degree of conversion

For the degree of conversion, the highest value was recorded for Heliosit adhesive with a 2% wt nanoparticle group. The lowest value was recorded within the 4% wt nanoparticle group in comparison to the control adhesive group, as shown in Table 1. One-way analysis of variance (ANOVA) showed that there was a significant difference at $p \leq 0.05$ among the mean values for the three test groups (see Table 1). Tukey's test showed that there was a significant difference among the three test groups ($p \leq 0.05$).

3.3. Shear bond strength

As shown in Table 1, SBS for metallic brackets bonded with a 2% wt calcium hydroxyapatite nanoparticle adhesive presented the highest values, followed by a control adhesive group. The lowest mean value was noted with an orthodontic bracket bonded with a 4% wt nanoparticle group. One-way analysis of variance (ANOVA) showed that there was a significant difference at $p \leq 0.05$ among the mean values for the three test groups. Turkey's test revealed that there was a significant difference among study groups at $p \leq 0.05$ (see Table 1).

4. Discussion

Deminerization of the facial surfaces of teeth during orthodontic treatment is of clinical concern, as it might result

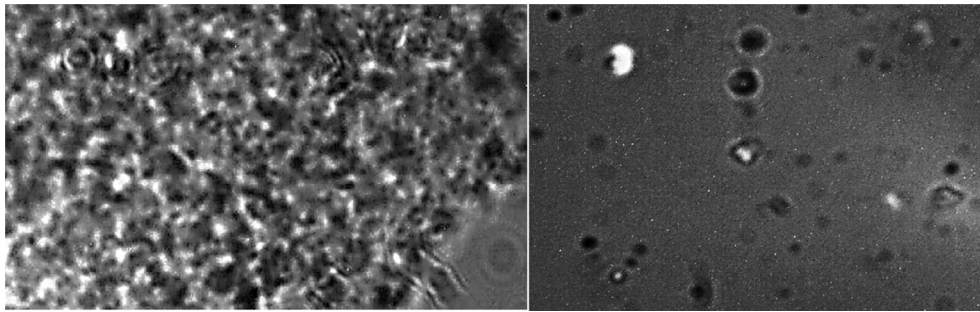


Fig. 1 Calcium hydroxyapatite nanoparticle transmission electron microscopy images.

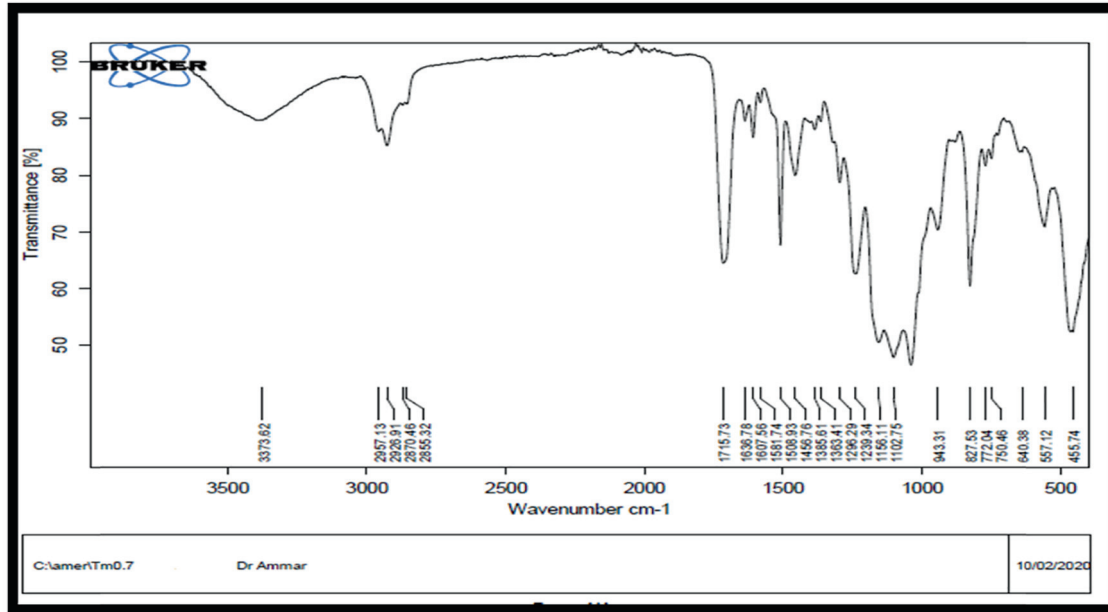


Fig. 2 FTIR chart for Heliosit adhesive incorporated with a 2% wt nanoparticle. C = C with an absorption rate of (1638 cm-1). C-C approximately (1608 cm -1).

Table 1 Descriptive statistics for the degree of conversion and shear bond strength for the control, 2% wt and 4% wt nanoparticle groups. There was a significant difference among the three test groups.

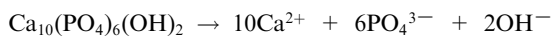
Group	Degree of conversion						Shear bond strength (MPa)					
	No	Minimum	Maximum	Mean	Std. Deviation	Tukey group	No	Minimum	Maximum	Mean	Std. Deviation	Tukey group
Control	15	62.50	67.73	64.79	1.55	B	20	8	12	10	3.6	B
2% wt nanoparticles	15	77.10	85.60	80.57	2.24	C	20	12.5	14.1	13.75	4.1	C
4% wt nanoparticles	15	49.00	53.40	50.73	1.42	A	20	4.6	6.75	6.1	2.9	A
					p - value 0.000							p - value 0.000

Tukey grouping-means with different letters indicate statistically significant differences (p < 0.05).

in decayed teeth even after more than 5 years (Uysal et al., 2010; Kim et al.,2018). From this starting point, this study aimed to develop an orthodontic adhesive with remineralizing features. The tested hypotheses in this study were rejected since

there were statistically significant differences in the degree of conversion and shear bond strength of conventional orthodontic adhesive and calcium hydroxyapatite nanocomposite as shown in Tables 1.

Within this work, incorporation of 2% wt nanoparticles with Heliosit orthodontic adhesive improved the adhesive polymerization in terms of DC. This coincides with Zhang et al. (2013) and Jalalian et al. (2019), who reported that the addition of hydrophilic nanocalcium hydroxyapatite particles with a large surface area increases the acid-base ionic reaction process. Zhang and Wang (2012a) mentioned that other factors, such as a change in viscosity enhanced by physically mixing calcium hydroxyapatite powder, resulted in a higher reaction rate and monomer conversion. Zhang and Wang (2012a), found that the incorporation of calcium hydroxyapatite might elevate the system pH by releasing OH groups and improving the DC.



In this study, incorporation of 2% wt nanoparticle with Heliosit adhesive improved the SBS for adhesive. Ostertag et al. (1991) and Venhoven et al. (1996) stated that adding more filler improved adhesive mechanical properties.

Akhavan et al. (2013) thought that nanofillers act as stress absorbers resulting in an increase in the load that the bracket can withstand by the nanoparticles acting as an elastic layer. In agreement with these study results, Kim et al. (2018) found that bioactive glass (calcium sodium phosphor silicate) incorporation with orthodontic adhesive resulted in an increase in the shear bond strength. Memarpour et al. (2019) explained this increase in SBS because hydroxyapatite acts as a drug delivery carrier due to its superior adsorptive properties, which may in turn increase bond strength. Furthermore, Scribante et al. (2020) found a higher SBS associated with remineralized tooth surfaces.

An alternative viewpoint that might support our results is that presented by Zhang and Wang (2012b), who emphasized that calcium hydroxyapatite can shift the environment to the basic environment. This conversion can improve the bonding efficiency

The results obtained in this research within this test group of 2% wt nanoparticle are in contrast with the perspectives provided by Bishara et al. (2007) and Uysal et al. (2010). Bishara mentioned that SBS for nanocomposite systems were not different from those obtained with standard orthodontic adhesive, while Uysal et al. concluded that nanocomposite has the lowest bond strength values in comparison with the conventional adhesive system.

The reduction in DC and SBS within 4% wt calcium hydroxyapatite may be because nanoparticles aggregated and porosities increased within the resin matrix, resulting in more water being absorbed and subsequently reducing the SBS and other mechanical properties (Santos et al., 2002). This coincide with Sari et al. (2014), who found that a high concentration of calcium hydroxyapatite added to resin modified glass ionomer cement resulted in a reduction in SBS for the tested material. Altmann et al. (2017) found that nanoparticles reduced DC, suggesting that this reduction may have been due to the limited mobility of reactive groups caused by the rapid formation of a cross-linked polymeric network. Furthermore, Elsharkawy (2018) mentions that adding more fillers has an effect on the adhesive viscosity, which may lead to incomplete penetration into the tubule. This can be supported by their results where 10% added filler yielded higher strengths than 20%.

This high 4% wt calcium hydroxyapatite nanoparticle agglomeration is the main cause for the reductions in DC and SBS since they interfere with light penetration through the adhesive layer, resulting in a clear reduction in the photopolymerization process (Altmann et al., 2017; Elsharkawy, 2018). Nobre et al. (2020) mention that calcium hydroxyapatite had the tendency to agglomerate, which might be related to the characteristic of hydroxyapatite being a dipole molecule and consequently to the van der Waals and electrostatic forces between the particles.

The study results are in contrast with Yaseen et al. (2020) perspectives. Yaseen et al. mentioned that there was no significant difference in the incorporation of cinnamon nanoparticle with Heliosit adhesive on the SBS of metallic brackets.

In general, the mean SBS for the three test groups was within or above the minimum requirement range according to Reynolds' (1975) recommendation (5.9–7.8 MPa). The mean DC for the three test groups was within the clinically accepted limit according to the recommendation of Kauppi and Combe (2003); the degree of conversion typically ranges from 55 to 75%.

This innovation of new nanocomposite will help to maintain tooth structure integrity during extended orthodontic treatment periods especially in those with high caries incidence, by utilizing the remineralizing ability of calcium hydroxyapatite nanoparticles. However, many other studies should be conducted to ensure that all properties for this novel nanocomposite are within the clinically accepted scope.

5. Conclusions

We found that 2% wt calcium hydroxyapatite nanoparticle incorporation with conventional Heliosit/ Ivoclar Vivadent adhesive improved both the degree of conversion and the shear bond strength, while incorporation of 4% wt nanoparticle resulted in clear reduction of the degree of conversion and shear bond strength for orthodontic adhesive.

6. Suggestions

Further research should be conducted to study the other adhesive properties such as pH control, ionic release, the adhesive remnant index and the antimicrobial effect, to find the concentration with over all clinically accepted limits and with optimum remineralizing features.

Ethical approval

For this research, Ethical approval was provided by the Authorized Committee of College of Dentistry, University of Mosul, Mosul, Iraq on 27/1/2020.

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Conflict of interest

The authors declared that there is no conflict of interest.

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