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Influence of Nanosilica Coating on the Color and the Microleakage of Zirconia Ceramics

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***Abstract*---**Objective The purpose of this study was to assess the effect of Nano-silica Coating on the Color and the Microleakage of Zirconia Ceramics. Materials and methods: Eighty-five specimens of commercially available Yttrium-stabilized-zirconia ceramics were constructed and divided into forty-five discs (8 mm in diameter) with thickness (1.2mm) They were divided into three groups (thermocycling – nano-silica surface treatment – and thermocycling with nano-silica surface treatment) to assess the color parameters, while the other forty samples were divided into (control group – thermocycling – nano-silica surface treatment – and thermocycling with nano-silica surface treatment) to perform a fully contoured crowns to assess the microleakage. Initial color measurements were taken using a digital spectrophotometer based on CIE L*a*b* color system using a spectrophotometer Standard permanent molar tooth preparation was conducted into a full veneer preparation. Nano silica sol-gel material painted on the fitting surface of zirconia ceramics in groups of nano-silica and nano-silica with thermocycling surface treatment and processed to firing that were finally cemented on the prepared teeth using resin adhesive luting agent. Samples in aged group were subjected to 5000 thermal cycles then crown samples were sectioned to measure the microleakage. Color measurements of all samples were measured again.

***Keywords*---**microleakage, nanosilica coating, restorative materials, teeth, zirconia ceramics.

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Introduction

Zirconia-based restorative materials have been used as core materials for single crowns and fixed dental prostheses due to their superior esthetics, biocompatibility, and high mechanical strength. Its mechanical properties are similar to metals and, at the same time, its color is similar to that of natural teeth. The excellent strength, high fracture toughness, excellent abrasion resistance, toughness, fatigue resistance, and aesthetic properties of zirconia are the reason for it being labeled as “ceramic steel”. These properties are considered to be the ideal set of characteristics required of a material used in dental applications [1]. Apart from fracture resistance and esthetics, marginal and internal fitting accuracy are some of the most important parameters for the clinical quality and success of ceramic crowns. The increased marginal discrepancy of a crown favors an increased rate of cement dissolution and microleakage. Microleakage from the oral cavity may cause inflammation of the vital pulp and pulp necrosis and potentially presents the need for endodontic treatment. Poor marginal adaptation of crowns increases plaque retention and changes the composition of the subgingival microflora, contributing to the onset of periodontal disease. Microleakage may be defined as the passage of bacteria, fluids, molecules or ions between the tooth structure and the restorative material. Clinical complications of microleakage include postoperative sensitivity, marginal discoloration, and recurrent caries. The microleakage of crowns is considered to be one of the main causes of failure, that is, one of the factors that most influence the clinical longevity of indirect restorations [2]. Unlike glass ceramics, etching is not possible for yttria-stabilized tetragonal zirconia due to the high crystalline content and glass free structure. Several surface treatment methods have been tried, including roughening the surface with diamond rotary systems, airborne-particle-abrasion, surface fluorination, Nano alumina coating, hydrofluoric acid etching after fused glass-ceramic application, hot chemical etching, selective infiltration etching, laser irradiation, and modifying the surface through a silica coating. Of all these methods, airborne-particle-abrasion is to create micro retentions [1]. Many methods for silica coating have been developed, which include the tribochemically silica-coated technique, as well as the tentative plasma spray technique and the vapor-phase deposition technique. Among these, the tribochemical silica-coating technique has reportedly improved bonding performance and offers more convenient handling than any other technique; therefore, it is the most commonly used method for the silica coating of high-strength ceramics [3]. The tribochemically silica-coated technique air-abrades the bonding surface with special alumina particles that have been coated with nanosilica, embedding/coating the surface with Nano silica. This process not only provides a silica-contained surface for silanization, but it also offers micromechanical retention. However, the drawback with this technique is that sandblasting with special Nano silica-coated alumina particles results in stress on the Yttria-stabilized tetragonal zirconia surface. This has resulted in an increasingly low temperature degradation of Yttria-stabilized tetragonal zirconia, which is caused by stress-induced tetragonal-to-monoclonic phase transformations [3]. Therefore, a nondestructive, alternative technique for silica coating is required. A solution– gelatin (sol–gel) process for making high-purity Nano silica has been well developed and dense silica components without resulting in any stress-induced processes. In this study, we evaluated the effects of silica coatings prepared with our experimental sol–gel technique on the color and fracture resistance of Yttria-stabilized tetragonal zirconia will be evaluated [3]. The null hypothesis is that nano silica will not affect the color and the microleakage of zirconia ceramics.

Eighty-five specimens of commercially available Yttrium-stabilized-zirconia ceramics (Cercon XT Dentsply Sirona Prosthetics) were equally divided into two groups according to the to the fabrication structure into crowns and discs (40 for crowns and 45 for discs). The crown group was further subdivided into four subgroups (10 for each) according to the surface treatment with nano-silica sol-gel and thermocycling aging (A: aging, S: silica, C: control and SA: silica-aging). While the disc group was further subdivided into three subgroups (15 for each) according to the surface treatment with nano-silica sol-gel and thermocycling aging (A: aging, S: silica and SA: silica-aging).

Sol-gel preparation

The sol-gel solvent mixture was prepared at room temperature using tetraethyl orthosilicate (TEOS; 98%, Sigma-Aldrich, USA), absolute ethanol (99.8%, Riedel-deHaën, Seelze, Germany), deionized water, and ammonia catalyst (28–30%, Sigma-Aldrich, USA). Tetraethyl orthosilicate was added to a mixture of ethanol and deionized water, and then aqueous ammonia solution was added. Zirconia samples were immersed in the solvent mixture. The solution was slowly stirred. After the sol-gel deposition process, silica-coated zirconia samples were allowed to dry at room temperature, followed by rinsing with 70% ethanol and drying at room temperature.

Tooth preparation for receiving samples

The teeth preparations were made with a 1 mm shoulder margin positioned just occlusal to the cemento-enamel junction with axial wall convergence of 6± 2 degree for every wall using flat-end taper diamond bur (#847, Brassler, USA) by high-speed air turbine handpiece with profuse water coolant. Teeth was mounted in a parallelometer to standardize the taper of the preparation. Bur was changed every preparation to ensure good cutting efficiency. The occlusal surface was prepared to create a non-anatomical occlusal table and maintain a vertical height of 4±0.25 mm using wheel diamond bur (#2909, Komet, USA) in a high-speed air turbine handpiece with copious water coolant.

Fabrication of zirconia crowns by CAD/CAM

A CAD/CAM system (Cerec InLab SW 4.0, Sirona, Bensheim, Germany) was depended upon for the fabrication of samples of cercon xt zirconia discs.

Fabrication of the zirconia disc samples

Unsintered zirconia was cut into disks with 12.4 mm diameter and 2 mm height using IsoMet 30HC precision cutting blade2 (Diameter 127 mm, thickness 0.76 mm) mounted on micricut 150/201 precision cutter.3 The machine was adjusted using its built-in micrometer to cut the cercon xt block under water coolant into slices of approximately 1.5 mm thickness, 20% larger than the desired final size to compensate for sintering shrinkage caused during the sintering stage. The thickness of each slice was checked with a digital caliper 4 and minor corrections were done using Wetordry5 Sandpaper sheet of grit 400, 600, and 1200 in the presence of water to ensure flat surface and desired thicknesses.

Nanosilica coating of Y-TZP crowns

To provide a clean surface before coating, the selected samples that designed to be silica coated were vigorously washed under running distilled water, placed in an acetone/ethanol ultrasonic bath for 20 minutes, and then air-dried. The Y-TZP blocks were coated by the silica by dipping in the solution for 5 days and then calcinated at sintering furnace for 2 hours at 400°C (5°C/min)

Cementation procedure

hydrofluoric acid 9.0% (HF) etching gel, porcelain etchant Bisco ,USA. for 90 seconds. The etched surfaces were then thoroughly missed using water spray for 60 seconds, followed by ultrasonic cleaning in distilled water for another 60 seconds, then dried by oil-free compressed air for 30 seconds. Silane coupling agent (Bisco, schaumburg, IL 60193 USA)was applied to the etched intaglio surfaces of each crown and allowed to dry for 60 seconds. Samples were cemented with RelyX™ Unicem Self-Adhesive Universal Resin Cement shade A2(3M ESPE Dental products, St. Paul, USA).

Aging of samples

Samples were then transferred to the plastic chamber of a chewing simulator (chewing simulator CS-4, SD Mechatronik Co., Germany) and secured in their place using putty rubber base impression material (Provil novo putty, Heraeus Kulzer, Germany) and custom-made acrylic ring (Ivolen, Ivoclar Vivadent, Liechtenstein, Germany). samples were then subjected to compressive cyclic loading for 500,000 cycles at speed 2 Hz in a water bath of 37 C°. The chewing simulator stainless steel antagonist cone, with 30° total taper and its end machined to a curvature equivalent to 3 mm diameter, was adjusted to contact the central fossa of the crowns and preload the crowns with 80 N force by 0.2 mm vertical displacement. It was adjusted to move in two directions: vertical movement of 1 mm and horizontal movement 1mm, to induce both vertical and horizontal forces during the simulated chewing cycles.

Microleakage testing procedures

After thermocycling, crowns were subjected to microleakage testing by immersion in 0.5% basic fuchsin solution dye (Pararosanilin, Imperial Chemical Industries) for 24 hours. Then the samples were removed from the solution and rinsed thoroughly in water for five minutes and embedded in a clear self-cure acrylic resin then sectioned bucco-lingually using a low-speed saw with a diamond blade of 1mm thickness (Isomet 1000, Buehler Ltd., Lake Bluff, IL)(Figure 70). The sections were equal in thickness. Images of tooth sections were examined under stereomicroscope at 10x magnification, and a five-point scale was used to score microleakage at both buccal and lingual aspects of each section

Color measuring test procedures

The primary step of color change test was obtained using the spectrophotometer (VITA Easyshade®Advance 4.0, vita zahnfabrik) . All zirconia disc samples that acquired aging, silica treatment and aging and silica together were collected and measured over blue and a gray background before submitting to aging and silica treatment and aging and silica together. Each sample was measured twice and obtained the average reading. The intimate contact between the device and samples assured no external optical contact influence.

This test was done using reflectance spectrophotometer. This device is wireless comprising a dental shade matching device which consists of a base unite and hand piece. The spectral power distribution of the light source in the device was set to CIE illuminant D65 which corresponds to the average daylight, and the average wavelength range was set from 360-700µ with 10µ intervals. The values expressed in CIE L* a* b* parameters represented as L* was the lightness, where 100 was white and 0 denotes black. a* and b* were the red-green and yellow-blue chromatic coordinates respectively. A positive a* value represent red while a* negative value denotes green. A positive b* value represents yellow while negative b* value denotes blue. Then the difference in color (ΔE) between both groups was calculated using the represented CIELAB coordinates equation
$\Delta E = [(\Delta L^*)2 + (\Delta a^*)2 + (\Delta b^*)2]$ ½.

Results

Means & standard deviations of the effect of silica-coating on zirconia by sol-gel process on the color change of zirconia was evaluated and shown in Table (1) and figure (1). In the present study, the statistically significant difference in color change result between the groups was verified. Thermocycling treated group showed the lowest color change record of 1.26 ± 0.29 that followed by nano-silica treated with mean of 2.13 ± 0.45. The mean of the thermocycling with nano-silica group recorded with 3.27 ± 0.72The difference between groups was statistically significant ($p<0.05$).

In this study, Means & standard deviations of the effect of silica-coating on zirconia by sol-gel process on the microleakage of zirconia was evaluated and shown in Table (2) and figure (2). In the present study, the statistically significant difference in microleakage result between the groups was verified. Nano-silica treated group showed the lowest microleakage record of 908.5 ± 83.88µm that followed by silica aging with mean of 908.5 ± 83.88µm. The mean of the control group with no surface treatment recorded with 1300.4 ± 79.37µm that followed by thermocycling treated group with the largest microleakage with mean of 2011.1 ± 111.7µm. The difference between groups was statistically significant (p value >0.05).

Table 1
Comparison between the three studied groups according to ΔE

ΔE	Aging (n = 15)	SILICA (n = 15)	Silica -aging (n = 15)	F	p

Min. – Max.	0.86 – 1.74	1.04 – 2.79	2.01 – 4.66		
Mean ± SD.	1.26 ± 0.29	2.13 ± 0.45	3.27 ± 0.72	56.609*	<0.001*
Median (IQR)	1.30 (1.02 – 1.47)	2.22 (1.92 – 2.35)	3.23 (2.89 – 3.70)		
Sig. bet. grps.	$p_1<0.001^*$,$p_2<0.001^*$,$p_3<0.001^*$				

IQR: Inter quartile range SD: Standard deviation
F: F for One way ANOVA test, Pairwise comparison bet. each 2 groups was done using Post Hoc Test (Tukey)

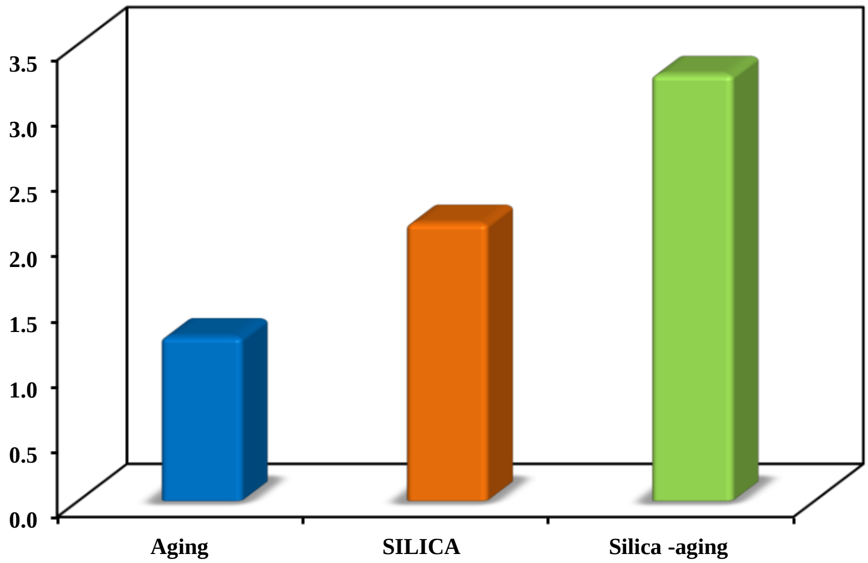

Figure 1. Comparison between the three studied groups according to ΔE

Table 2
Comparison between the different studied groups according to micro leakage

Micro leakage	Control (n = 10)	Aging (n = 10)	Silica (n = 10)	Silica -aging (n = 10)	F	p
Min. – Max.	1186.5 – 1386.4µm	1798.3 – 2181.9µm	183.8 – 325.4µm	767.8 – 1047.1µm	770.985*	<0.001*
Mean ± SD.	1300.4 ± 79.37µm	2011.1 ± 111.7µm	262.7 ± 44.38µm	908.5 ± 83.88µm		
Median (IQR)	1315.8 (1198.3 – 1366.9)	1985.0 (1965.5 – 2131.4)	269.5 (222.6 – 295.1)	928.4 (885.4 – 935.0)		
p_0		<0.001*	<0.001*	<0.001*		
Sig. bet. grps.	$p_1<0.001^*$,$p_2<0.001^*$,$p_3<0.001^*$					

IQR: Inter quartile range SD: Standard deviation
F: F for One way ANOVA test, Pairwise comparison bet. each 2 groups was done using Post Hoc Test (Tukey)

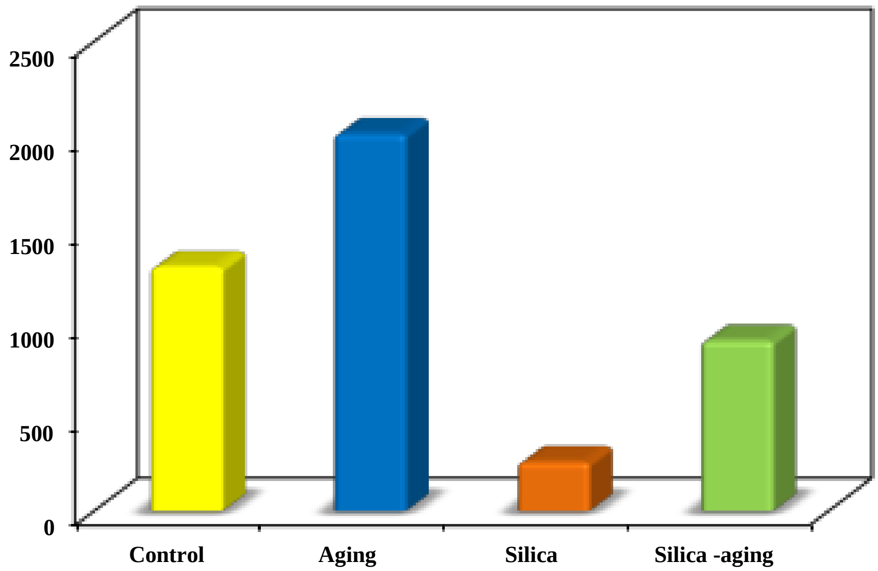

Figure 2. Comparison between the different studied groups according to micro leakage

Discussion

The sol–gel process is one of the most useful and flexible methods for oxide film fabrication due to the low homogeneity of coatings, easy control of coating thickness, processing temperature and the potential to produce coatings on solids with complex shapes. In this study, acid-catalyzed alkoxide–hydrolysis was used to prepare silica sol with nanosized particles [4]. In line with previous findings, it was clear that the increased hydroxyl groups resulting from silica and the subsequent silanization contributes to the improved bonding of the sol–gel silica-coated surface [5&6] while the micro-interlock does not seem to be as important [4]. As the solvent evaporated, the silica sol transformed to a gel on the Y-TZP surface. According to previous studies, the nano-silica gel coating sticks to ceramic surfaces by van der Waals electrostatic interaction forces, and the Si–O–Si network is strengthened by baking [4]. Finally, Si-O-Si bonds in silica are strengthened by heat treatment and a solid layer bind to the surface. Then, hydroxyl groups (a chemical group with high polarity) in silica coated samples increase the surface polarity and reactivity of chemical bonds with other chemicals [7]. This reaction result in formation of a new layer on the zirconia surface as confirmed by SEM, EDS, FTIR and XRD analysis in the previous study [8]. T. M. B. Campos et al reported in their study that the Weibull analysis showed that the homogeneity of the sol-gel silica to zirconia was superior, possibly due to healing of surface flaws. The increase in the wettability of zirconia changes this material into a good substrate for bonding resinous materials, that can be treated with HF and silanes. Moreover, the sol-gel infiltration presents the advantage of being a user-friendly method that does not invade on the ceramic surface, as does air abrasion [9]. They also assumed that silica infiltration was able to improve not only the immediate bond strength to resin cement, but also the long-term bond strength after 6x103 thermal cycles. As silica infiltration formed an intermediate crystalline phase and not only a juxtaposed silica layer [9]. Regarding microleakage studies, in vitro initial screening methods were designed to assess the sealing ability of restorative materials while in vivo. microleakage analysis investigates the sealing ability of adhesive interfaces. Clinically, if the adhesive interface or the adhesion process is compromised this results in microleakage, which could be a chance for further adhesive failure [10]. Dye penetration was selected as it still a popular leakage test, and India ink has been used in other studies [11]. It has been stated that penetration into marginal gaps is dependent on molecular size (dye, isotope vs toxin, microorganisms), surface interaction between the dye and restoration, molecular polarity and time [12]. In this study, crowns and discs were subjected to chewing forces at a rate of 2 Hz, in a water bath of 37 C°, with load of 80 N and 1 mm vertical and horizontal movement of the blunt indenter, for 5000 cycles which is considered to correspond to 10 years of normal function in the oral cavity. it was reported that the normal chewing cycles frequency can range from 1-2 Hz. 224 The selected load to be applied was 80 N that lies within the range of the normal masticatory forces which was expected to be in the range of 50 to 250 N and could reach 500 to 800 N in parafunctional conditions similar to clenching and bruxism [10]. In this study, microleakage was investigated using a dye leakage technique with basic fuchsin. The penetration of basic fuchsin and the India ink provided a similar effect for the sealing ability of the materials tested. On the other hand, in-vitro microleakage investigations carried out with dyes are considered to be stricter than those assessed in the oral cavity. Consequently, if a material performs well in the dye test, it is likely to perform even better on a clinical level [12]. In the current study, two of the most commonly used artificial aging techniques were performed (storage in distilled water for four weeks and thermocycling). Wahab, et al [13] described that thermocycling increased the amount of microleakage. After thermocycling, microleakage may be observed between the tooth and the restorative material [12]. Various laboratory devices, such as light microscopes, stereomicroscopes, digital microscopes and SEMs ranging from 80x to 225x magnification, were used in previous studies in order to observe the accuracy of the fitting surface of crowns. A computerized stereomicroscope was selected in the current study as the measurements were attached by captured digital images. In this study, the effect of silica-coating on zirconia by sol-gel process on the color change of zirconia was evaluated. Regarding color stability test, Previous studies suggested that color value of $\Delta E<1.3$ is imperceptible to the human eye, while a value of $\Delta E>3.7$ is considered as clinically undesirable [14]. However, the findings in our study revealed that the highest ΔE values among the three groups were noted in silica-aging group with values of $\Delta E=3.27 \pm 0.72$ the color change still below the clinically perceptible threshold. Hamza et al [15] studied the impact of accelerated aging on the color stability of various ceramic systems, they used 2-mm-thick translucent monolithic zirconia samples. According to the CIE Lab system, they detected a statistically insignificant color change ($\Delta E^* = 0.8743 \pm 0.32837$) in translucent monolithic zirconia. In the present study, similarly to the study of Hamza et al [15] while the maximum amount of color change was observed in silica-aging samples ($\Delta E = 3.27 \pm 0.72$), the minimum amount of color change was observed in thermocycling aging samples ($\Delta E = 1.26 \pm 0.29$). At all groups, a color change below the clinically perceptible threshold was observed. Color change of thermocycled tested samples can be explained as increase in the L^*, a^*, and b^* values indicated increase in the brightness, redness, and yellowness

of the sample after cyclic aging. Correspondingly, many previous studies stated a change in the color of cemented dental ceramics as a consequence of aging [15][16][17]. In our study, significant color change of nano-silica treated samples observed to be more than thermocycled samples (ΔE = 2.13 ± 0.45) but still within the acceptable range. This may be explained as Si elements and trace Zr elements on this surface, which suggests that the Y-TZP surface was totally replaced by a new smooth silica-coating surface. This was predominately detected by Chen, et al [4] which traced by EDS mapping. In the present study, statistically significant of color change of nano-silica aged group may be contributed to both thermocycling and nano-silica surface treatment that still within the acceptable range. In general, color change can vary with aging conditions and can be attributed to an alteration in the color of the ceramic itself and the underlying cement. Ceramic material discoloration can be due to a loss of surface stain, increased surface roughness, surface cracks, change in ceramic translucency, and reduction of ceramic thickness. On the other hand, resin cement discoloration can be manifested as a result of the degradation of unreacted polymers in the polymerization process. Therefore, the polymerization mode, time, cement shade, and composition all affect the cement color stability [19]. Chaiyabut et al. [20] reported that if a ceramic is less than 2 mm-thick, its optical color will be influenced by the underlying substrate color. In the current study, the high-translucency nature of the employed ceramic and the reduced thickness of 1 mm may be allowed for visualization of the color change of the underlying cement. The overall mean value for color change found in this study was 3.59 ± 1.6. The color difference is proposed to be perceptible when it can be detected by the human eye, and acceptable when it is tolerable. Considering a threshold of perceptibility of ΔE = 1 and a threshold of acceptability of ΔE = 3.7 as concluded by Khashayar et al.
[21] that the color change value in the current study is considered perceptible but clinically acceptable for all samples. In this study, the effect of silica-coating on zirconia by sol-gel process on the microleakage of zirconia was evaluated. In the present study, the statistically significant difference in microleakage result between the groups was verified. Silica treated group showed the lowest microleakage record of 908.5 ± 83.88 that followed by silica aging with mean of 908.5 ± 83.88. The mean of the control group with no surface treatment recorded with 1300.4 ± 79.37 that followed by thermocycling treated group with the largest microleakage with mean of 2011.1 ± 111.7. Microleakage testing investigates the seal ability of adhesive interfaces. Clinically, if the adhesive interface or the adhesion process is compromised, this results in microleakage, which in turn could result in further adhesive failure [10]. The result can be explained according to LUNG KCY et al [22] finding regarding the effect of silica-coating by sol-gel process on resin-zirconia bonding. They proved that the interfacial failure occurred between the resin cement and silica-coated layer on zirconia for thermocycle group showed even debonding. Therefore, it was apparent that resin-zirconia bonding was adversely affected by thermal fatigue and hydrolytic degradation during thermocycling. For the sol-gel process, Silica particles chemically react with the zirconia surface, through the interaction of hydroxyl groups of silica particles with the surface hydroxyl groups of zirconia, to form a silica layer on the zirconia surface. Besides, hydrolyzed tetraethyl orthosilicate may also condense on the zirconia surface first and then build up to form a silica layer on it [21]. This contributed to increase in the wettability of zirconia turns this material into a good substrate for bonding resinous materials, treatable with

HF and silane. The silane coupling agent formed silanols after hydrolysis, which then reacted with the surface hydroxyl groups of the silica layer on zirconia surface to form siloxane linkages (-O-Si-O-) after condensation. Finally, resin cement was bonded to the silica-coated and silanized zirconia surface by light curing. During polymerization, the >C=C< bonds in the composite resin reacted with the >C=C< bonds in the silane coupling agent to form –C–C– single bonds [22]. On the line with previous studies, the effect of bonding protocol also may show effect on microleakage, [23][24] these studies showed that the strong etching and resin infiltration of the etch-and-rinse adhesive based resin cement with subsequent hybrid layer formation, which can result in stronger micromechanical bonding to tooth structure and low microleakage scores. On the other hand, the weaker adhesion of the self-adhesive resin as a consequence of their weak demineralization of tooth structure and tangible resin infiltration with no hybrid layer formation could be the reason of high microleakage scores [9]. T. M. B. Campos et al reported in their study that silica infiltration was able to improve not only the immediate bond strength to resin cement, but also the long-term bond strength after 6x103 thermal cycles. As silica infiltration formed an intermediate crystalline phase and not only a juxtaposed silica layer. This may explain the results of silica aging is better than controlled and aged samples [9] Regarding thermocycling , the least mean value for aged samples may be contributed to the limited durability of MDP primers as it explained by T. M. B. Campos et al [9] results that showed the lower bond strengths between infiltrated zirconia and cement after thermocycling.

Conclusion

Within the limitation of this in vitro study, it is following conclusion can be drawn that:

- The sol–gel process is one of the most useful and flexible methods for surface treatment of zirconia ceramics.
- Artificial aging significantly increases the microleakage of zirconia ceramics through thermo-mechanical loading
- Sol-gel processed nano-silica coating can improve the resin bond compatibility of zirconia ceramic that clue to decrease the microleakage at the margins of the crown
- Sol-gel processed nano-silica surface treatment showed color change of the zirconia ceramics which was below the clinically detectable color change limit.

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