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Research Article

The effect of zirconia dipping time frequencies with different nanoparticles suspension on the micro-shear bond strength of zirconia to resin cement

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Abstract: Background: Zirconia restoration attracted great attention because of its high mechanical and esthetic properties that meet the requirements of anterior and posterior restoration. This study aimed to evaluate the micro-shear bond strength between resin cement and zirconia surfaces coated with different nanooxides with different dipping time frequencies. Materials and Methods: HT Zirconia blocks (n=55) were divided into 5 groups according to the coating suspensions and the frequency of dipping times. Group A: dip in 50% SiO_2+50%ZrO_2 for 1min in frequency 4 times, group B: dipping in 50% SiO_2+50% ZrO_2 for 4min in frequency of 1 time, group C: dipping in 50% SiO_2+50%Al_2O_3 for 1min in frequency of 4 times group D: dipping in 50%SiO_2+50%Al_2O_3 for 4min in frequency of 1 times, group E: nontreated zirconia (control group). The coated surfaces were characterised using Field Emission Scanning Electron Microscopy (FESEM), X-ray diffraction (XRD), Energy Dispersive Spectroscopy (EDS), and atomic force microscopy (AFM). Resin cylinders were cemented onto the treated zirconia surfaces, and micro shear bond strength testing was performed. One-way ANOVA and Tukey's post hoc test analyzed differences in bond strength between groups ($p<0.05$). Results: FESEM showed nanosilica-nano-zirconia coatings full coverage with porosity within the coating, and nanosilica-nano-alumina shows full coverage with crosslinking of the silica-alumina on the surface of Zirconia with creating a high ratio of porosity within the coating. XRD detected only a tetragonal phase in coated and nontreated zirconia, indicating no phase transformation in the coated zirconia surface by the dipping technique. The microshear bond strength was higher for all coating groups versus controls ($p<0.05$). Conclusion: The dip coating with the nanoparticle suspension consists of 50%SiO_2+50%Al_2O_3 with a dip frequency of 1min for 4 times better than continuous dipping

Keywords: Alumina, Zirconia, Silica, Shear Strength, Bonding, Nano Coating

Introduction

Recently, zirconia has become more popular in dentistry due to its improved aesthetic properties along with excellent mechanical, good biocompatibility and its highest fracture strength among all dental ceramics, which is more than 1,000MPa(1–5). The need for a durable bond of zirconia ceramic to resin cement is essential in clinical work. The inertness and stability leads to difficulty in the establishment of durable chemical and mechanical bond to resin cement.

The durable bond can be established for zirconia by activating both mechanical bonds, which are established by roughening the zirconia surface, and the chemical bond by using 10-MDP (6). The effective way to roughen and cleaning the surface is by sandblasting with alumina particle (size 50-110 µm at 0.25 MPa) or by surface grinding to enhance the bonding to zirconia ceramic (7,8). However, the surface after air abrasion can lead to reduction in the strength and zirconia surface damage (9). So, alternative methods for surface treatment may be needed to improve zirconia bonding to resin cement to avoid the damaging effect of airborne particle abrasion (10), coating the zirconia with ceramic powder (11), or using a porcelain pearl layer with low fusing temperature as examples of alternative conditioning methods.

Recently, an alumina nanostructured coating (Al_2O_3) and silica nanostructure coating was developed for zirconia ceramics, by which the establishment of a strong resin bond was established to partially stabilized tetragonal zirconia ceramics with yttria (Y-TZP) with high durability (3). This surface is produced using the dip coating technique which has been proven to have a good bond strength of zirconia to the resin cement In addition to surface conditioning, bifunctional molecules, which promote the chemical bonding of zirconia ceramics luting resins chemical bonding, should be considered for bonding . A bi functional molecule such as the 10-methacryloxydecyl- di hydrogen phosphate (MDP), which promotes chemical bonding to zirconia ceramic, is contained in composite resins and primers (12).

In several studies, it has been demonstrated that primer containing provided durable bond strength to zirconia ceramic after airborne particle abrasion of the bonding surface (13), while primer without MDP did not achieve durable bonding (14). Creating a coating layer on the surface of zirconia with its thickness and morphology may effect the bonding strength as combination of nanostructured alumina coating and nano silica coating seems to be effective.

In creating microretention, providing favorable resin bonding (13). However, it is unknown whether the frequency of the dip time may effect on the bonding, different studies stated the efficiency of silica-zirconia dip coatings as a feasible method that could significantly improve the resin-zirconia bond strength (15) ,others stated that the tuning effect of the suspension dip coating of zirconia alumina nano oxides had a dramatic effect on the micro-shear bond strength (3).

Therefore, the purpose of this in vitro study was to evaluate the influence of two frequencies of dipping time with nanosilica nanoalumina and nano-silica and nano-zirconia on the bond strength of a luting resin. The null hypothesis of the strength of the study was that the resin bond to zirconia wouldn't be affect by the type of dip-coated nano oxide suspension, also the zirconia dipping frequencies such as layering or continuous dipping would not have any effect on the microshear bond strength between zirconia and resin cement.

Materials and Methods

Zirconia disc VITA YZ^R HT high translucent zirconia (Vita zahnfabrik H, Germany), Silica nanopowder 20 nm (US research Nanomaterials, Inc., USA), Alumina nanopowder 20nm (US research Nano-materials, Inc., USA), Zirconia nanopowder 20nm (US research Nano-materials, Inc., USA), Z-prime plus (Bisco Inc. Schaumburg. USA), G-CEM ONE, self-adhesive resin cement (GC Corporation, Tokyo, Japan)

Preparation of the Zirconia Disk

Vita YZ HT zirconia cylinders (diameter: 100mm, height: 18mm) cut into 55 sample with dimensions (8×8×2.5mm) using low speed saw machine (MTI Corporation) all surfaces of the sample were smoothened by using grinder polisher (MDP200 dual speed grinder polisher) with silicon carbide papers 1200 grid of silicon with and an ultrasonic bath (GT sonic device) was used for cleaning up to 10 minutes, all specimen were placed in an oven for one hr. for drying under 100 C. The zirconia dip coating suspension of nano oxide particles (SiO_2, ZrO2, and Al_2O_3) was prepared, and samples were grouped according to the volume fraction of the percentage of nanooxide powder and frequencies of dipping time frequencies in the dip-coating suspension, each group 11samples, 8 for the micro shear bond strength test and 3 for characterisation, as follow :

Group A: Zrconia dipped in suspension contain 50%V_f SiO_2 + 50% V_f ZrO_2 dipped for 1min with a frequency of 4 times

Group B : Zirconia dipped in suspension contains 50% V_f SiO_2+ 50% V_f ZrO2 dipped for 4min with frequency of 1 time.

Group C: Zirconia dipped in suspension contains 50% V_f SiO_2+50% V_f AL_2O_3 dipped for 1 min with a frequency of 4 times

Group D: Zirconia dipped in suspension contains 50% V_f SiO_2+50% V_f AL2O3 dipped for 4 min with a frequency of 1 time.

Group E: Zirconia without surface treatment

Preparation of Zirconia Dip-Coating Solution

The nanooxide powder was mixed with 20 ml of 99 % methanol (Spain). Continuous mixing of the solution with a magnetic stirrer for 30 min then sonicate the solution in an ultrasonic bath for 1 hr, followed by adding 1 ml of polyvinyl alcohol (India) and mixing for 30 min; then using a high-power ultrasonic probe for 10 min (Hielscher ultrasonic probe). The samples were dipped in each suspension for a 1 min for frequency of 4 times(the sample was dipped for 1 min then removed from suspension to dry for 1 min, then dipped again and the procedure repeated for 4 times) and 4 min for frequency of 1 time(the sample dipped in the suspension continuously for 4 minutes then removed).

Zirconia Sample Sintering:

ZYRCOMAT 6000 MS was used to sinter the samples into two steps according to the manufacturer's instructions. Step 1: 850 ° C for 15 min, Step 2: 1450 °C for 120 min Characterization

Three randomly selected samples from each group were then analysed using a field emission scanning electron microscope(FESEM), energy dispersive X-ray spectroscopy EDS (thermo scientific), an X-Ray Diffractometer (XRD) (PANalytical,), and atomic force microscopy(AFM; NaioAFM ,the samples for FESEM were prepared by coating with a gold thin film by the sputtering technique to increase the surface electrical conductivity

This laboratory study was used to evaluate the effect of zirconia surface treatment with dip coating technique using different nano oxide particle and different dipping time frequencies on microshear bond strength

Micro-shear bond strength (μSBS) testing

Micro-shear testing was used to check the bond strength(16). First, the zirconia surface was covered with a tape that had a hole. Then a zirconia primer "Z- Prime". Let it dry. After that, injection of resin cement (G CEM ONE) into a Teflon mould measuring 1 mm in diameter and 3 mm in height. It was cured with light for 10 seconds following the manufacturer's instructions. This process created a resin cement sample that was attached to the zirconia surface.

For the bond strength test, a testing machine with a knife-edge shearing tool was used. The tool was placed perpendicular to the zirconia resin interface(17). Then a speed of 0.5 mm/min until the bond failed. The bond strength was measured in units of MPa.

Statistical analysis

Data were analysed using SPSS version 26. The Shapiro-Wilks test was used to assess the normality of the data, the one-way test ANOVA and the Tuckey post hoc test were used to compare groups at a significance level of $p < 0.05$.

Mode of failure

After µSBS test, the failure mode was studied using a digital microscope at 250X magnification.

The failure mode was categorised into 3 types as follow

Adhesive I: failure in the interface between the zirconia substrate and the coating.

Adhesive II: failure in the interface between the coating and the resin cement.

Cohesive: failure within the resin cement.

Mixed failure (adhesive II /cohesive): failure occurs in both coating and Resin(3)

Results

Microstructure analysis

The zirconia surface morphology was examined by scanning electron microscopy (FESEM) after coating procedures. The FESEM images of zirconia (group E) showed a smooth surface, with minimal scratches and grooves caused by polishing (Figure1A1). At higher magnification (10KX), the zirconia grains were tightly packed and visible with grain boundaries in close contact without gaps or defects (Figure 1A2).

Group A: showed a full coverage coating with discontinuity in the coating layer (figure 1B1), at high magnification(10KX) (figure 1B2) shows a zirconia nano particle disperse in the fused silica with a porosity within the coating.

While the group shows full coverage of the coating on the zirconia substrate with more coating continuity than in group A (Figure 1C1), at higher magnification (10KX) (Figure 1C2), nanosilica powder diffused in fused silica shows, but with higher quantity than group A and little porosity within the coating.

The FESEM of group C shows more Zirconia surface coverage coated with nanoparticles with less Zirconia substrate shown and full surface coverage (high ratio of coated to uncoated surface area), at high magnification (10KX) (Figure 1D2) cross linking of the silica- alumina upon the surface of Zirconia with the creation of a high ratio of porosity within the coating

Although for group D, the homogeneous islands coating showing behind some Zirconia surface (Figure 1E1) at high magnification (10KX) (Figure 1E2) the nano Alumina particles dispersed in the melted Silica on the surface of Zirconia appear as a highly porous layer.

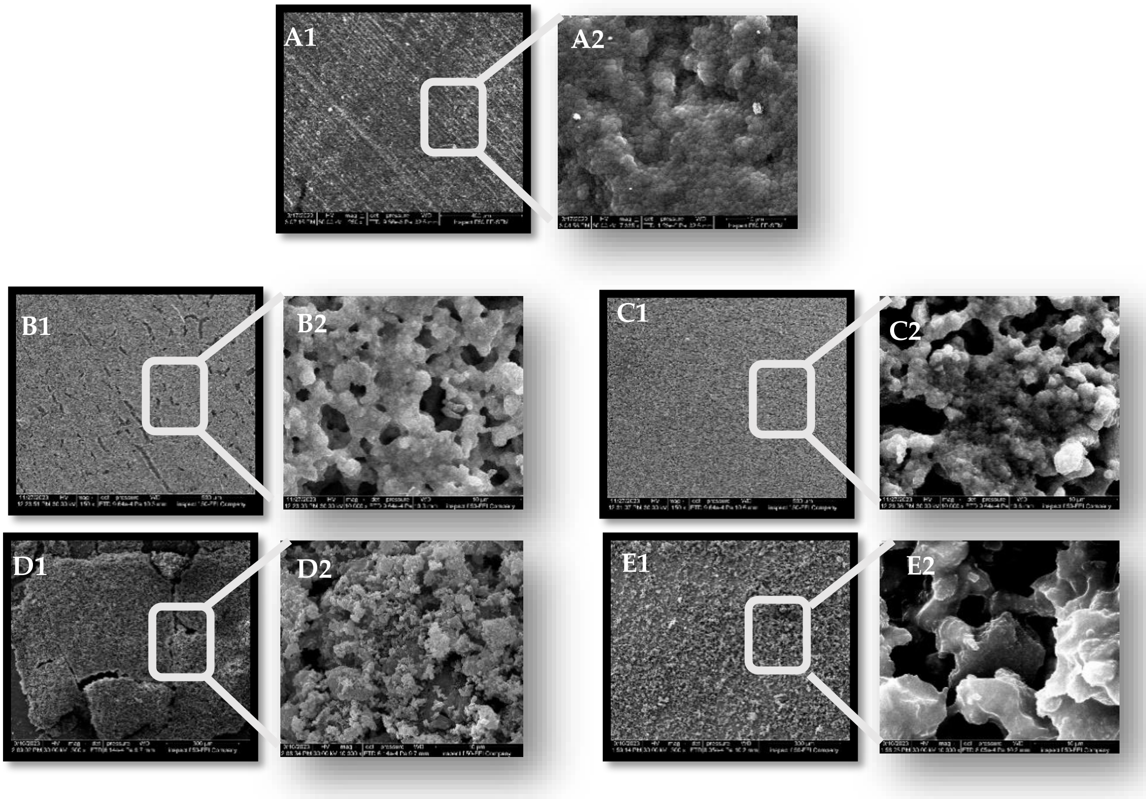

Figure 1: FESEM of HT zirconia,A1untreated zirconia at low magnification, A2: high magnification,B1: Zirconia coated with 50% SiO_2+50%ZrO_2 at a: 1 min at frequency of four times at low magnification, B2:high magnification,C1: Zirconia coated with 50% SiO_2+50%ZrO_2 at: 4 min at frequency of 1 time at low magnification ,C2:high magnification,D1: Zirconia coated with 50% SiO_2+50% Al_2O_3 at: 1 min at frequency of 4 times at low magnification,D2:high magnification, E1: Zirconia coated with 50% SiO_2+50% Al_2O_3 at: 4 min at frequency of 1 time at low magnification, E2: high magnification

Elemental analysis

The EDS spectrum (Table 1) shows the elements of oxygen, yttrium, hafnium, and zirconium without the presence of silica or alumina on the zirconia surface of the untreated zirconia. For groups A and B, the EDS shows the presence of Si in addition to the previous element on the surface, and the percentage of Zr is high compared to the untreated zirconia substrate as the coating is mainly zirconia and silica with slight effect of the frequency of dipping time. In group C and D, shows the presence of Si and Al in addition to the previous element on the surface, and the percentage of zirconia substrate is less than that of other groups due to the coating of the zirconia substrate with silica and alumina nanoparticles and that the Si% and Al % increase with increasing the frequency of time, (4.7 to 13.7Wt%) for silicon and (21.8 to 28.6Wt%) for aluminium while the zirconium decrease from 17.8 to 3.5 Wt% for zirconium decreases from 17.8 to 3.5 Wt%

Table 1: Element weight % of EDX result for HT zirconia groups

Groups	**Element weight percentage (Wt %)**					
	O	**AL**	**Si**	**Zr**	**Y**	**Hf**
A	23.2	0	6.6	65.9	4.3	
B	24.3	0	7	63.1	5.5	
C	55.5	21.8	4.7	17.8		
D	52.9	28.6	13.7	3.5	1.1	
E	38	0	0	50.4	7.4	2.0

XRD Characterization

XRD analysis provided critical insights into the crystallographic phases present in zirconia after different nanocoating treatments. When diffraction peak angles and intensities are measured, the atomic lattice structure can be elucidated. For untreated polished zirconia controls, distinct XRD peaks were observed at 2θ values of 30.3°, 35.2°, 50.5°, 60.0°, and 62.8°, corresponding to the crystal planes (101), (110), (112), (211) and (202) crystal planes of the metastable tetragonal zirconia phase (18). The nanosilica-zirconia and silica-alumina coated samples showed identical peaks, indicating that the coatings did not disrupt the zirconia tetragonal lattice structure (Figure 2). Preserving the tetragonal phase is crucial for maintaining optimal mechanical properties in zirconia restorations (19) (20).

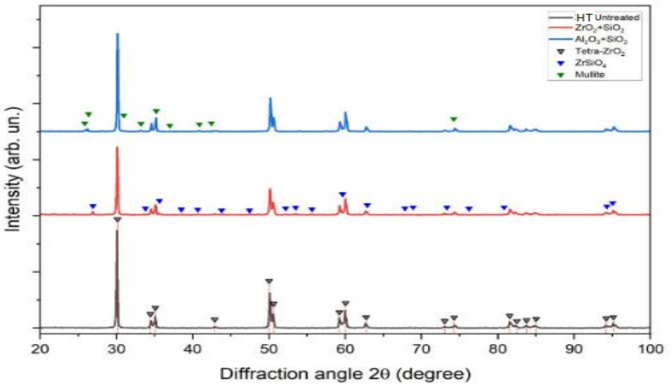

Figure 2: XRD patterns of coated and uncoated zirconia surfaces with crystallographic phases.

Topographic analysis

Atomic force microscopy (AFM) were used to study the surface topographies of the coated and noncoated surfaces. The surface roughness of each surface was measured in nanometer. The surface roughness of zirconia dipped in 50% SiO_2+50%ZrO_2 and 50% SiO_2+50%Al_2O_3 dipped for 1 min with frequency of 4 times is higher (934nm and 600 nm, respectively) than zirconia dipped for 4 min with frequency of 1 time (842.7nm and 218 nm, respectively), with a high surface roughness for the silica zirconia system Table 2.

Table 2: Surface roughness of different Zirconia dipping groups.

GROUPS	Surface roughness (nm)
A	934
B	842.7
C	600
D	218
E	244.8

Micro-shear bond strength (μSBS)

The micro-shear bond strength value are shown in Table 3. With their standard deviation.

One-way ANOVA showed that surface treatments significantly affected the strength ($P \leq 0.05$). Nanosilica-zirconia and nanosilica-alumina with different proportions and dipping frequencies to the zirconia surfaces showed significantly higher μSBS than the untreated zirconia groups ($p < 0.05$).

The highest value of μSBS is for group C (50% SiO_2+50Al_2O_3 with a dip of 1 min with frequency of 4 times) (49.28±4.19MPa) than for all other groups with significant differences, while group E (nontreated zirconia) has the lowest μSBS (22.43±4.05MPa) than other dip groups with significant differences than group A,C and D. The dipping suspension content had a great effect on the μSBS, with the highest μSBS was for zirconia that is dipped in silica alumina suspension than zirconia dipped in silica zirconia suspension with both dipping frequencies. On the other hand, the dipping frequency also affect on the μSBS, it was found that dipping for 1 min with frequency of 4 times had a higher microshear bond strength than dipping for 4 min with one frequency for both silica alumina and silica zirconia suspension.

Table 3: Micro-shear bond strength results for resin cement bonded to zirconia surfaces with different coating treatments.

Group	Mean MSBS (MPa)	Std. Deviation
A	$35.69^{c,e*}$	6.72
B	$29.6^{c,d}$	7.26
C	$49.28^{a,b,d,e}$	4.19
D	$38.51^{b,c,e}$	7.11
E(Non-treated)	$22.43^{a,c,d}$	4.05

*small letters related to significant differences among groups

Mode of failure

The mode of failure for each group had been studies under the digital microscope as shown in Table 4 to distinguish the nature of failure according to the separation area between the coating and the cement of the zirconia of the surface and resin and coasting from another side.

The post-fracture can be seen in Figure (3)

Table 4: Mode of failure of treated and untreated zirconia

Group (HT) Dipping	Adhesive I	Adhesive II	Mixed(adhesive/ Cohesive	Cohesive	Total
A		4(50%)	4(50%)		8(100%)
B		7(87.5)	1(12.5%)		8(100%)
C		4(50%)	3(37.5%)	1(12.5%)	8(100%)
D		5(62.5%)	2(25%)	1(12.5%)	8(100%)
E (Non-treated)	8(100%)				8(100%)

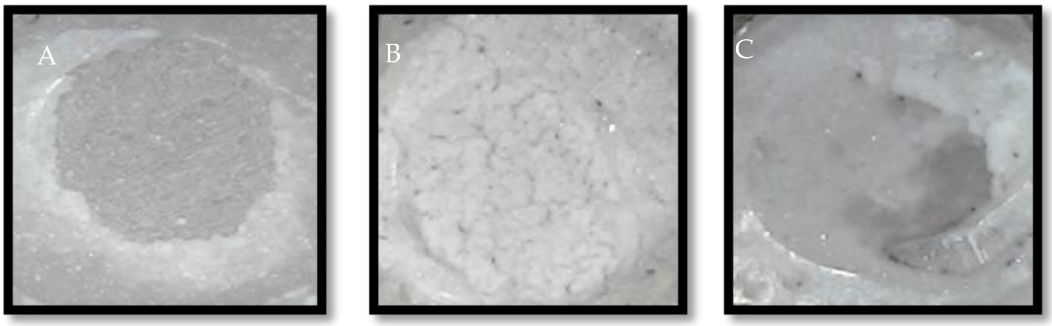

Figure 3: Mode of failure. A: adhesion failure, B: adhesive failure, C: mixed failure

Discussion

The bond strength test is more reliable and clinically acceptable using Microshear bond strength tests rather than using macroshear bond strength to eliminate false positive results, so in this study microshear bond strength testing was adopted, in which the interfacial adhesion of zirconia to the resin is quantified after zirconia surface treatment using a dip coating technique (21) (22). Microshear testing is performed by bonding small cylindrical samples and applying an increasing shear force parallel to the interface (16) (23). Failure occurs by deboning at the weakest point, which is often within the microscopic bonding complex. This provides a more representative measure of interfacial adhesive versus the cohesive strength of the cement itself (21).

In this study, all nano-suspension coatings significantly enhanced the zirconia bond strength compared to the polished zirconia surface. The polycrystalline zirconia surface without surface treatment provide no mechanical or chemical reactivity with resin cements, producing low bond strength (3) (20) (24) (25). But by creating nanoscale textures and porosities after zirconia dipping in nano suspensions, which is evidenced in FESEM, the coatings provide an increase in surface area and surface energy to be micromechanically retentive.

Dip coating is proven to be a good way to increase the resin bond to zirconia with a simple non-complicated procedure and provide better bond strength without compromising substrate properties compared to conventional abrasive blasting approaches in which surface roughness is induced by surface damage, cracks, and phase transformation (3) (15).

The 50:50 silica-alumina ratio provided an optimal balance between chemical and mechanical bonding with the resin cement when zirconia dipped for 1min with a frequency of 4 times as the roughness of the surface was high (600nm) which contributes to increase the mechanical bond strength and the presence of balance between the percentage of aluminium and zirconium elements (21.8,17.8Wt %respectively) as a sign for the presence of alumina and zirconia on the coating surface that enhance the chemical bonding between the phosphate group of the Z-prime and the zirconia and alumina .

The null hypothesis is rejected as it was shown that the frequency of increase in the dip coating has a direct effect on increasing the shear bond strength as it shows increased surface roughness, presence of higher porosity and presence of surface burrs and sharp edge that formed during the sintering of silica on the zirconia substrate with the zirconia nanoparticle for silica- zirconia system, and with the alumina nano particle for silica alumina system, as shown in FESEM and AFM, which contribute in higher μSBS as it cause increase in the mechanical bonding of both silica zirconia and silica alumina suspension. These observation supports the assumption that micro locking between zirconia and resin has increased

This high shear bond strength was reflected in the mode of fracture of group C, which showed that the adhesive strength of coating to the zirconia substrate was too high (adhesive I is 0%) and failure occur in the resin cement /coating interface (adhesive II) by 50 %and 37.5 % of mixed failure between adhesive II and cohesive 12.5% (Table 4)

The limitation of the study was that a lack of thermocycling and other factors such as mastication load and fluctuation of the pH level could considerably increase the degradation of the bond.

Conclusion

The dip coating method is simple and commonly used in laboratories and improves the shear bond strength between zirconia and cement.

Conflict of interest

The authors have no conflicts of interest to declare.

Authors' contributions

ZSA contributed to the main idea of the articles with writing MR contributed to the supervision and editing MH contributed to the project admiration All authors have read and agreed to the published version of the manuscript.

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Informed consent

Informed consent was obtained from all individuals, or their guardians included in this study.

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تأثير التكرارات الزمنية لغمس الزركونيا مع معلقات الجسيمات النانوية المختلفة على قوة رابطة القص الدقيقة للزركونيا مع الأسمنت الراتنجي

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المستخلص:

حظيت ترميمات الزركونيا باهتمام كبير بسبب خصائصها الميكانيكية والجمالية العالية التي تلبي متطلبات الترميم الأمامي والخلفي. قيمت هذه الدراسة تأثير تكرار مرات الغمس في معلقات الجسيمات النانوية المصنوعة من نانو-سيليكا نانو-زركونيا ونانو-سيليكا نانو-ألومينا على قوة رابطة القص الدقيقة لأسمنت راتينج الزركونيا. الطرق: تم تقسيم كتل الزركونيا **HT** (العدد = 55) إلى 5 مجموعات حسب معلقات الطلاء وتكرار مرات الغمس. المجموعة أ: الغمس في 50% **SiO_2 + 50% ZrO_2** لمدة دقيقة واحدة بتردد 4 مرات، المجموعة **B**: الغمس في 50% **SiO_2 + 50% ZrO_2** لمدة 4 دقائق بتردد واحد، المجموعة **C**: الغمس في 50% **SiO_2 + 50 %Al_2O_3** لمدة دقيقة واحدة بتردد 4 مرات المجموعة **D**: غمس في 50% **SiO_2+50%Al_2O_3** لمدة 4 دقائق بتردد 1 مرات، المجموعة **E**: زركونيا غير معالجة (مجموعة مراقبة). تم تشخيص الأسطح المطلية باستخدام المجهر الإلكتروني لمسح الانبعاثات الميدانية (**FESEM**)، حيود الأشعة السينية (**XRD**)، التحليل الطيفي المشتت للطاقة (**EDS**)، ومجهر القوة الذرية (**AFM**). تم لصق أسطوانات الراتنج على أسطح الزركونيا المعالجة وتم إجراء اختبار قوة رابطة القص الدقيقة. قام اختبار **ANOVA** أحادي الاتجاه واختبار **Tukey** اللاحق بتحليل الاختلافات في قوة الرابطة بين المجموعات (**P** <0.05). النتائج: أظهر **FESEM** تغطية كاملة لطلاءات نانو سيليكا نانو زركونيا مع مسامية داخل الطلاء، وأظهر نانو سيليكا نانو ألومينا تغطية كاملة مع تشابك السيليكا والألومينا على سطح الزركونيا مع خلق نسبة عالية من المسامية داخل الطلاء. اكتشف **XRD** الطور الرباعي فقط في الزركونيا المطلية وغير المعالجة، مما يشير إلى عدم وجود تحول في الطور في سطح الزركونيا المطلي عن طريق تقنية الغمس. كانت قوة رابطة القص الجزئي أعلى بالنسبة لجميع مجموعات الطلاء مقارنة بعناصر التحكم (**P** <0.05). الاستنتاجات: الطلاء الغمسي بمعلق الجسيمات النانوية يتكون من 50% **SiO_2+50%Al_2O_3** مع تردد غمس قدره دقيقة واحدة لمدة 4 مرات أفضل من الغمس المستمر.