Al-Rafidain Dental Journal, Vol. 24, No. 2, 2024 (331- 341)

Al-Rafidain Dental Journal

rden.mosuljournals.com

Does the Addition of Seashell or Zirconium Oxide Nanoparticles at Different Concentrations Improving Tensile Bond Strength of Orthodontic Adhesive? *In-vitro* study

Alaa Gharis Adnan [1], Neam Agha * [2]

[1] Ministry of Health/ Nineveh Health Directorate / Iraq.

[2] Department of Pedodontics Orthodontics and Preventive Dentistry, College of Dentistry, Mosul University / Iraq

Article information

Received: 17 October 2022
Accepted: 23 December 2022
Available online:1September 2024

Keywords
Seashell
Biomaterials
Nanoparticles
Shear bond strength
Adhesive remnant index

***Correspondence:**
E-mail:
neamagha@uomosul.edu.iq

Abstract

Aims: The study aimed to assess the tensile bond strength of modified-Heliosit orthodontic adhesive with different concentrations of seashell or zirconium oxide nanoparticles. **Materials and methods:** Thirty-five sound-extracted human premolars were collected. Hydroxyapatite nanoparticles were manually prepared from natural snail seashells. Heliosit orthodontic adhesive was modified by (8%, 10%, and 12% seashell), and (1%, 3%, and 5% zirconium oxide) nanoparticles. Standard edgewise brackets were bonded to buccal enamel surfaces of the samples of control and six modified adhesive groups. At 24 h after bonding, tensile bond strength was measured. The adhesive remnant index was scored under (10X) magnification power of the stereomicroscope after de-bonding. The chemical characteristics of orthodontic adhesive material were explored before and after mixing with seashell and zirconium oxide nanoparticles by using Fourier Transform Infrared Spectrometry. **Results:** Seashell groups (8, 10, & 12 %) containing hydroxyapatite nanoparticles and zirconium oxide nanoparticles groups (1%, 3%, and 5%) had a higher tensile bond strength mean value than the control group, (10%) seashell group had the highest mean value, while the least mean value was in the control group with statistically significant differences between them. Regarding the adhesive remnant index, no significant differences were found among the studying groups. **Conclusions:** the addition of (10%) seashell nanoparticles had the best performance and improved the tensile bond strength of Heliosit orthodontic adhesive without violating the remnant of adhesive on the buccal enamel surface after brackets de-bonding or inducing any chemical reactions with the resin adhesive.

هل إضافة جزيئات الصدف أو أكسيد الزركونيوم النانوية بتركيزات مختلفة تحسين قوة رابطة الشد لاصقة لتقويم الأسنان؟ دراسة في المختبر

الملخص

الأهداف: تهدف الدراسة إلى تقييم قوة رابطة الشد للمادة اللاصقة لتقويم الأسنان المعدلة مع تركيزات مختلفة من جسيمات الصدف أو أكسيد الزركونيوم النانوية. **المواد وطرائق العمل:** تم جمع 35 ضاحكًا بشريًا سليماً. تم تحضير جزيئات هيدروكسي اباتيت النانوية يدويًا من صدف الحلزون الطبيعي. تم تعديل لاصق تقويم الأسنان الهليوسيت بنسبة (8٪، 10٪، 12٪ صدف) و (1٪، 3٪، 5٪ أكسيد الزركونيوم). تم لصق حاصرات تقويم الاسنان بأسطح المينا الخدية لعينات المجموعة القياسية والمجموعات الستة المعدلة. بعد 24 من اللصق، تم قياس قوة رابطة الشد. تم تسجيل مؤشر بقايا اللاصق تحت قوة التكبير (10x) للميكروسكوب المجسم بعد فك اللصق. تم استكشاف الخصائص الكيميائية للمادة اللاصقة لتقويم الأسنان قبل وبعد الخلط مع الصدف وجزيئات أكسيد الزركونيوم النانوية باستخدام FTIR. **النتائج**: مجموعات الصدف (8٪، 10٪، 12٪) التي تحتوي على جزيئات هيدروكسي اباتيت النانوية ومجموعات (1٪، 3٪، 5٪ أكسيد الزركونيوم) كان لها قيمة متوسط قوة شد أعلى من المجموعة القياسية، مجموعة الصدف (10٪) كان لها أعلى متوسط قيمة بينما أقل متوسط قيمة كان في المجموعة القياسية مع وجود فروق ذات دلالة إحصائية بينهما. بالنسبة لمؤشر بقايا المادة اللاصقة، لم تظهر فروق ذات دلالة إحصائية بين مجموعات الدراسة. **الاستنتاجات**: إضافة (10٪) جسيمات نانوية صدفية كان لها أفضل أداء وقوة شد محسّنة لمادة لاصقة تقويم الأسنان بدون زيادة بقايا اللاصق على سطح مينا الخدي بعد فك اللصق أو إحداث أي تفاعلات كيميائية مع مادة لاصقة تقويم الاسنان.

DOI: 10.33899/RDENJ.2022.136509.1176

INTRODUCTION

The success of treatment with fixed orthodontic appliances depends in great value on the correct positioning of the brackets and successful brackets adhesion to the enamel surface to release optimum orthodontic force. This adhesion must be able to withstand vertical and lateral force from mastication during periods of active treatment without de-bonding as well as, must be safely removed with less damage to the enamel surface (1).

Nanotechnology is used in different branches of dentistry, such as conservative dentistry, root canal treatment, periodontics, prosthetics, and maxillofacial surgery (2). In orthodontics, Nanomaterials give better opportunities to both orthodontists and patients because these materials have better antimicrobial and mechanical properties and can be used for brackets coating, orthodontic wires, elastomeric ligatures, and modifying orthodontic adhesive. However, the important problem of nanomaterials is the cytotoxic possibility, so further researches are necessary (3 & 4).

Hydroxyapatite crystals are considered the essential constituents of the dentine and enamel parts in the tooth structure (5). Hydroxyapatite nanoparticles are highly soluble, highly biocompatible, and have small-size particles that can efficiently fill the enamel micro-pores by releasing inorganic ions (calcium and phosphate) which enhance the remineralizing potential (6). These nanoparticles have a good bactericidal effect (7). For all these biocompatibility, antimicrobial properties, and remineralizing potential, calcium hydroxyapatite nanoparticles have received more attention in the past few years (8). Due to the high cost associated with commercial types of calcium hydroxyapatite nanoparticles, they are synthesized from seashells, fish scales, and eggshells as a natural source as well as the bones and teeth (9). Zirconium oxide nanoparticles have high biocompatibility and wonderful esthetic and mechanical properties (10).

So, the study aims to assess the tensile bond strength of modified-Heliosit orthodontic adhesive with different concentrations of seashell and zirconium oxide nanoparticles, to explore the remnant of adhesive on the buccal tooth surface after brackets de-bonding, and to determine any chemical reactions between the orthodontic adhesive and seashell or zirconium oxide nanoparticles.

MATERIALS AND METHODS

The ethical approval with Ref. no. (UoM.Dent/ DM.L.24/ 22) for this research was obtained from the research ethics committee of the College of Dentistry at University of Mosul. The main materials that were used in the study were:

a. Nano hydroxyapatite (nHA) was manually prepared from snail seashells.
b. Zirconium oxide (ZrO_2) Nanoparticles from (Nano shell, USA).
c. Heliosite orthodontic adhesive from (IVOCLARE Viva dent).
d. Stainless steel standard edgewise metal brackets from (Dentarum, Germany).

Laboratory Synthesis of Nano-hydroxyapatite from Seashell

nHA that was used in this study was manually prepared from snail seashells with a procedure illustrated by Alhussary *et al*. (2020) (11) at the College of Dentistry, University of Mosul (patent 6987, A61C13/08, A61L27/12). The prepared seashell nanoparticles were further characterized by the Transmission Electron Microscope (TEM) (Philips, em208s 100Kv) at different magnification powers (92000 and 130000X) to determine the shape and size of nanoparticles.

Modified Orthodontic Adhesive Preparation

Three different concentrations of seashell-modified orthodontic adhesive (8%), (10%), and (12%) were prepared in a weight-weight ratio according to the following equation: (required nanoparticles weight = Adhesive weight × percentage of nanoparticles) [12]. The adhesive and seashell nanoparticles were accurately weighted by electrical sensitive balance (KERN, Germany). Then the two components were vigorously mixed in a semi-dark room with a plastic spatula on a glass slab until a homogenous form and uniform color of the adhesive material was obtained [13]. The modified orthodontic adhesive resin was placed in a sterile disposable syringe and covered with black tape to prevent exposure to the light. The same procedure was used for the preparation of zirconium oxide-modified orthodontic adhesive at three different concentrations (1%), (3%), and (5%), but with a new glass slab.

Samples, design, and Criteria of Samples Selection of the Study

Thirty-five sound-extracted upper premolars for orthodontic reasons were collected from governmental health centers and dental clinics in Mosul city and were kept in distilled water [14]. The sample size was calculated by using equation [$n = (z\ r/D)^2$]. In which, n = the number of samples, z (constant) = 1.96 for 95% confidence, r (standard deviation) = 0.2 (from previuos study), and D (precision) = 0.2. The resulted number will be adjusted and the final sample size in each group = (5). Carious teeth, teeth with enamel defects, and visible cracks were excluded from the study sample collections. The design of the study is described in Figure (1).

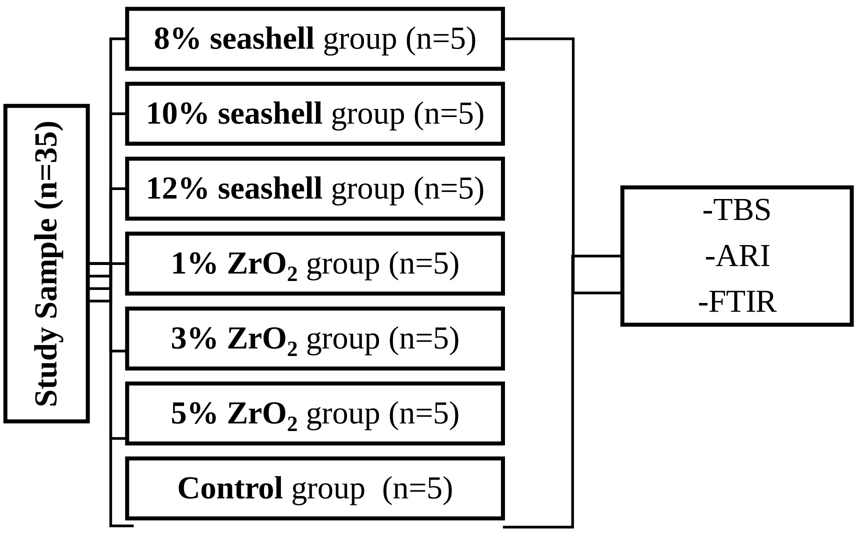

Figure (1): The Study Design

Samples Preparation

The collected teeth were brushed with a simple toothbrush to remove the adherent soft tissue and stored in distal water at room temperature [15]. Dental stone was poured to the half-height of a plastic (18 mm diameter) and (30 mm height) Poly Vinyl Chloride rings until set. Each tooth was cut from below the CEJ by a high-speed handpiece using a diamond fissure bur with copious water. Then auto polymerization cold cure acrylic resin was used to fill the rings, and before complete setting of acrylic resin, the lingual aspect of each tooth's crown was immersed into the acrylic resin in a way that the buccal aspect of the tooth was parallel to the floor [16].

Bonding Procedure

A low-speed handpiece (Mena, China) with rubber cups and fluoride-free pumice (PD, Germany) was used to polish each tooth sample for (10 sec.), rinsing with water, and drying the samples with airflow [17]. The buccal enamel surface of the samples was etched for (30 sec.) according to the manufacturer's instructions with phosphoric acid gel (37%) (SDI, Australia), rinsing with water for (15 sec.), drying until a chalky appearance was observed. The orthodontic adhesive was distributed by a dental probe on the base of the brackets, while it was held by a bracket clamp. To ensure correct positioning, the bracket was placed using a boons gauge on the buccal enamel surface (4 - 4.5 mm from the occlusal surface). We applied a known load attached to the articulator's arm (Quick perfect, France) and directed it vertically to the bracket slots of all specimens by the anterior pin of the articulator to ensure a standard pressure and produce uniform adhesive layer thickness and to prevent air bubbles [18]. Removal of excess adhesive from the boundaries of the brackets was performed by a sharp dental explorer. Curing was done using a light-curing device (Benq, china) with intensity (1500 MW/cm) and (420-480 nm wavelength), at a 2 mm distance from the edges of the bracket base in all samples. The total curing time was (40 sec.) for each sample, (and 20 sec.) at each mesial and distal side of the bracket [19]. The specimens were placed in distilled water for 24 h. at 37°c, [20]. We used a curing radiometer (China) to measure the light intensity through the polymerization of all specimens to ensure steady light intensity.

Fourier Transform Infrared Spectrometry (FTIR)

FTIR spectrophotometer device (Alpha, Bruker, Germany) at the (Dentistry College, Mosul University) was used for the identification of the chemical characteristics of conventional orthodontic adhesive material before and after modification with seashell and zirconium oxide nanoparticles. Seven drop-like samples were prepared as a drop of Heliosit orthodontic adhesive and a drop of each concentration of modified orthodontic adhesives placed on a clean and sterile glass slab [16]. The samples were cured by the same time (40 Sec.), intensity, and curing device that was used for all study samples.

Tensile Bond Strength (TBS)

The metal brackets were circled by a (0.010) inch threaded stainless steel ligature wire; this will ensure equal distribution of force on both sides of the bracket wings. TBS was tested after 24 h of brackets bonding at Dentistry College, Mosul University, using a universal testing machine (Gester, China). The cross-head speed was (0.5 mm/min**)**. The testing machine applied a pulling force perpendicularly until the bracket debonding. TBS values were calculated using this equation: TBS in (MPa) = "Force in Newton's unit / the surface area of bracket base in mm^2" [21].

Adhesive Remnant Index (ARI)

The enamel surface of the buccal aspect of all specimens in the study was examined using a stereomicroscope (Optika, Italy) at (10X) magnification power. The criteria that were used for measuring ARI scores were: **(Score 0)** if there is no adhesive material remnant on the buccal enamel surface. **(Score 1)** if less than half of the adhesive material remained on the buccal enamel surface. **(Score 2)** if the remnant on the buccal surface of the tooth was more than half of the adhesive material. **(Score 3)** means that all adhesive material remained on the buccal surface of the tooth, with a distinct mesh impression of the bracket base [22].

RESULTS

TEM

Results of TEM examination reveals that the size of both seashell and zirconium oxide nanoparticles ranged from (25-75) nm and the particles were round or oval as seen in Figure (2).

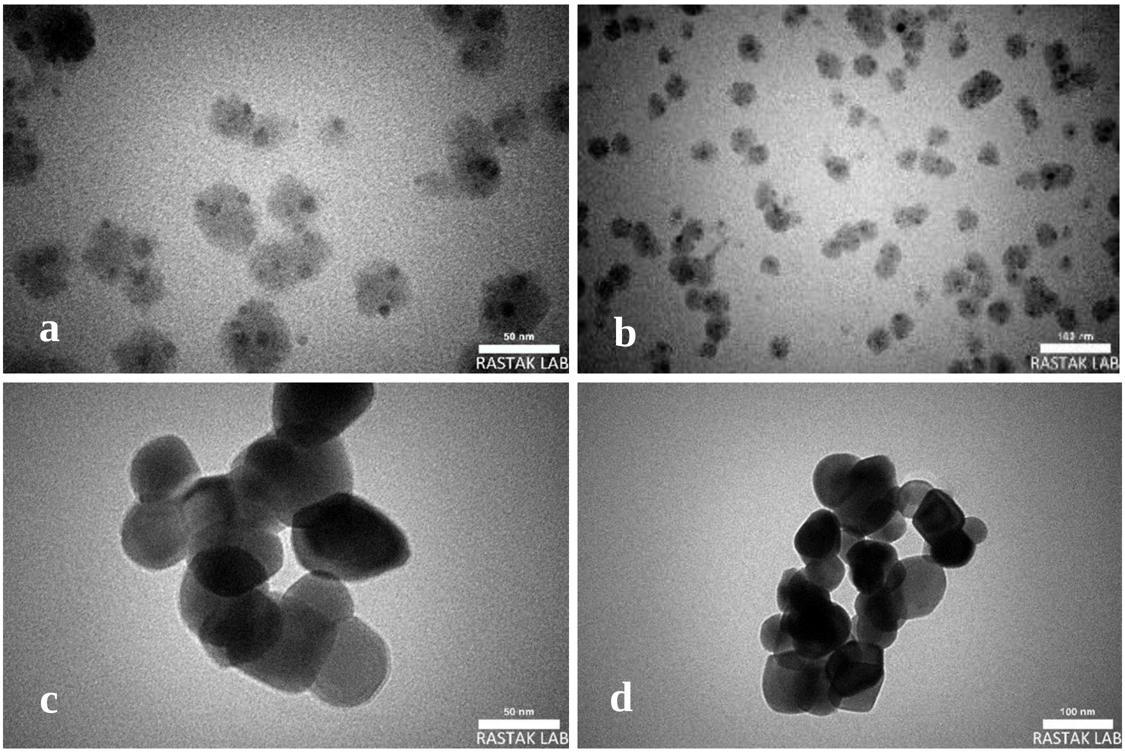

Figure (2): TEM images. a: Seashell Particles at 50 nm Scale, b: Seashell Particles at 100 nm Scale, c: ZrO_2 Particles at 50 nm Scale, d: ZrO_2 Particles100 nm Scale

FTIR

Figure (3) illustrates the appearance of some bands at 2956, 1717, and 1296 wave-number-cm^{-1}, by FTIR spectra of a control group which is due to C-H stretching and C-O., the same bands at the same region appeared by FTIR spectra of the modified orthodontic adhesive, which showed no shifting or disappearing bands when compared with control group spectra.

TBS

The data of all groups were normally distributed, the significant values in all groups were greater than (0.05) according to Shapiro-Wilk tests. Table (1) shows the descriptive statistics including the sample number per group, minimum, maximum values, and mean ± standard deviation, in addition to "Duncan's Multiple Range" test. According to these results, the 10% seashell group had the highest TBS mean value followed by 8% seashell group, while the control group showed the lowest TBS mean values. The data analysis by one-way ANOVA showed that a significant difference was present among the mean TBS values of the groups in the study where the significant value was (0.037). A more sensitive "Duncan's Multiple Range" test among all groups of the study showed that 10% of seashell groups significantly had the highest TBS mean value followed by 8% seashell group, the control group had the lowest TBS mean value with no significant difference with other groups in the study.

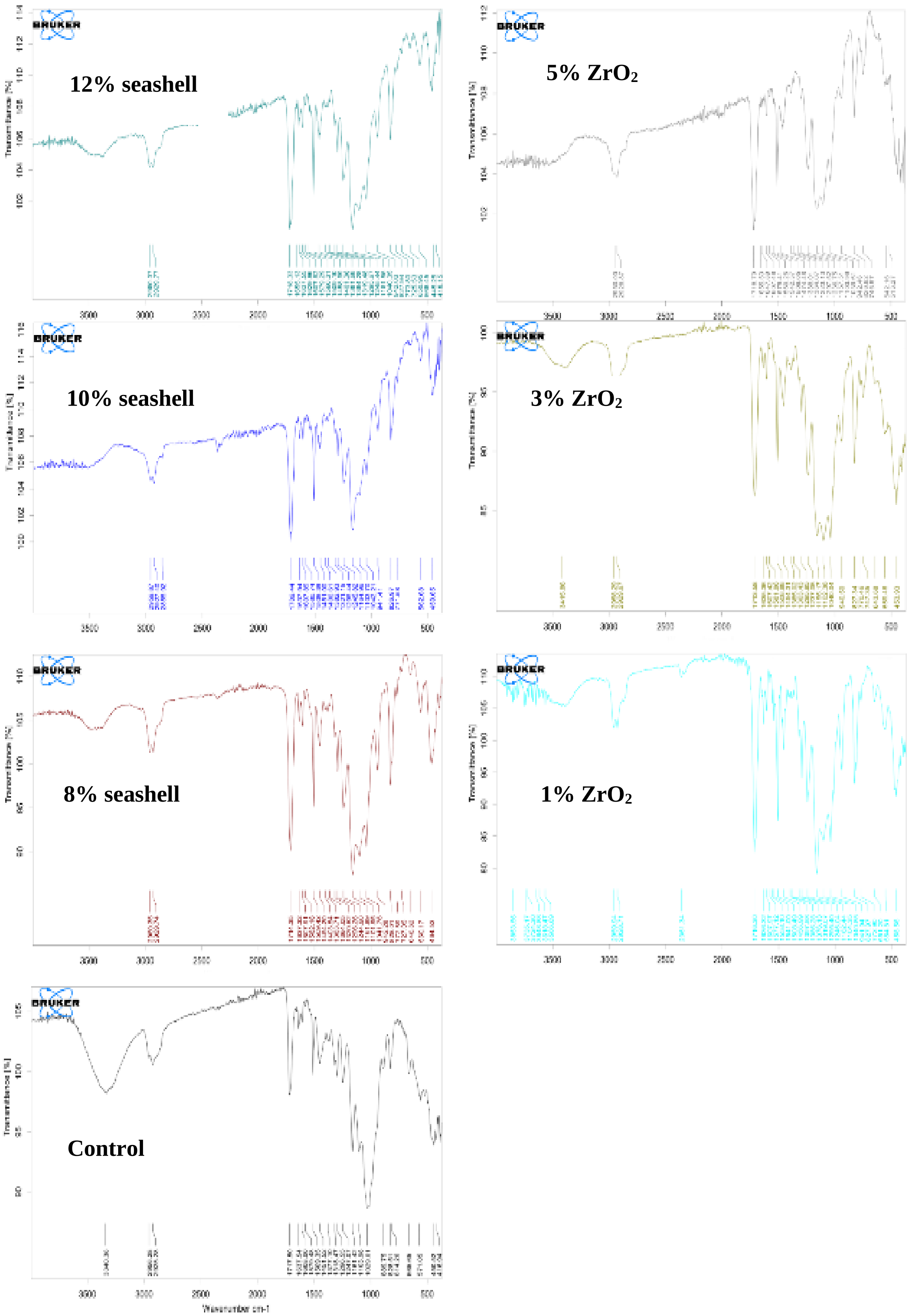

Figure (3): FTIR Spectra for all study group

Table (1): Descriptive Statistics of TBS

Groups	N	Min	Max	Mean ± SD.
Control	5	3.00	6.67	4.76 ± 1.37 (a)
8% seashell	5	2.44	8.89	6.33 ± 2.63 (ab)
10% seashell	5	7.22	8.56	7.67 ± 0.55 (b)
12% seashell	5	3.78	5.56	4.87 ± 0.70 (a)
1% ZrO_2	5	4.44	7.22	5.55 ± 1.11 (a)
3% ZrO_2	5	3.22	6.67	5.49 ± 1.51 (a)
5% ZrO_2	5	4.11	6.33	5.13 ± 0.79 (a)
Sig.				0.037

N: number of samples, **Min:** minimum, **Max:** maximum, **SD:** standard deviation, **ZrO_2:** zirconium oxide nanoparticles, **sig.** significant level, Different small letter means significant differences between groups at $P \leq 0.05$.

ARI

Table (2) represents the descriptive statistics of ARI including the sample number per group, minimum, maximum score, and mean ± standard deviation for each group in the study. Analysis of data subjected that the least mean scores were in the control and 10% seashell groups, while the highest mean scores were in the 12% seashell group. The percentages of the mean score for each group in the study are described in Figure (4), showing that the 10% seashell group and the control group have the least percentage of the mean score. However, no significant differences were presented by the Kruskal-Wallis test among the mean scores of the groups compared to the control group as the significant value was (0.374).

Table (2): Descriptive Statistics of ARI

Groups	N	Min	Max	Mean ± SD
Control	5	1	3	1.8 ± 0.84
8% seashell	5	2	3	2.6 ± 0.55
10% seashell	5	0	3	1.8 ± 1.30
12% seashell	5	2	3	2.8 ± 0.45
1% ZrO_2	5	2	3	2.4 ± 0.55
3% ZrO_2	5	1	3	2.2 ± 0.84
5% ZrO_2	5	2	3	2.6 ± 0.55
Sig.				0.374

N: number of samples, **Min:** minimum, **Max:** maximum, **SD:** standard deviation, **ZrO_2:** zirconium oxide nanoparticles, **sig.** significant level at $P \leq 0.05$.

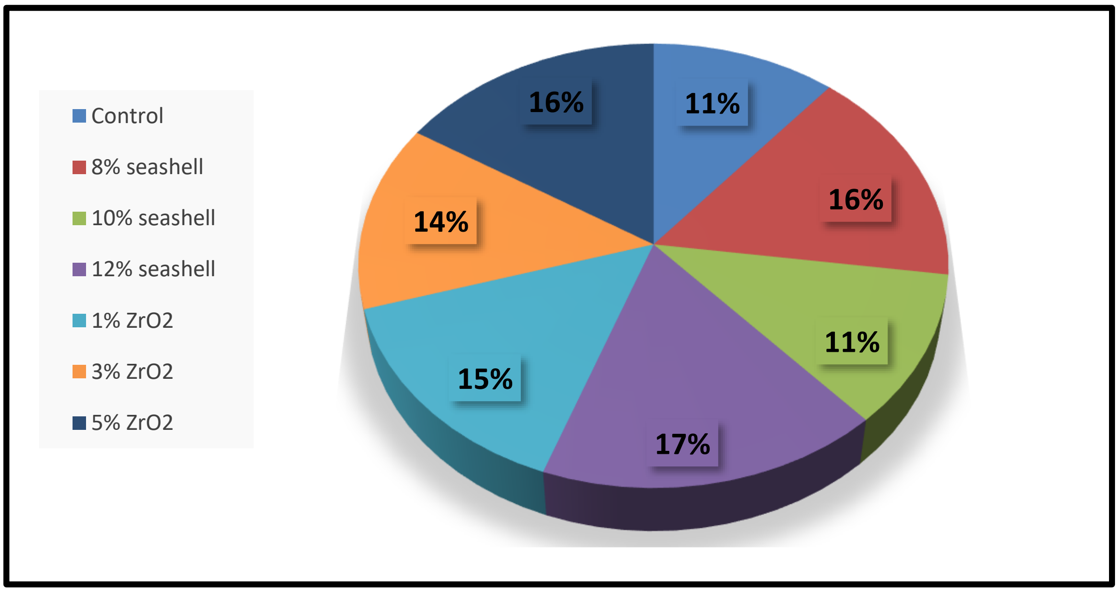

Figure (4): Percentages of ARI Mean Scores for All Groups

DISCUSSION

This study employed a possible solution to enhance the bonding strength of the orthodontic adhesive and prevent the de-bonding of orthodontic brackets, which was the addition of seashell and ZrO_2 nano-filler to the orthodontic adhesive. nHA contains calcium and phosphate remineralizing ions and is highly biocompatible [(23)]. These advantages encouraged us to incorporate nHA derived from natural seashells in the adhesive due to affordable production and biocompatibility and analyze their particle's shape and size by TEM, measure TBS to represent the experimental group's ability to resist de-bonding force, explore the remnant of orthodontic adhesive material on the buccal enamel surfaces after brackets de-bonding by ARI. and identify a possible chemical reaction between nanoparticles and adhesive by FTIR.

Observation of seashell and ZrO_2 nanoparticles under TEM revealed that the particle's sizes were ranging from (25-75) nm with round or ovoid shapes, the spherical-shaped particles have a lubricating effect on the material so increasing its flowability, whereas amourphous particles cause an increase in its viscosity according to Aljamhan, *et al.*, (2021) [(23)].

In the current study, the mean TBS values of (8, 12 %) seashell and (1, 3, 5 %) ZrO_2 groups were higher than the mean value of the control group with no statistically significant differences among them, except (10%) seashell group was significantly higher than all experimental and control groups.

Lee *et al.*, (2010) studied the Physical properties of resin-reinforced glass ionomer cement modified with nano-hydroxyapatite, they found that (10%) nano-HA presented the greatest bonding strength with a significant difference with the control group [(24)], these results agreed with our (10%) seashell result.

Aljamhan *et al.,* (2021), worked on micro-TBS of modified adhesive resin after different dentin surface manipulation and they concluded that "the presence of nano-HA particles (10 wt. %) in the adhesive increased its bond strength" [(23)], this result agreed with our result.

The elevation in TBS mean value after the addition of (8, 10, 12%) seashell groups may be due to the remineralization of the enamel surface by the action of calcium hydroxyapatite, which is releases OH group, and the shifts of the environment to the basics and improve the bonding efficiency as stated by Zhang and Wang, (2012) [(25)].

The reduced TBS mean value of 12% seashell group less than (8, 10%) groups may be due to the use of a high concentration of nanoparticles that could decrease bond strength due to increased viscosity [(26)]. This result agreed with Elsharkawy (2018), who concluded that (20%) of added Nano filler yielded lesser micro tensile strength than (10%) [(27)].

Increased mean TBS values of three different concentrations (1, 3, 5%) ZrO_2 groups may be due to it being a biocompatible material [(28)] or maybe nano-size inorganic fillers have a high surface area that can improve the interfacial adhesion resulting in greater bond strength. [(29)]. Our results agreed with Felemban and Ebrahim (2017), who concluded that the addition of ZrO_2–TiO_2 nanoparticles on resin-based adhesives increases TBS of the adhesive [(13)], this improvement may be due to the presence of ZrO_2 nanoparticles. Also, our study results are in agreement with Gad *et al*., (2018), who investigated the effect of three concentrations (2.5%, 5%, and 7.5%) of ZrO_2 nanoparticles on the translucency and tensile strength of the poly methyle methacrylate (PMMA) denture base material, the mean tensile strength values in all test groups were higher than the control group [(30)]. All study groups exhibited better performance on TBS than the control group as their mean values were higher than the (2.86 MPa) accepted mean value suggested by Fajen *et al*., (1990) [31].

According to our results of ARI, there was no significant difference among all study groups that's mean the addition of different concentrations of (1,3,5%) ZrO_2 and (8,10,12%) seashell nanoparticle didn't increase the amount of adhesive that remained on the tooth surface after de-bonding. Low ARI scores may be beneficial because it leads to easy orthodontic bracket removal, less iatrogenic damage to the tooth by the orthodontist, and an easy re-bonding procedure during orthodontic treatment [32].

FTIR spectra for the control and modified adhesive groups with (1,3,5%) ZrO_2 and (8.10.12%) seashell nanoparticles, showed the same bands and positions without shifting or changing. That means the modification of Heliosit orthodontic adhesive with seashell and ZrO_2 nanoparticle were not revealed any chemical reactions or changes or formation of new material.

CONCLUSIONS

The addition of (8, 10, 12 %) Seashell containing nano-hydroxyapatite and (1, 3, 5 %) zirconium oxide nanoparticles had better performance and improved tensile bond strength of Heliosit orthodontic adhesive. The addition of seashell and zirconium oxide nanoparticles neither compromised the amount of adhesive that remained on buccal enamel surfaces after the de-bonding of brackets nor induced any chemical reaction with Heliosit orthodontic adhesive.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

REFERNCES

1. Roelofs, T., Merkens, N., Roelofs, J., Bronkhorst, E., & Breuning, H. *A retrospective survey of the causes of bracket-and tube-bonding failures.* Angle Orthod. 2017; 87(1): 111-117.
2. Sharan, J., Singh, S., Lale, S.V., Mishra, M., Koul, V., & Kharbanda, O.P. *Applications of nanomaterials in dental science: A review.* J. Nanosci. Nanotechnol. 2017; 17(4): 2235–2255.
3. Song, W. & Ge, S. *Application of Antimicrobial Nanoparticles in Dentistry*. Molecules. 2019; 24(6): 1033.
4. Zakrzewski,W., Dobrzynski, M., Dobrzynski,W., Zawadzka-Knefel, A., Janecki, M., Kurek, K., Lubojanski, A., Szymonowicz, M., Rybak, Z., & Wiglusz, R.J. *Nanomaterials Application in Orthodontics*. Nanomaterials. 2021; 11(2): 337.
5. De Dios Teruel, J., Alcolea, A., Hernández, A., & Ruiz, A. J. O. *Comparison of chemical composition of enamel and dentine in human, bovine, porcine and ovine teeth.* Arch Oral Biol. 2015; 60(5): 768-775.
6. Juntavee, N., Juntavee, A., & Plongniras, P. *Remineralization potential of nano-hydroxyapatite on enamel and cementum surrounding margin of computer-aided design and computer-aided manufacturing ceramic restoration*. Int J Nanomedicine. 2018; 13: 2755-2765.
7. Seyedmajidi, S., Rajabnia, R., & Seyedmajidi, M. *Evaluation of antibacterial properties of hydroxyapatite/bioactive glass and fluorapatite/bioactive glass nanocomposite foams as a cellular scaffold of bone tissue.* J Lab Physicians. 2018; 10(3): 265-270.
8. Nobre, C.M.G., Pu¨ tz, N., & Hannig, M., *Adhesion of Hydroxyapatite Nanoparticles to Dental Materials under Oral Conditions*. Scanning. 2020, ArticleID: 6065739: https://doi.org/10.1155/2020/6065739.
9. Majhooll, A.A., Zainol, I., Jaafar, C.A, Alsailawi, H.A., Hassan, M.Z., Mudhafar, M., Majhool, A.A., & Asaad, A. *A Brief Review on Biomedical Applications of*

Hydroxyapatite Use as Fillers in Polymer. J Chem Chem Eng. 2019; 13: 112-119.

10. Al-Saleh, S., Alateeq, A., Alshaya, A. H., Al-Qahtani, A. S., Tulbah, H. I., Binhasan, M., Shabib, S.; Farooq, I.; Vohra, F & Abduljabbar, T. *Influence of TiO2 and ZrO2 nanoparticles on adhesive bond strength and viscosity of dentin polymer: A physical and chemical evaluation.* Polymers. 2021; 13(21): 3794.
11. Alhussary, B. N., A Taqa, G., & Taqa, A. A. *Preparation and characterization of natural nano hydroxyapatite from eggshell and seashell and its effect on bone healing*. JAVS. 2020; 5(2): 25-32.
12. Hasan, L. A. *Evaluation the properties of orthodontic adhesive incorporated with nano-hydroxyapatite particles.* Saudi Dent J. 2021; 33(8): 1190-1196.
13. Felemban, N. H., & Ebrahim, M. I. *The influence of adding modified zirconium oxide-titanium dioxide nano-particles on mechanical properties of orthodontic adhesive: an in vitro study*. BMC Oral Health. 2017; 17(1): 1-8.
14. Cerone, M., El-Badrawy, W., Gong, S.G., & Prakki, A. *Bond Strength of Universal Self-Etch 1-Step Adhesive Systems for Orthodontic Brackets.* J Can Dent Assoc. 2019; 85(j6): 1488-2159.
15. Eslamian, L., Borzabadi-Farahani, A., Karimi, S., Saadat, S., & Badiee, M. R. *Evaluation of the shear bond strength and antibacterial activity of orthodontic adhesive containing silver nanoparticle, an in-vitro study*. Nanomaterials. 2020; 10(8): 1466.
16. Al Khayat, Z. E., & Al Hamdany, A. K. S. *Shear and Tensile Bond Strengths of Titanium Dioxide Nanoparticles Modified Orthodontic Adhesive*. IJERMDC. 2018; 5 (12): 1-10.
17. Tonetto, M.R., De Campos, E.A., Fernández, E., Kuga, M.C., De Andrade, M.F. & Bandéca, M.C. *Bond Strength and Adhesive Remnant Index of Experimental Brackets Bonded with Self-adhesive Resin Cement*. Rev. Clin Periodoncia Implantol Rehabil Oral. 2017; 10 (2):115-117.
18. Degrazia, F.W., Genari, B., Leitune, V.C., Arthur, R.A., Luxan, S.A., Samuel, S.M., Collares, F.M. & Sauro, S. *Polymerization, Antibacterial and Bioactivity Properties of Experimental Orthodontic Adhesives Containing Triclosan-loaded Halloysite Nanotubes*. J Dent. 2018; 69: 77-82.
19. Blöcher, S., Frankenberger, R., Hellak, A., Schauseil, M., Roggendorf, M.J. & Korbmacher-Steiner, H.M. *Effect on Enamel Shear Bond Strength of Adding Microsilver and Nanosilver Particles to the Primer of an Orthodontic Adhesive.* BMC Oral Health. 2015; 15 (1): 1-9.
20. Vargas O.A, E., Nuernberg C., C., Maciel V.B, J., & Bolognese M, A. *Influence of Primekote_ polymer in orthodontic bonding*. Rev Odontol UNESP. 2017; 46 (2): 61–65.
21. Enan, E., Tawfik, M. A., Mehesen, R., & Basha, S. *Remineralization Potential and Shear Bond Strength of Surface Treated Hypomineralized Enamel in Bonding of Orthodontic Brackets: An In Vitro Study*. J Adv Oral Res. 2021; 12(1): 127-133.
22. Yaseen, S. N., Taqa, A. A., & Al-Khatib, A. R. *The effect of incorporation Nano Cinnamon powder on the shear bond of the orthodontic composite (an in vitro study)*. J Oral Biol Craniofac Res. 2020; 10 (2): 128-134.
23. Aljamhan, A. S., Alrefeai, M. H., Alhabdan, A., Alhusseini, S. A., Farooq, I., Vohra, F., Naseem, M., & Alkhudhairy, F. *Influence of ER-CR-YSGG Laser and Photodynamic Therapy on the Dentin Bond Integrity of Nano-Hydroxyapatite Containing Resin Dentin Adhesive: SEM-EDX, Micro-Raman, Micro-Tensile, and FTIR Evaluation.* Polymers. 2021; 13 (12): 1903.
24. Lee, J. J., Lee, Y. K., Choi, B. J., Lee, J. H., Choi, H. J., Son, H. K., Hwang, J.W., & Kim, S. O. *Physical properties of resin-reinforced glass ionomer cement modified with micro and nano-hydroxyapatite*. J

Nanosci Nanotechnol. 2010; 10(8): 5270-5276.

25. Zhang, Y., & Wang, Y. *Improved degree of conversion of model self-etching adhesives through their interaction with dentine*. J dent. 2012; 40(1): 57-63.
26. Wagner, A., Belli, R., Stotzel, C., Hilpert, A., Muller, F. A., & Lohbauer, U. *Biomimetically-and hydrothermally-grown HAp nanoparticles as reinforcing fillers for dental adhesives*. J Adhes Dent. 2013; 15(5): 413-422.
27. Elsharkawy, D.A. *Effect of Incorporation of Bioactive Glass Nano Particles in Adhesive Resin on Microtensile Bond Strength to Dentin in Self Etch and Etch and Rinse Mode*. A.D.J. 2018; 5(4): 399-404.
28. Yousefi, K., Manesh, H. D., Khalifeh, A. R., & Gholami, A. *Fabrication and Characterization of a Nanofast Cement for Dental Restorations.* BioMed Res Int. 2021.
29. Ashraf, M. A., Peng, W., Zare, Y., & Rhee, K. Y. *Effects of size and aggregation/agglomeration of nanoparticles on the interfacial/interphase properties and tensile strength of polymer nanocomposites.* Nanoscale Res Lett. 2018; 13(1): 1-7.
30. Gad, M. M., Abualsaud, R., Rahoma, A., Al-Thobity, A. M., Al-Abidi, K. S., & Akhtar, S. *Effect of zirconium oxide nanoparticles addition on the optical and tensile properties of polymethyl methacrylate denture base material.* Int J Nanomedicine. 2018; 13: 283.
31. Fajen, V.B., Duncanson, M.G., Nanda, R.S., Currier, G.F. & Angolakar, P.V. *An in vitro Evaluation of Bond Strength of Three Glass Ionomer Cements.* Am J Ortho Dentofacial Orthop. 1990; 97(4):316-322.
32. Kechagia, A., Zinelis, Z., Pandis, N., Athanasiou, A.E. & Eliades, T. *The Effect of Orthodontic Adhesive and Bracket-Base Design in Adhesive Remnant Index on Enamel*. J World Fed Orthod. 2015; 4(1):18-22.