Adhesion to Zirconia Restorations a Problem Can Be Solved

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ABSTRACT

This study was conducted to evaluate the effect of a chemically conditioning intaglio zirconia surface onmicro-shear bond strength of the dual cure resin cement bonded to the four types of yttria stabilized tetragonal zirconia (Y-TZP) ceramics as well as the effect of such conditioning on the topography of that surface. Fifteen plate for (M-SBS test)and nine plates for (AFM test)from each zirconia material milled by a CAD/CAM system, sintered and randomly divided into three groups. For each group, a different surface conditioning was applied: 30% Hydrogen Peroxide, 30%Citric acid and a control group. M-SBStest for aluting cement adherence to zirconia and the AFM test for the zirconia topography was determined and data were analyzed using one-way ANOVA, followed by Duncan's Multiple Range Test($p \leq 0.001$). Significant differences were found for the Citric acid in compared to Hydrogen Peroxide and control groups in relation to the cement adhesion value as well as the surface roughness significantly increased when the intaglio surface of the UPCERA and Multilayer zirconia plates conditioned with Citric acid in compered to other types of zirconia tested.

Keywords: bonding, cement, ceramic, microshear, resin, surface metrology.

INTRODUCTION

In dentistry, zirconia-based ceramics have become a very popular type of all-ceramic restorations used due to their esthetical, mechanical strength, biocompatible, and cost-effective alternative to ceramic-metal restorations[1,2].

Zirconia is the name given to zirconium dioxide (ZrO_2), a polycrystalline material that exhibit a more than one crystalline structure depending on pressure and temperature condition. Pure zirconia is monoclinic (M) at room temperature. This phase is stable up to 1170°C. It will transform into a tetragonal (T) phase under higher temperatures and later into a cubic phase (C) at 2370°C[3].

The yttria tetragonal zirconia polycrystal (Y-TZP), a type of zirconia used in dentistry. The yttria is added to zirconia to stabilize the structure and maintain the materials desirable properties.Y-TZP ceramics have a unique characteristic of "Stress Induced Transformation" that gives them superior mechanical properties compared to other ceramics and that is why this material is referred to as a" Ceramic Steel"[4].

Although improved mechanical properties are important for the long-term performance of a ceramic material, the clinical success of fixed ceramic prostheses seems to be strongly dependent on the cementation procedure. The conventional methods of adhesive cementation include prior hydrofluoric acid etching of the ceramic surface and further salination, are not efficient for zirconia ceramics because of their lack of silica and glass phase[5].

Adhesion is still an issue, since a very low and unstable resin bond is obtained when the Y-TZP ceramic is untreated or has received primer application only. Clinically, crown de-bonding (loss of retention) is a type of failure of zirconia-based restorations, and the research for surface treatments that improve resin adhesion to zirconia has increased in intensity[5,6].

Various conventional mechanical test methods, been used to assess dental adhesion. More recently, some researchers advocated a new test method using specimens with reduced dimensions, as a substitute for the conventional shear test: the so-called "microshear" bond strength (M-SBS) test[7]. The M-SBS test would allow small areas to be tested that provides a higher bond strength mean, a better stress distribution accomplished since the number of voids and stress-raising factors is lower than the ones that possibly occur in larger areas[8].In spite of there are several surface treatment methods and studies that have evaluated the efficacies of bonding cement to zirconia, there is still no consensus regarding the best surface treatment method for achieving optimal bond strength between zirconia and resin cement[9].Thus, the aim of this study was to evaluate the M-SBS of a dual cure luting resin cement bonded to the intaglio surface of the sintering zirconia and the influence different chemical solutions conditioning prior to the application of resin cement. In addition to evaluate possible effect of such conditioning on the intaglio surface topography of the zirconia materials.

MATERIAL AND METHODS

For the M-SBS test, a (60) specimens were made of zirconia plates measuring (15.0mmx 5.0mmx 1.0 mm) and milled of a compact Y-TZP zirconia blocks, (15) for each of (Framework Blank UPCERA HT White, China (UH), Full Dental Zirconia Blank colored with Zircoloy™ Coloring system (BC)and non colored (BN) BruxZir® Solid Zirconia, USA, and Full Dental Zirconia Blank Copran® Zr-i Monolith Symphony, White-peaks Multilayer , Germany (MM)using the yen dent dental CAD/CAM systems after a soft ware data designed program prepared to obtain such dimension for specimens. Once milling process was completed (Figure 1), the specimens were carefully separated and removed from the zirconia blanks, margins of each specimen were adjusted with a football shaped and fine fissure diamond burs (379-023M-HP, NTI-Kahla GmbH rotary instruments, Germany) to remove any excess.

Then the zirconia plates were sintered in furnace Kavo Everest® Therm, Kavo, Germany) to a temperature and time according to their manufactural instructions. Each specimen has a two base surfaces, the opposing and bonding (intaglio) surfaces. The opposing surface of the specimens that prepared from (BC, BN, and MM) were fine ground, polished, cleaned, dried, and finally a glaze firing to simulate the external finished surface of the extra coronal restoration and to eliminate any evidence of flaws and defects on that surface. But the opposing surface of the specimens prepared from (UH), remain as it to simulate the external surface of the core for the extra coronal restoration. Then, all specimens ultrasonically cleaned for (15) minutes before any testing by using Bio Sonic® UC50DB.

Figure 1: The attached pre-sintered specimen after milled from the zirconia blank.

The specimens of each type of the zirconia materials were randomly assigned into three groups according to the type of the surface conditioning preformed on their intaglio surface, (5) specimens for each group (control,30% Hydrogen Peroxide (HP) (TEDIA company INC, China) applied for (5) min., and 30% Citric Acid(CA) (Loba Chemie Pvt. Ltd., India) applied for (10) min.,

A Tygon mold (grade type "standard" (size 6) Œ 0123. Absaugkatheter, Germany) with a central hole measuring (0.97 mm diameter, and 1 mm height) were placed in the center of the intaglio surface of the specimen and fixed in place by aid of an orthodontic wax (Figure 2). The hole of a Tygon tube of a (5) specimens of each type of zirconia materials remain as it (control group) while the other (10) specimens of each type of zirconia materials undergo a certain chemical surface conditioning either by a HPor a CA(Figure 3), by the aid of a disposable dental applicator to performed a chemical surface conditioning. Later the mold will be washed using tap water followed by a genital air dryness to the hole of the mold.

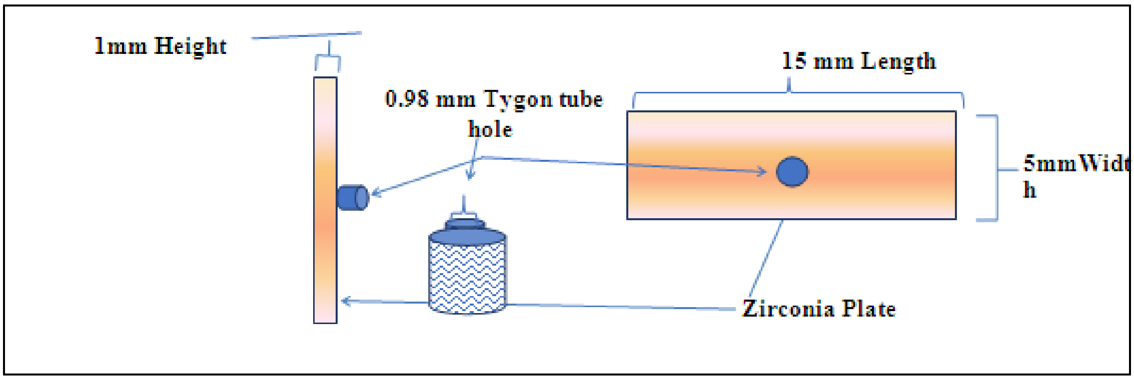

Figure 2: Diagram of specimen preparation for the micro-shear bond test

A Dual cure cement RelyX Unicem Maxicap Self-Adhesive Resin Cement (3M ESPE, USA) (RX) mixed and applied within a mold according their manufactural instructions by aided of an aplicap capsule applier supplied within a cement kit, then a clear sallied stripe was placed over the cement, pressed gently and photo-cured from opposite side of the zirconia plate (with the tip of the curing unit closed to the surface of plate). All bonding procedures were carried out by a single operator at a room temperature of (22±2)°C. The specimens stored in distilled water for 24 hours in an incubator at 37 °C[10,11].

later, the Tygon tube separated from the cement rod and removed by aid of a shaving knife, all the samples were checked under a digital camera (at 10 X with 12 megapixel picture resolution) for bonding area defects. The samples that showed apparent interfacial gap formation, bubble inclusion, or other obvious defects were excluded and replaced with new samples testing[12].

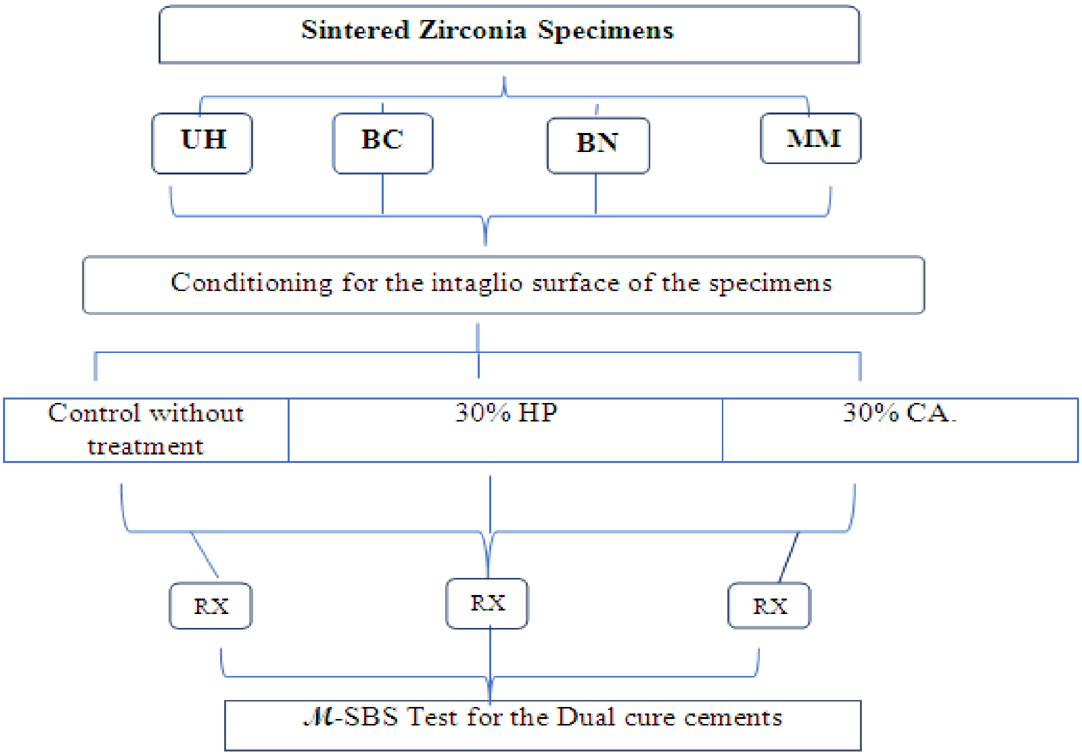

Figure (3): Experimental design for Micro-shear bond strength test (M-SBS) for the Dual cure cement bonded to the zirconia intaglio surface.

The M-SBS was performed by using a universal test machine (Sans Testing Machine Co. Ltd, Shenzhen China) within which a modified micro-shear bond testing apparatus was fixed. A modified micro-shear bond testing apparatus were prepared in which the tested device was suspended on a metal plate that was connected to a load cell (Pressure sensor 5 kg HX 711 AD module, JAPAN). The ends of a thin thread (Mint Waxed Dental Floss, 0.2 mm in diameter) looped and fixed to screws placed with in the metal plate of the test device. Which intern connected to the upper part of the universal test machine by action of a magnetic force. The zirconia specimen was placed and fixed within a slot in a metal bar by aiding of two screws, the metal bar weld to a rod that in turn connected to the base of the universal test machine by action of a magnetic force. For testing the thread looped around the resin cement cylinder, making contact through half. The zirconia specimen was placed and fixed within a slot in a metal bar by aiding of two screws, the metal bar weld to a rod that in turn connected to the base of the universal test machine by action of a magnetic force as shown in (Figure 4). For testing the thread looped around the resin cement cylinder, making contact through half its circumference and was gently held flush against the resin /zirconia interface[13]. as shown in (Figure 5).

A shear force was applied to each specimen at a cross-head speed of 0.5 mm/min until failure occurred. The micros hear forces were recorded in Newtons (N) and calculated as megapascals (MPa) by dividing to the bonding area (mm^2). The resin zirconia interface, the thread loop and the center of the load cell were aligned as straight as possible to ensure.

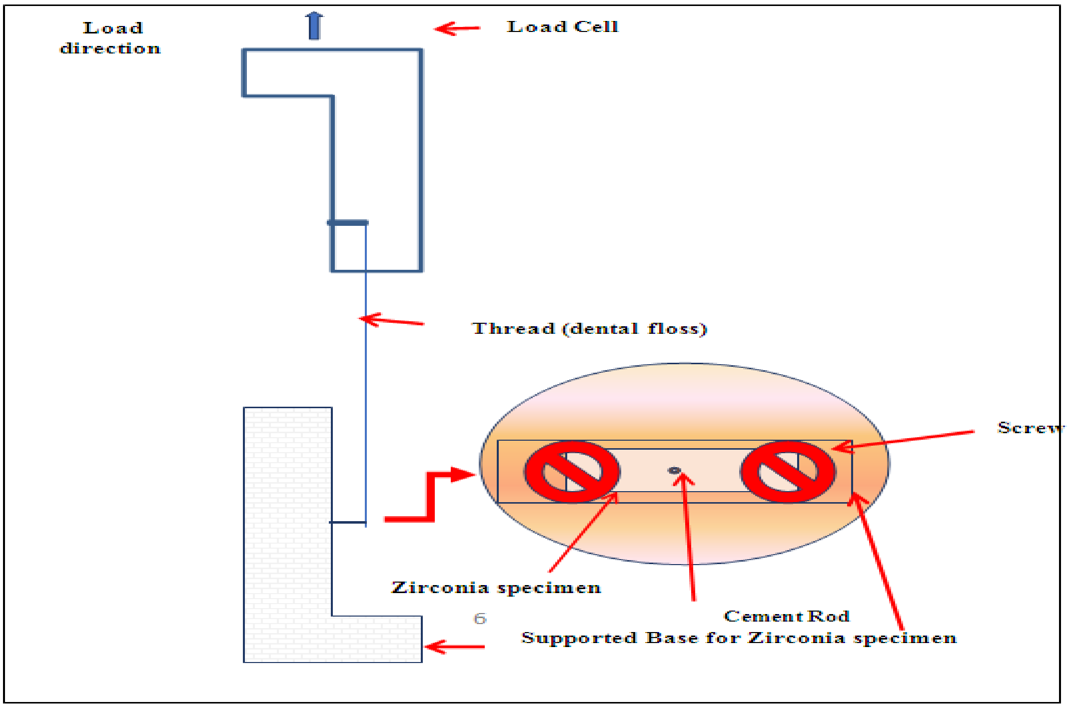

Figure (4): Schematic diagram of the Modified micro-shear bond test apparatus.

the desired orientation in the shear test force [14].All the modifications were done under supervision of Laboratory Staff of Mechanical Engineering Collage in Mosul University.

After testing, the de bonded surfaces of all specimens were examined under an Optical Microscope, (B-159, OPTIKA) at 10 X magnification to identify the types of failure which

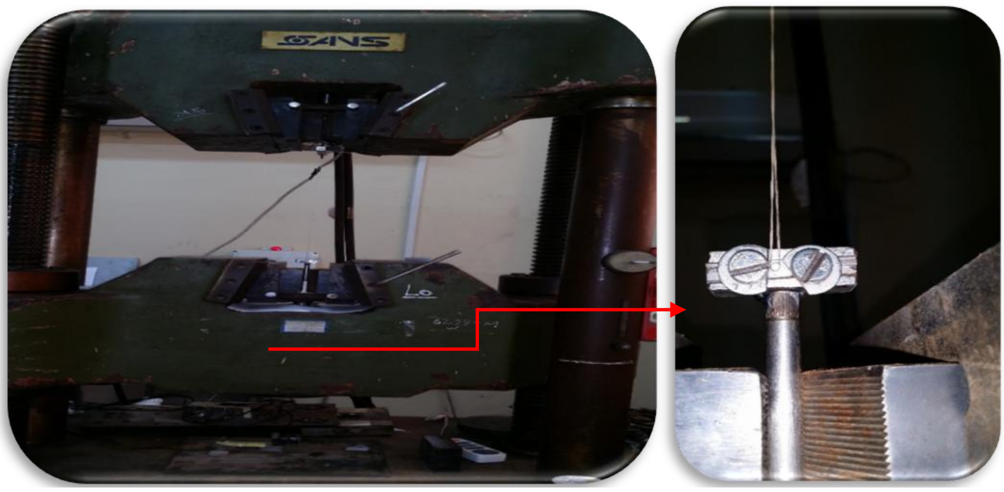

Figure (5): Universal Test Machine (Sans Testing Machine Co. Ltd, Shenzhen China) with a modified supporting apparatus and zirconia specimen.

theoretically could have occurred: 1)-adhesive failure between zirconia and adhesive system, 2)-Cohesive failures within the adhesive system [15].

To evaluated the effect of a chemical conditioning on the topography of the intaglio surface of zirconia materials, the atomic Force Microscope (AFM) test was used. A(36)specimens of zirconia (nine for each of UH, BC, BN, and MM)prepared, divided and a chemically conditioning in the same manner mentioned above for M-SBS test except the cement application step. The intaglio surface of zirconia materials topography was tested by using the AFM(AA300, Angstrom Advancal Inc., Scanning Probe Microscope), two readings nearly in the center of the zirconia plate for each specimen, and 3D images with areas of ($144*10^{(6)}$ nm^2) were captured. The value of root mean square of roughness (Rq) was determined via the AFM software program.

STATISTICAL ANALYSIS

For statistical analysis, the micro Shear bond strength means of rod specimens of each cemented block in (MPa) and root mean square of roughness (Rq) data for the zirconia intaglio surface were calculated and the data analyzed using parametric one-way ANOVA at the confidence level of 95% and Duncan's Multiple Range Test utilized to compare the difference in value of mean of that data obtained.

RESULTS

Regarding to the M-SBS of luting cement to the different zirconia materials (UH, BC, BN, and MM) tested in this study, one-way ANOVA showed that there was no significant difference between control groups of all evaluated zirconia materials at $p \leq 0.001$, a similar result was obtained for the HP conditioned groups atp ≤ 0.001. In regarding to the CA conditioning groups, the result was differing from the other two conditioned groups (control and HP) and the one-way ANOVA showed that there was a significant difference between CA conditioned groups of all evaluated zirconia materials at $p \leq 0.001$ the as shown in (Table 1).Regarding to the AFM test, the Rq value of the intaglio surface for the different zirconia materials specimen(UH, BC, BN, and MM)tested in this study, one-way ANOVA showed that there was a significant difference

Table 1: One-way analysis of variance (ANOVA) showed the effect of the chemical conditioning on the zirconiaintaglio surface in relation to M-SBS value between the RX cement the tested zirconia materials

M-SBS value for the RX cement bonded to a chemicallyconditioning of the intaglio surface of zirconia materials		**Sum of Squares**	**df**	**Mean Square**	**F**	**Sig.**
Control	Between Groups of zirconia materials	3.581	3	1.194	1.288	.313
	Within Groups of zirconia materials	14.834	16	.927		
	Total	18.415	19			
30% HP	Between Groups of zirconia materials	1.759	3	.586	1.585	.232
	Within Groups of zirconia materials	5.919	16	.370		
	Total	7.678	19			
30% CA	Between Groups of zirconia materials	4.078	3	1.359	6.161	.005
	Within Groups of zirconia materials	3.530	16	.221		
	Total	7.608	19			

between control groups for all evaluated zirconia materials at $p \leq 0.001$, and a similar result was obtained for the HP, CA conditioned groups atp ≤ 0.001as shown in (Table 2) .

To determine the level of significantthat obtained,Duncan's New Multiple Range Test showed the means and standard deviations (SD) of **M** -SBS value of RX cement To determine the interaction for the each type of the tested zirconia materials with the chemical surface conditioning solutions in relation to the M-SBS value of the RX cement with the intaglio surface of the tested zirconia materials as well as theRqvaluefor the that zirconia surface for each type of tested zirconia materials in this study, one-way.

Table (2): One-way analysis of variance (ANOVA) showed the effect of the chemical conditioning on the zirconia intaglio surface in relationto root mean square of roughness(Rq) value of surface.

Rq value for the chemically conditioning of the intaglio surface of zirconia materials.		Sum of Squares	df	Mean Square	F	Sig.
Control	Between Groups of zirconia materials	7213.908	3	2404.636	1517.251	.000
	Within Groups of zirconia materials	31.697	20	1.585		
	Total	7245.606	23			
30 % HP	Between Groups of zirconia materials	1999.955	3	666.652	3451.442	.000
	Within Groups of zirconia materials	3.863	20	.193		
	Total	2003.818	23			
30 % CA	Between Groups of zirconia materials	14121.377	3	4707.126	40643.489	.000
	Within Groups of zirconia materials	2.316	20	.116		
	Total	14123.693	23			

bonded to zirconia intaglio surface and the (Rq)value of the zirconia intaglio surface in relation to the type of a chemical surface conditioning as shown in (Table3).

Table (3): Means and standard deviations of bond strength results (MPa)of RX cement bonded to zirconia intaglio surface and the (Rq)value of thezirconia intaglio surfacein relation to the type of a chemical surface conditioning.(different letters in superscript indicate statistical differences with in the same group of conditioning for theDuncan's New Multiple Range Test. ((UH): UPCERA;(BC): BruxZir colored; (BN): BruxZir non colored; (MM): Multilayer. Zirconia materials); HP: Hydrogen Peroxide, CA: Citric acid: M-SBS: micro-Shear test, (Rq).: root mean square of roughness; (RX) RelyX Unicem Cement.

Type of testing	chemically conditioning solution	Zirconia materials			
		UH	**BC**	**BC**	**MM**
M-**SBS value of the RX bonded to the chemically conditioning zirconia intaglio surface**	Control	5.11±0.44 [a]	3.97±0.71 [a]	4.22±0.44 [a]	4.43±0.63 [a]
	30% HP	6.92±0.45 [a]	6.92±0.45 [a]	6.26±0.83 [a]	6.41±0.62 [a]
	30% CA	8.39±0.68 [a]	9.04±0.15 [b]	7.81±0.39 [a]	8.16±0.49 [a]
Rq value for the Chemically conditioning zirconia intaglio surface	Control	39.6±0.88 [c]	20.67±0.53 [b]	18.38±0.41 [a]	61.45±0.47 [d]
	30% HP	42.62±0.57 [c]	32.83± 0.32 [b]	29.52±0.35 [a]	52.9±0.47 [d]
	30% CA	50.8±0.47 [c]	18.79± 0.32 [b]	18.08±0.33 [a]	76.19±0.18 [d]

ANOVA test showed a significant difference between the control, HP and CA groups at $p \leq 0.001$as shown in (Table 4 and 5 respectively),and to determine level of significant obtained,Duncan's New Multiple Range Test showed means and standard deviations (SD) of **M** -SBS value of RX cement bonded to zirconia intaglio surface and the Rqvalue of thatzirconia surfacein relation to the type of a chemical surface conditioning used as shown in (Table 6).

After **M** -SBS testing, the deboned surfaces of all zirconia specimens were examined under an optical microscope (B-159, OPTIKA, ITALY) at (10 X) magnification to identify the failure modes. Failure modes were categorized as: a) adhesive: failure between RX cement and zirconia materials, no luting cement remnants left on zirconia materials surface. b) cohesive: failure within the cement themselves, no evidence of fracture within the cement surface obtained.

Table (4): One-way analysis of variance (ANOVA) results of the surface M-SBS value of RX cement bonded to each type of zirconia materials and their interaction with the chemical surface conditioning.

Tested Zirconia Materials		Sum of Squares	df	Mean Square	F	Sig.
UH	Between Groups of the chemical treatment	27.093	2	13.546	47.258	.000
	Within Groups of the chemical treatment	3.440	12	.287		
	Total	30.532	14			
BC	Between Groups of the chemical treatment	64.996	2	32.498	30.897	.000
	Within Groups of the chemical treatment	12.622	12	1.052		
	Total	77.618	14			
BN	Between Groups of the chemical treatment	32.376	2	16.188	47.467	.000
	Within Groups of the chemical treatment	4.092	12	.341		
	Total	36.468	14			
MM	Between Groups of the chemical treatment	28.446	2	14.223	25.108	.000
	Within Groups of the chemical treatment	6.797	12	.566		
	Total	35.243	14			

Figures (6 and 7) showed a representative positive and negative scanning prop microscope (SPM) images for the geometry, shape, and size of the particles of zirconia material tested in this study obtained from the AFM test for their intaglio surface in a control and after a chemically conditioning state.

Table (5): One-way ANOVA results of the (Rq) value of the intaglio surface of each tested zirconia materials and their interaction with the chemical surface conditioning.

Tested Zirconia Materials		Sum of Squares	df	Mean Square	F	Sig.
UH	Between Groups of the chemical treatment	403.014	2	201.507	97.289	.000
	Within Groups of the chemical treatment	31.068	15	2.071		
	Total	434.083	17			
BC	Between Groups of the chemical treatment	697.530	2	348.765	2145.219	.000
	Within Groups of the chemical treatment	2.439	15	.163		
	Total	699.968	17			
BN	Between Groups of the chemical treatment	510.367	2	255.184	1943.845	.000
	Within Groups of the chemical treatment	1.969	15	.131		
	Total	512.337	17			
MM	Between Groups of the chemical treatment	1666.137	2	833.068	5205.738	.000
	Within Groups of the chemical treatment	2.400	15	.160		
	Total	1668.537	17			

Table (6): Means and standard deviations of for each type of zirconia materials and their interaction with the chemical surface conditioning in relation to the **M** -SBS value of RX cement bonded to their intaglio surface and the (Rq)value of theirintaglio surface. (different letters in superscript indicate statistical differences with in the same group of conditioning for theDuncan's New Multiple Range Test. ((UH): UPCERA; (BC): BruxZir colored; (BN): BruxZir non colored; (MM): Multilayer. Zirconia materials); HP:HydrogenPeroxide,CA: Citric acid: M-SBS: micro-Shear test, (Rq).: root mean square of roughness; (RX) RelyX Unicem Cement.

Type of testing	Zirconia materials	chemically conditioning solution		
		Control	30% HP	30% CA
M-**SBS value of the RX bonded to the chemically conditioning zirconia intaglio surface**	UH	5.1±0.4 [a]	6.9±0.5 [b]	8.4±0.7 [c]
	BC	3.9±0.7 [a]	6.9±0.5 [b]	9.1±0.2 [c]
	BC	4.2±0.4 [a]	6.3±0.8 [b]	7.8±0.4 [c]
	MM	4.7±0.6 [a]	6.5±0.6 [b]	8.1±0.5 [c]

Rq value for the Chemically conditioning zirconia intaglio surface	UH	39.6±0.9 [a]	42.6±0.6 [b]	50.8±0.5 [c]
	BC	20.6±0.5 [b]	32.8±0.3 [c]	18.8±0.3 [a]
	BC	18.4±0.4 [a]	29.5±0.4 [b]	18.1±0.3 [a]
	MM	61.5±0.5 [b]	52.9±0.5 [a]	76.2±0.2 [c]

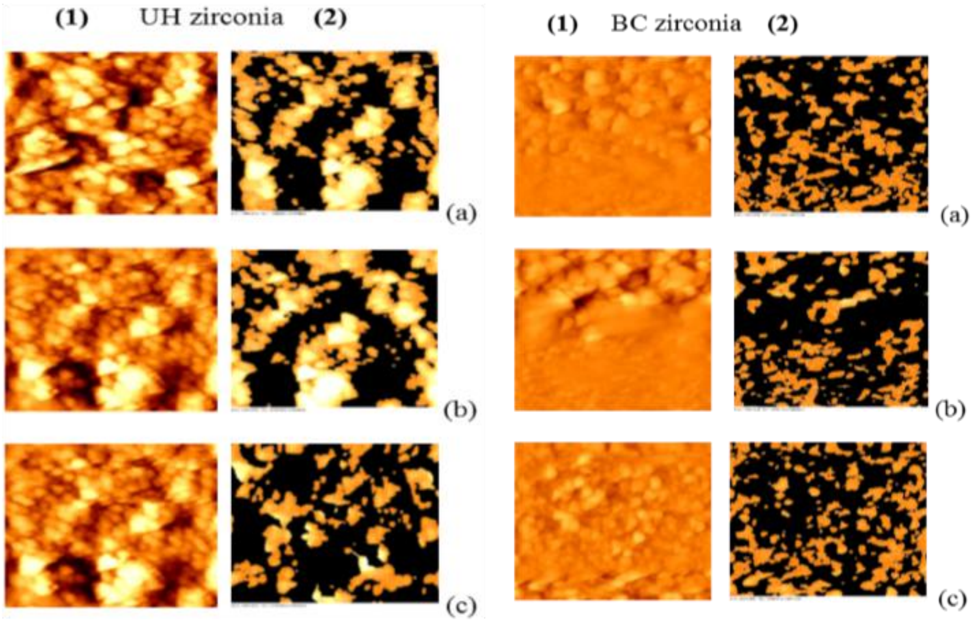

Figure (6): SPM Image for the intaglio surface of UH, and BCzirconia showed the geometry, shape, and size of its particles (a - control, b- conditioned with HP, & c- conditioned with CA. (1-positive image, 2- negative image) ((UH): UPCERA; (BC): BruxZir colored Zirconia materials); HP:HydrogenPeroxide,CA: Citric acid).

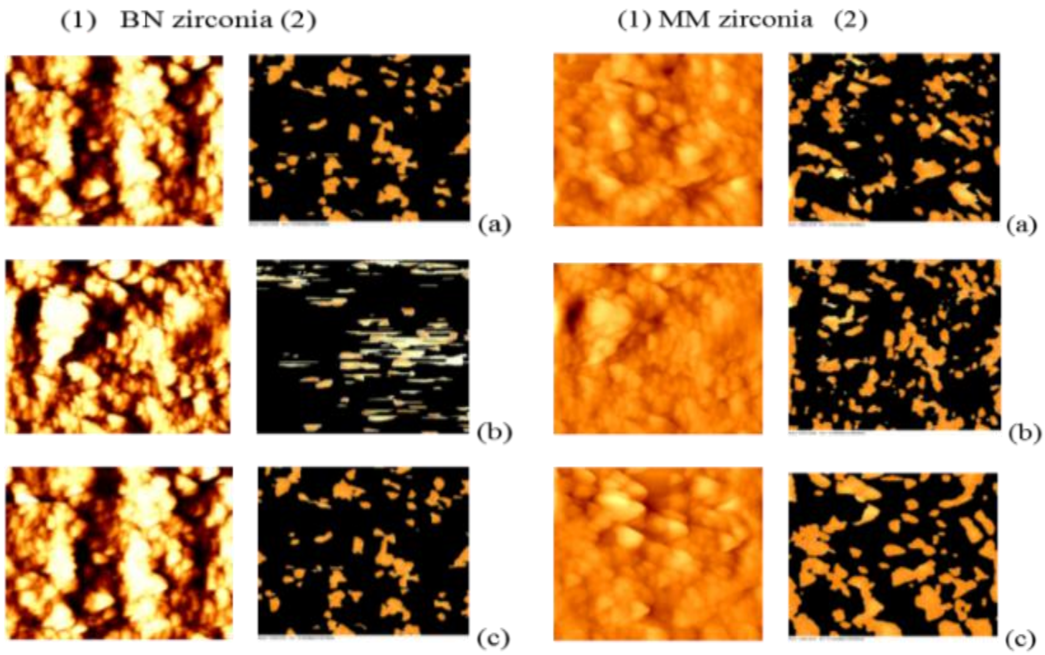

Figure (7):SPM Image for the intaglio surface of BN and MM zirconiashowed the geometry, shape, and size of its particles (a - control, b- conditioned with HP, & c- conditioned with CA. (1-positive image, 2- negative image)). (1-positive image, 2- negative image)) (BN): BruxZir non colored, (MM) Multilayer Zirconia materials); HP:Hydrogen Peroxide

DISCUSSION

Zirconia considered not only as a promising material, but also as one that is difficult to bond to[16.17].Unfortunately, the composition and physical properties of certain materials like zirconia differ from conventional silica-based materials, and require very aggressive mechanical abrasion methods to increase surface roughness, possibly creating strength-reducing surface flaws[18]. As it is essentially inert and nonpolar, chemical conditioning techniques commonly used in ceramics that have silica in their composition are ineffective on zirconia surfaces, unless they are pretreated[17].Therefore, in order to achieve acceptable cementation in a wide range of clinical applications, alternate attachment methods, ideally utilizing chemical adhesion in addition to mechanical retention, are required[19].Very often, mechanical tests, like micro tensile bond strength and shear bond strength are used to evaluate the bond quality between resin luting agent and zirconia substrate. But it seems that these are not sufficient to show the behavior of the material under function, limiting the treatment effects and for the credibility of study results, it is proposed to combine mechanical tests with a topographical analysis of intaglio surfaces prior to clinical recommendation of a new solutions for zirconia conditioning[20].

For the SBS (conventional shear)testhas been widely used, mainly because of their relative simplicity, predictability, and there is no need for further preparation of the specimens prior to test when compared to tensile bond strength tests, in which it is difficult to prepare and align the specimen in the testing machine without creating deleterious stress distribution[21].A new test method using specimens with reduced dimensions has been advocated by certain researchers as a substitute for the conventional shear test: so-called micro-bond or**M** -SBS test[7,10].. According to them, this test allows for testing of small areas, thus permitting a regional mapping or depth profiling of different substrates and preparing multiple specimens from the same sample plate[12].The method employed in this study showed a significantly improved in bonding strength between the chemically conditioned zirconia intaglio surface and the luting cement and this may be due to creating a micro-irregularities with in zirconia intaglio surface as a result of a chemically conditioned of that surface either by a HP or CA as an oxidizing or chelating agent respectively, used prior to applied the dual cure RX luting cement in compered to control group and this result confirmed with the AFM test analysis in this study, and this finding agreed with Kamal et al.(2018)whom stated that the surface roughness and micro-irregularities formed on the surface of zirconia after surface conditioned were considered one of the main prerequisites to achieve an improved in bonding strength with the luting cement [22].

In this study the BC zirconia conditioned with a CA and bonded with the dual cure luting cement RX showed a significant difference in its M-SBS value in comparing to the other tested zirconia materials and this may be due to effect of the coloring and dipping liquids on the zirconia surface and infiltration of the metal oxides of that coloring system during dipping of the pre-sintered milled zirconia specimens and this may be related to a balance between metal oxides and yttria in Y-TZP which protects the tetragonal structure, and coloring by the manufacturer can affect this structure and the infiltration of the metal oxides during dipping may promote or suppress this balance and may create a shift in tetragonal/monoclinic phase transformation and this shift may alter the surface microstructure, moreover the zirconia surface structure may be influenced by the dipping time and the concentration of color liquids, these factors may potentially affect the infiltration of oxides into the zirconia surface[23]. Some investigators founded that prolonging the dipping time lowered the zirconia biaxial flexural strength was attributed to an increase in open porosity.

The effect of concentration of color liquids on zirconia surface and strength was studied, and the solutions with higher concentrations had a greater negative effect on the zirconia strength[23,24].Consequently, in coloring by dipping, the interaction effect of all the above-mentioned factors may influence the surface composition and surface microstructure of zirconia, which can directly influence the bonding mechanism of zirconia to monomer-based phosphate cements. Minoo et al. (2015) take advantage of the liquid color with optimal performance to improve the zirconia surface bond[25].At the same time the use of CA; as a chelating agent; that enhance the solubility of many metal salts. Accordingly, the addition of certain chelating agents to systems in which the metal salt is dispersed in the liquid solvent will produce a clear homogeneous solution, this provides a method for producing substantially un-agglomerated metal oxide particles having a small diameter, uniform particle size distribution, and increase of the surface area [26,27].So, a large surface area, a more the effect of the coloring and dipping liquid on zirconia surface [23,24]and this may be e explain the increased in the M-SBS value of the BC zirconia conditioned with a CA and bonded with the RX dual cure resin cement in comparing to the other tested zirconia materials.

For the Failure modes, in this study the result showed a no evidence of a luting cement remnants left on zirconia materials surface (adhesive failure), and no evidence of fracture within the luting cement surface (cohesive failure)[15,28].The AFM test provides a true three-dimensional surface profile, and samples viewed by AFM do not require any special treatments, like metal/carbon coatings, that would irreversibly change or damage the sample [29,30].To investigate the surface morphology and roughness of zirconia after different surface conditioning, the Rqof the intaglio surface of the specimens was evaluated using a surface texture measuring instrument. The amplitude parameters are the principal parameters in characterizing the surface topography and the most common parameters are the average roughness (Ra) and the root mean square roughness (Rq) are the most used amplitude parameters.

Particularly, the last one is used to study temporal changes in the creation of a new surface as well as spatial differences when studying the surface feature using different scales. The reason for that, this parameter is more sensitive to large deviations with respect to the mean line[31].Surface morphology of a specimen from each group was evaluated with AFM. For the control, HP and CA groups, the MM zirconia showed a significant difference in its surface (Rq) value in compare to the three other tested materials, and this finding may be due to a larger particle size of the MM as showed in (Figure 7) in compared to the other tested material and this may related toa direct relation between the surface roughness and grain size of the material and whenever the grain size increases the surface roughness increases, also a large particles promoted a more punctual damage in a surface than small particles one[32].For the HP group both BC and BN zirconia showed a significantly rough surface in compare to the control and CA groups and this may be due to a more prominent solvate effect of the HP on a small particle with a large surface area in compare to other tested materials that contain a larger particle as showed in (Figures 6 and 7)[33]. At the same time the oxidation process of bleaching agent can facilitate water absorption and lead to loss of particles and reducing superficial integrity of the surface[34].

For the CA group, UH and MM zirconia showed a significantly rough surface in compared to the control and HP groups and this may be due to their surface degradation that resulted from the titratable acidity that seemed to play a more crucial role on surface degradation of materials than pH[35].Moreover, zirconia materials with large particles might be more susceptible to degradation when submitted to acidic challenges. In chemistry, Titratable acid generally refers to any acid that can lose proton(s) in an acid-base reaction[36].

CONCLUSIONS

Within the limitation of this invitro study, a chemical conditioning of the intaglio surface of zirconia with HP or CA may become a novel method for enhancing the micro-mechanical retention and bonding of the luting cementto Y-TZP.

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