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**Influence of aging on biaxial flexural strength, hardness and resin
bond strength of the translucent zirconia**

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List of abbreviations

Y-TZP	Yttria-stabilized tetragonal zirconia polycrystals
wt. %	Weight percentage
mol%	Mole percentage
t→m	Tetragonal change to monoclinic phase
Al₂O₃	Aluminum oxide
LTD	Low temperature degradation
ZrO₂	Zirconium dioxide
R_a	Surface roughness parameter (the average roughness of a surface)
R_z	Surface roughness parameter (the arithmetic mean value)
3Y-TZP	3 mol% Yttria-stabilized tetragonal zirconia polycrystals
s	Second
min	Minute
XRD	X-ray diffraction
°	Degree
BFS	Biaxial flexural strength
TBS	Tensile bond strength
Y₂O₃	Yttrium oxide
vol. %	Monoclinic phase ratio

1. Introduction

1.1. Zirconia restorations

All-ceramic dental materials became the first choice restorative materials for many dentists because of their superior biocompatibility and esthetic appearance. However, brittle behavior with their extreme sensitivity to microcrack-like defects limits their use to relatively low stress-bearing areas.

Nowadays, 3Y-TZP ceramics (3 mol% yttria-stabilized tetragonal zirconia polycrystals) are the strongest alternative biomedical dental materials for all-ceramic dental restorations because of their esthetic, durability, and availability [2, 4, 41]. In addition, they are more biocompatible than other ceramics, titanium, and metal alloys, particularly when it is in direct contact to the gingival tissues [93]. Zirconia has three crystalline phases: monoclinic, tetragonal and cubic [93]. The monoclinic phase is stable from room temperature to 1170 °C, then it transforms to a tetragonal phase 1170 °C to 2370 °C. The structure becomes cubic above 2370 °C and up to melting point. A transformation of tetragonal to monoclinic occurs while cooling down to the temperature of 1170 °C, this is associated with a volume expansion of 3% to 5% [28, 93]. Stabilizing oxides such as calcium oxide, magnesium oxide, cerium oxide and yttrium oxide were added to prevent this transformation upon cooling. Yttrium is added to stabilize zirconia at room temperature in the tetragonal phase [72], accordingly this transformation is responsible for its high mechanical properties [5].

Monolithic zirconia ceramics used as full-contour ceramic restorations, overcome the problems of veneering porcelain chipping [16, 17, 81], and have the advantages of restorations requiring less invasive tooth preparations and providing high mechanical and esthetic properties [35]. The translucency of zirconia has a strong relationship with its microstructure and chemical composition [21]. Previous studies showed that the translucency of zirconia influenced by grain size of Y-TZP ceramics, grain boundary structures and phase composition [6, 54, 78]. Different dental manufacturers eliminated or reduced the alumina amount or increased the yttria content to improve the translucency of dental Y-TZP ceramics [126]. Increasing the content of yttria in the new-generations zirconia influences negatively the mechanical properties of zirconia [70]. The amount of yttrium oxide stabilizer was focused in an effort to fully stabilize the material and enhance its optical properties to optimize the characteristic of full-contour monolithic restorations [108].

The first generation of translucent zirconia was based on conventional Y-TZP ceramic, containing 3 mol% Y_2O_3 which reported high opacity, high resistance, toughness and enhanced

mechanical properties. The second generation also consisted of 3Y-TZP with a reduction in the grain size; this small change in the materials composition enhanced light transmission and optical/esthetic properties, maintaining their characteristics (toughening mechanism) [106]. The third and fourth generation had the important change of increasing the percentage of Y₂O₃ stabilizer (4 mol% or 5 mol%), which resulted in significant amount of cubic zirconia phase [57]. The optical/esthetic properties were improved, although it was expected that the mechanical properties would be somewhat compromised [126] (Table 1). Recently some literature [126] reported that the co-doping of 0.2 mol% lanthanum oxide and 0.1- 0.25 wt.% Al₂O₃ in 3Y-TPZ zirconia resulted in a high translucent zirconia with improved mechanical properties [126, 127].

Table 1. Generations of translucent zirconia

(3Y-TZP)	(5Y-TZP)	(4Y-TZP)
(1st and 2nd generation)	(3rd generation)	
Y ₂ O ₃ (4.5 - 5.6 wt.%)	Y ₂ O ₃ (< 10 wt.%)	Y ₂ O ₃ (< 8 wt.%)
Highest mechanical properties	Lower mechanical properties	High mechanical properties
Lowest translucency	Highest translucency	High translucency
Mainly tetragonal phase	More cubic, less tetragonal phase	Tetragonal and cubic phases

1.2. Aging and surface treatments of dental zirconia

Aging or LTD of zirconia has a great interest from the researchers in medical and dental fields, as it affects many properties of zirconia such as strength and hardness. The mechanical behavior of Y-TZP zirconia might be deteriorated by a spontaneous transformation of the tetragonal phase to monoclinic phase (t→m) when triggered by external stresses such as grinding [47, 69] particle air-abrasion [44, 48] and hydrothermal aging [66, 87] which is defined as a hydrothermal degradation or low temperature degradation [67].

LTD initially occurs at the superficial grains, where water is incorporated into zirconia grains by filling oxygen vacancies, and later spreads to the surface increasing its roughness [101, 125]. Afterwards, LTD proceeds into the bulk material [125] and affects negatively the density and mechanical properties of Y-TZP structures [15, 41, 50, 74]. LTD can be induced by steam autoclave treatments at increased temperatures (120-140 °C), whereby phase transformation of Y-TZP crystals might occur in the presence of water or steam [15, 20, 25].

Many studies reported that the real influence of aging of zirconia in an autoclave is not clear; it may positively or negatively influence the mechanical properties of Y-TZP ceramics. First the aging leads to an increase in the mechanical properties, through the transformation

toughening mechanism ^[5, 49], then it results in a mechanical deterioration of the material ^[22, 41, 67]. The improvement of mechanical properties of zirconia by LTD was revealed in several studies ^[44, 87, 104], while other studies ^[88, 89] showed that LTD had no effect on the mechanical properties of zirconia.

Additionally, when studying adhesion to zirconia as received from the dental laboratory, it is clinically relevant to evaluate adhesion to aged zirconia, because of the need to repair fractured restorations which had been exposed to the oral environment. To date, the influence of aging and surface treatment on high translucent zirconia have not been investigated in detail. A previous study showed that aging of a 3Y-TZP zirconia for 5 hours at 134 °C under a pressure of 2 bar resulted in a reduction of microtensile bond strength after 24 hours of storage, when the specimens of zirconia were bonded with the resin RelyX Unicem ^[86]. Whereby in this study the specimens did not undergo any thermocycling and also the long-term resin bond strength was not evaluated.

Surface treatment of dental materials is considered as an important procedure to increase the surface roughness and surface energy, resulting in an improved wettability for bonding procedures ^[75]. Zirconia undergoes several different treatments during fabrication procedures in the laboratory and sometimes during clinical adjustments such as particle air-abrasion with Al₂O₃, silica coating, acid etching or combinations of any of these methods.

It has been reported that surface treatments might influence the mechanical properties of dental zirconia ^[18, 19, 118]. Some studies showed that different surface treatments such as grinding, might influence the strength of zirconia ceramic negatively ^[69, 76, 119, 128]. The different surface damages, which were caused by the surface treatments, act as stress concentration areas which later on act as a potential site of crack initiation and propagation ^[1]. On the other hand, many studies reported an increase in the strength of zirconia with particle air-abrasion treatment due to the created compressive stresses induced by the tetragonal-monoclinic transformation on the surface of zirconia ^[47, 52, 69].

Some studies ^[3, 53] reported that tested polishing systems were not able to induce a tetragonal-monoclinic transformation. While another study ^[52] showed a significant t→m transformation which resulted in an increase in the biaxial flexural strength of Y-TZP with the polishing treatment. Many surface treatments are provided either replacing the classic method with new surface roughening procedures, or combining it with other methods such as laser treatment and surface modifications that induce chemical bonding between zirconia and resin ^[105, 110].

Alumina particle air-abrasion is an important procedure to increase the adhesion of luting resin to zirconia ceramic restorations [99, 116]. In addition, some studies showed that air-borne particle abrasion followed by an appropriate chemical bonding process resulted in long-term durable bonding to zirconia [65]. On the other side, some studies reported that the presence of MDP in luting resin create a stable and strong bond to particle air-abrasion abraded zirconia before and after thermocycling [65, 77]. These finding were confirmed by a recently published systematic review including laboratory tests and clinical outcome revealing a strong clinical evidence that particle air-abrasion at a moderate pressure with alumina particles in combination with a phosphate monomer containing primer and/or luting resin provides long-term durable bonding to zirconia ceramic under stressful oral conditions [63].

The following factors are reported to affect the susceptibility of zirconia to LTD:

1. Density [27]
2. Stabilizer type and content [26, 101]
3. Grain size [59, 71]
4. Processing characteristics (homogeneity, manufacturing , preparation) [27]
5. Presence of residual stress [36, 74]

1.3. Surface roughness

Many parameters are used to characterize the roughness of the materials. Surface roughness R_a and R_z are mostly used in the literature. R_a is a measure of the texture of a surface. It is defined as the arithmetical mean of the absolute values of peaks and valleys measured from a medium plane. While the R_z parameter is defined as the mean value of the single roughness depths of consecutive sampling lengths. Z is the sum of the height of the highest peaks and the lowest valley depth within a sampling length. R_z is a more sensitive parameter to occasional high peeks or deep valleys than the R_a roughness. Rough surfaces usually wear more quickly and have higher friction coefficients than smooth surfaces. Surface irregularities act as potential sites for crack initiation and propagation leading to a reduction in the strength of ceramic restoration [28, 30, 47]. Contrarily to promote the adhesion and subsequently the bond strength of resin to ceramic restorations, roughness is highly recommended to get a good adhesion [9]. Ceramic restorations are subjected to different fabrication procedures in the laboratory and sometimes must be clinically adjusted to get a proper fit or occlusion. These processes are more likely to initiate subcritical flaws which, with clinical loading and/or presence of moisture may

grow leading to catastrophic failure. Polishing of dental ceramics is also highly recommended to reduce the wear of opposing enamel [40, 82].

The following factors are reported to affect the surface roughness of zirconia:

1. Milling technique [115]
2. Surface treatment performed [34, 83]
3. Polishing procedure [61, 96, 113]

1.4. Biaxial flexural strength

Strength is one of the most investigated parameters to understand the clinical potential and limitations of a dental material [46]. Brittle materials such as ceramics are much weaker in tension than in compression, therefore the flexural strength is used to evaluate the strength of these materials. Furthermore, failure stresses of brittle materials are statistically distributed as a function of the flaw size distribution in the material [32].

The flexural strength of a material is its ability to bend before it breaks [7]. Flexural forces are the result of masticatory forces generated under clinical conditions where dental materials need to withstand repeated flexing, bending, and twisting. Materials with high flexural strength provide restorations with less susceptibility to bulk fracture.^[1]

The following factors are reported to affect the biaxial flexural strength of zirconia:

1. Type of zirconia [23, 103]
2. Surface treatment performed [60, 83, 87]
3. Polishing procedure [84]
4. Dipping time in coloring liquid [51]
5. Aging [87]

1.5. Surface hardness

Hardness is defined as the resistance to permanent surface indentation or penetration. It is measured as a force per unit area of indentation. Hardness is a very important property of dental materials due to its direct correlation to ease of cutting, finishing, polishing and resistance to scratch of the material. The Vickers hardness test is considered as one of most common methods of hardness testing especially of ceramics, utilizing a square-based diamond indenter which is forced into the material with a definite load application. [97]

1.6. Luting resins

In recent years, the demand of using all-ceramic restorations as an esthetic material increased and led to the development of ceramic materials with improved mechanical properties and good esthetics. These ceramics are used widely in clinical applications, such as posts, fixed dental prostheses, implant abutments and even adhesive resin-bonded fixed dental prostheses. The successful clinical outcome of this dental material depends strongly on the adhesion to natural teeth, alloys, and other dental materials [80].

Demands of esthetic materials in dentistry require the use of luting resins to bond a restoration to the tooth structure. Luting resins have superior properties in comparison to conventional lutings including higher strength, lower wear and improved esthetics [8, 13, 55, 91]. Adhesive resins enhance the long-term fracture and fatigue resistance of the restored tooth in the oral environment through a strong bond between the adhesive dental restoration and remaining tooth structures [62, 110].

Adding the phosphate ester monomer 10-methacryloyloxydecyl dihydrogen phosphate (MDP) to the bonding agent (primer or resin) appears to produce a durable resin zirconia bonding, because P-O-Zr chemical bonds are formed between MDP and zirconia [24]. These findings were confirmed also in the related clinical trials [100, 109]. Self-adhesive resins are expected to bond to zirconia similarly to other phosphate-based adhesive material [79]. MDP-based primers are available to promote chemical adhesion between resin materials and hydroxyl group present on the zirconia surface and increase its surface wettability [56, 124].

A strong and durable bond of dental zirconia can be achieved through adequate cleaning of bonding surfaces, micromechanical roughening through particle air-abrasion and/or chemical activation using phosphate monomers such as MDP containing primers and/or luting resins [62, 92, 110].

The following factors are reported to affect the bond strength of luting resin to ceramics:

1. Adequate cleaning of bonding surfaces [92]
2. Pretreatment procedures:
 - a. Micromechanical retention and/or chemical activation [63, 92, 99]
 - b. Increasing the wettability of bonding surfaces [117]
3. The depth of cure and degree of polymerization of the luting resin

1.7. The rationale of the study

Various previous studies showed a conflicting effect of aging and various surface treatments on the mechanical properties and the bonding durability of zirconia on the long-term. Teeth and dental restorations are subjected intraorally to different mechanical, thermal and chemical challenges ^[27, 39, 58, 114]. These challenges collectively could induce aging (LTD) and fatigue of restorative materials and lead to restorative failure ^[27, 67, 90]. The real influence of zirconia aging on the mechanical properties of Y-TZP zirconia in an autoclave is not clear. Some reported a positive ^[104], some a negative ^[43] and others no effect ^[42]. Additionally, the evaluation of adhesion of luting resin to aged zirconia on the long-term has a clinical relevance especially in cases of repair of fractured restorations.

Up to date there are limited data, which evaluated the influence of aging and different surface treatments on the mechanical properties, such as BFS, hardness and the bonding durability of 3Y-TZP translucent zirconia, when various surface conditioning methods, such as alumina particle air-abrasion with different pressure and priming, have been applied.

2. Purpose of the study

In the oral environment there are many destructive stimuli affecting the dental restorations and the bonding durability of zirconia as force of mastication, the effect of water and the change of temperatures.

Currently, there are limited data concerning the influence of aging and different surface treatments on the mechanical properties of translucent 3Y-TZP monolithic zirconia and the bonding durability which play an important role during the repair of zirconia in the case of chipping or partial fractures under clinical conditions.

Therefore, the aim of this study was to evaluate the influence of aging and surface treatment on biaxial flexural strength (BFS), Vickers hardness and the tensile bond strength (TBS) of a 3Y-TZP high translucent dental zirconia.

The null hypotheses were that there is no influence of both, aging and different surface treatments, on BFS and Vickers hardness of a 3Y-TZP high translucent dental zirconia, as well as on the resin-3Y-TZP zirconia ceramic bonding durability, when two different pressures are applied during alumina particle air-abrasion, and two different luting resin systems are used.

3. Material and methods

3.1. Specimen preparations

A high translucent 3Y-TZP dental zirconia (Katana HT10), with a translucency of 30% according to the manufacturer's information was used in this study. For the BFS, eighty disc-shaped specimens with a final diameter of 12 mm and a final thickness of 1.2 mm were used. While for the TBS test, one hundred ninety two disc-shaped specimens, with a final dimension of 8 mm diameter and 3.2 mm thickness were used. All specimens were sintered in a sintering oven (Nabertherm GmbH, Bremen, Germany) according to the manufacturer's instructions (1500 °C, holding time: 2 h). After that, the top side of each disc was polished down to 1 µm grit for the BFS test, while with rotating carbide paper down to 600 grit for the TBS test. Afterwards, all specimens were divided according to aging procedures into two main groups, aged (group A) and non-aged (group N).

Furthermore, both main groups were subdivided, according to the applied surface treatment as shown in Tables 2 and 3.

3.2. Aging

After sintering, aging was performed on half of zirconia discs (n=136) using an autoclave (CS, WEBECO, Bad Schwartau, Germany) according to ISO standard 13356, at 134 °C, under 2 bar, over a period of 20 h. Then the specimens were cleaned in 99% isopropanol ultrasonic bath for 3 min.

3.3. Surface treatment

For the BFS test, all specimens aged or non-aged were subdivided into four sub-groups (n=10), according to the surface treatments applied on the bottom side:

- a. Group SIN: As-sintered (control).

No treatment was applied after sintering.

- b. Group 1B: Particle air-abrasion treatment using 1 bar.

Particle air-abrasion was done using 50 µm Al₂O₃ particles at 1 bar for 15 s at a distance of 10 mm, with nozzle motion in both lateral directions. Particle air-abrasion was performed using spot blasting unit (P-G 400 K Spot Fine Blasting Unit, Harnish+Rieth, Winterbach, Germany). Afterwards all specimens were cleaned ultrasonically for 3 min in 99% isopropanol.

- c. Group 2.5B: Particle air-abrasion treatment using 2.5 bar.

Same parameters as in 1B group except the pressure used was 2.5 bar.

d. Group POL: Polishing down to 1 μm

Specimens were polished under cooling water with a diamond grinding disc (Apex CGD, yellow, Buehler, Düsseldorf, Germany) for 10 min to produce a parallel surface, then 1200 and 2500 grit silicon carbide paper (CarbiMet, Buehler, Düsseldorf, Germany) for 140-150 min followed by 3 μm and 1 μm diamond suspensions (MetaDi, Buehler, Düsseldorf, Germany) for 5 min for each. The polishing was performed using a polishing machine (AutoMet 250 and EcoMet 250, Buehler, Düsseldorf, Germany). All specimens were cleaned ultrasonically as described above.

Table 2. Design of the study for the evaluation of the biaxial flexural strength

High translucent zirconia discs, n= 80							
Aged (A), n=10 (autoclave: 20 h at 2 bar and 134 °C)	Non- aged (N), n=10	Aged (A), n=10 (Autoclave: 20 h at 2 bar and 134 °C)	Non- aged (N), n=10	Aged (A), n=10 (Autoclave: 20 h at 2 bar and 134 °C)	Non- aged (N), n=10	Aged (A), n=10 (Autoclave: 20 h at 2 bar and 134 °C)	Non- aged (N), n=10
Without surface treatment (SIN)		Polishing up till 1 μm (POL)		Particle air-abrasion: 50 μm Al ₂ O ₃ at 1 bar (1B)		Particle air-abrasion: 50 μm Al ₂ O ₃ at 2.5 bar (2.5B)	
A-SIN n=10	N-SIN n=10	A-POL n=10	N-POL n=10	A-1B n=10	N-1B n=10	A-2.5B n=10	N-2.5B n=10

3.4. Phase analysis

Three specimens were selected from each group for X-ray diffraction (XRD) surface analysis to detect the available amount of tetragonal and monoclinic phases. Each specimen was analyzed in a diffractometer (Seifert XRD 3000 PTS, GE GmbH, Munich, Germany) using Cu K α radiation recorded within the range of 20–40°, a step size of 0.04° and a scanning time per step of 10 s. Voltage and current were 40 kV and 40 mA, respectively. Integrated intensities of the relevant peaks of the diffraction patterns were evaluated using the software Peakfit v4.12 (Systat Software GmbH, Erkrath, Germany).

The amount of monoclinic phase (X_m) was calculated using the method developed by Garvie and Nicholson as follows ^[45]:

$$X_m = \frac{IM(111) + IM(111^-) - IT(111)}{IM(111) + IM(111^-) + IT(111)}$$

where M(111) and M(111⁻) represent the intensities of the monoclinic peaks, and T(111) indicates the intensity of tetragonal peak. I_T and I_M represent the integrated area under the peaks

of the tetragonal (111) and monoclinic (111) and monoclinic (111⁻) peaks around 30°, 31° and 28°, respectively. The volumetric fraction V_m was calculated using the following equation ^[112]:

$$V_m = \frac{(1.311 \times X_m)}{(1 + 0.311 \times X_m)}$$

3.5. Surface roughness

A 3D laser scanning microscope (Keyence VK-X100, Keyence GmbH, Neu-Isenburg, Germany) was used to measure the average roughness value of the surface (R_a) and the mean roughness depth (R_z) for all groups. For each subgroup, three specimens were selected. The measurements were done using a wavelength of 658 nm. The used length for the measured line was 1180 μm and the distance between the separate scans was 0.27 μm .

3.6. Biaxial flexural strength

Ten specimens from each subgroup were subjected to biaxial flexural strength test using piston-on-three balls technique in a universal testing machine (Zwick Z010, Zwick, Ulm, Germany). Each disc was placed centrally on three of 3.2 mm diameter steel balls positioned equidistant from each other on a metallic platform with a diameter of 10 mm (Figure 1). Each specimens was loaded by a piston of 1.4 mm diameter at 0.5 mm/min crosshead speed using the universal testing machine as shown in Figure 2.



Figure 1: Metallic platform with steel balls

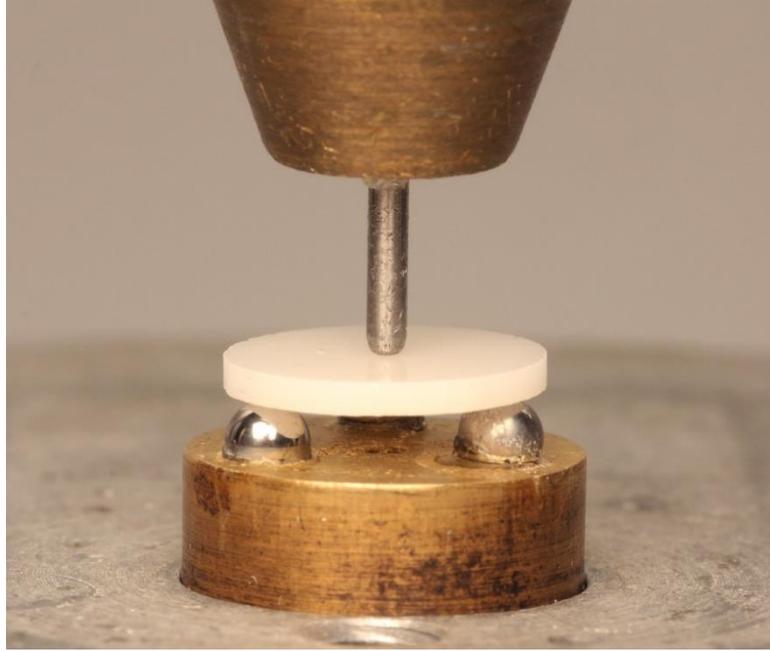


Figure 2: View of metallic platform, disc, and piston during the biaxial flexural strength test

The biaxial flexural strength was calculated using the following equation ^[88, 121]:

$$S = -0.2387 \times P \times [(X - Y)/(d^2)]$$

where S is the biaxial flexural strength (MPa); P is the fracture load (N); d is the specimen disc thickness (mm). X and Y were determined as follows:

$$X = (1 + \nu) \int [r^2 / r_3]^2 + [(1 - \nu)/2] [r^2 / r_3]^2$$

$$Y = (1 + \nu) \left[1 + \int (r^1 / r_3)^2 \right] + (1 - \nu) (r^1 / r_3)^2$$

where ν is Poisson's ratio (0.25), r_1 is the radius of support circle, r_2 is the radius of loaded area, r_3 is the radius of specimen.

3.7. Vickers hardness

An indentation tester (ZHV10, Zwick, Ulm, Germany) was used to measure the Vickers hardness (Figure 3). A piece from each broken specimen from group SIN and group POL (aged or non-aged) was randomly selected. All particle air-abrasion groups (1 or 2.5 bar) did not undergo hardness test as it is clinically not applicable and the measurements of rougher surfaces were not reliable. The rougher surfaces make it difficult to identify the exact margins of the indentations and to obtain reliable results, as observed with the SIN group measurements. An

indentation was made on each specimen on the bottom side using a loading mass of 5 kg and dwell time of 10 s (Figure 4). The measurements of Vickers hardness number were repeated 4 times at different points, the distance between each point and the other was at least 2 mm. The hardness was computed using the following equation ^[37, 94]:

$$VHN = 0.1891 \left(\frac{F}{d^2} \right)$$

where VHN is the Vickers hardness number, F is the applied load expressed in N, and d is the mean length of the two diagonals of the indentation (mm).

3.8. Fracture pattern

One typical sample of each group was selected and coated with a gold layer of 30 nm. Then the fracture pattern was examined using a scanning electron microscope (SEM, XL 30 CP, Philips, Surrey, UK) with an acceleration voltage of 15 kV.



Figure 3: Vickers hardness testing machine

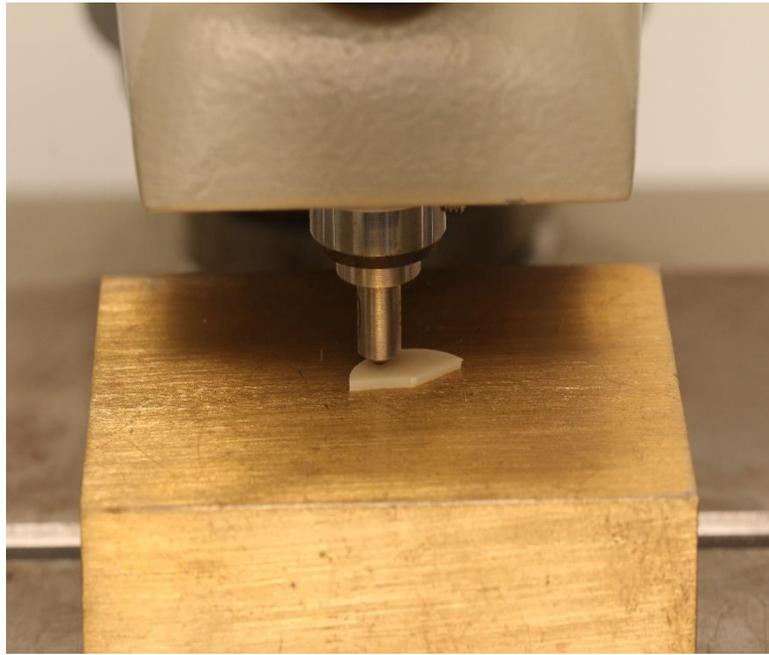


Figure 4: Vickers hardness indenter

3.9. Surface treatment and bonding

For each group, a different surface treatment was applied: no surface treatment was applied after sintering (group SIN), particle air-abrasion either with 1 bar (group 1B), or 2.5 bar (group 2.5B).

Particle air-abrasion was done using 50 μm Al_2O_3 particles at 1 or 2.5 bar for 15 s at a distance of 10 mm, with nozzle motion in both lateral directions. Particle air-abrasion was performed using spot blasting unit (P-G 400 K Spot Fine Blasting Unit, Harnish+Rieth, Winterbach, Germany). All specimens were cleaned in 99% isopropanol ultrasonic bath for 3 min. Three discs will be selected from each group to be examined in XRD to evaluate the phase transformation of zirconia.

Plexiglas tubes with standard diameter (3.2 mm) were filled with self-polymerizing restorative composite resin (Clearfil FII New Bond, Kuraray Noritake, Okayama, Japan) (n=16/group). The self-polymerizing restorative composite resin polymerized within 10 min. Every group allocated into 2 subgroups (n=16/subgroup) according to the used luting resin, group V5 and group SA. For group V5, discs were conditioned with a universal ceramic primer (Clearfil ceramic primer plus; Kuraray Noritake). The primer was distributed on the bonding surface by brush for 10 s and left to dry. Subsequently, the entire adherent surface was dried by using mild oil-free air flow. After that, the dual-polymerizing luting resin (Panavia V5; Kuraray Noritake)

was applied. For group SA; the specimens were bonded with dual-curing adhesive luting resin (Panavia SA Cement Plus Automix; Kuraray Noritake). After that, the filled tubes were bonded to the surfaces of zirconia using alignment apparatus (Figure 5) under a load of 7.4 N [12, 73, 122].

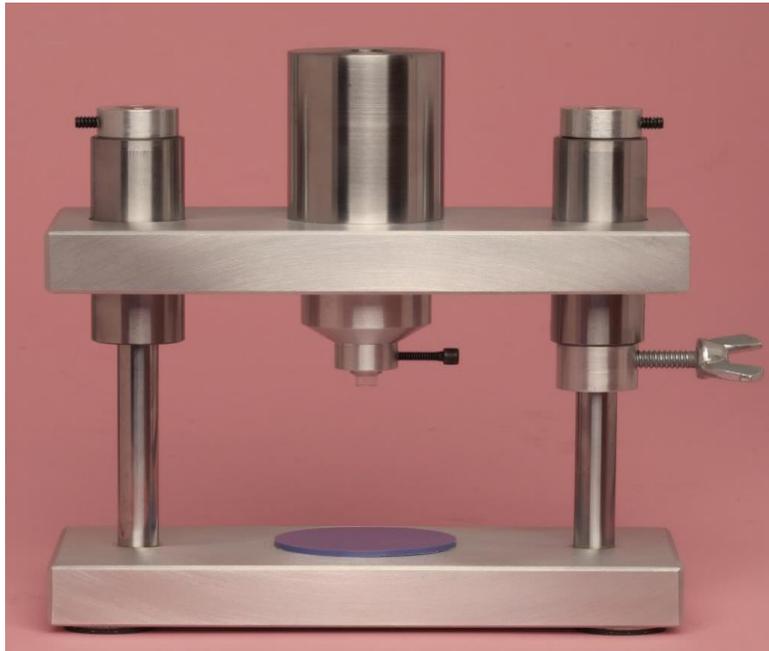


Figure 5: Alignment apparatus for bonding

The apparatus ensured that the tube axis was perpendicular to the zirconia bonded surface. Excess luting resin was removed by sponge pellet and Oxygen-inhibiting gel (Oxyguard II; Kuraray Noritake) was applied around the bonding surface. The bonded margins were light-polymerized for 20 s from two opposite sides using LED polymerizing unit (SmartLite TM PS, DENTSPLY) with a light intensity of 900-1200 mW/cm² followed by further polymerization for 90 s in a light-polymerizing unit (Heraflah, Heraeus Kulzer, Germany). Scheme of the groups is illustrated in Table 3.

Table 3. Scheme of the study design for evaluation of tensile bond strength

High translucent zirconia discs, n=192											
Aged (A), n=32 steam autoclave: 20 h at 2 bar and 134 °C		Non-aged (N), n=32		Aged (A), n=32 steam autoclave: 20 h at 2 bar and 134 °C		Non-aged (N), n=32		Aged (A), n=32 steam autoclave: 20 h at 2 bar and 134 °C		Non-aged (N), n=32	
Without surface treatment (SIN)				Particle air-abrasion: 50 µm Al ₂ O ₃ at 1 bar (1B)				Particle air-abrasion: 50 µm Al ₂ O ₃ at 2.5 bar (2.5B)			
A- SIN- SA n=16	N- AIN- SA n=16	A- SIN- V5 n=16	N- SIN- V5 n=16	A-1B- SA n=16	N-1B- SA n=16	A-1B- V5 n=16	N-1B- V5 n=16	A- 2.5B- SA n=16	N- A- 2.5B- SA n=16	A- 2.5B- V5 n=16	N- 2.5B- V5 n=16
Panavia SA Plus Automix (SA)		Clearfil ceramic primer plus + Panavia V5 (V5)		Panavia SA Plus Automix (SA)		Clearfil ceramic primer plus + Panavia V5 (V5)		Panavia SA Plus Automix (SA)		Clearfil ceramic primer plus + Panavia V5 (V5)	

Eight specimens from each group were stored in a distilled water bath at 37 °C for 3 days without thermal cycling to record the initial bond strength, and the remaining 8 specimens were stored for 150 days in 37 °C distilled water interrupted by thermal cycling for 150 days between 5 °C and 55 °C with a dwell time of 30 sec for 37,500 cycles before recording the tensile bond strength after thermal cycling [10, 64, 122, 123].

3.10. Tensile bond strength and failure mode

After the different storage conditions, tensile bond strength was tested with a universal testing machine (Zwick Z010; ZwickRoell Group, Ulm, Germany), at cross-head speed 2 mm/min using a self-aligning chain loop attachment [11, 12, 122, 123] (Figure 6).

The fracture interface of zirconia specimens were examined with a light microscope (Wild Makroskop M 420; Wild Heerburgg, Switzerland) at a 35x magnification. Debonded surfaces were assigned to cohesive failure within luting resin or composite resin, adhesive at zirconia/luting resin interface or mixed adhesive/cohesive modes. Failure areas of each mode were calculated and expressed as a percentage of the total bonding surface area for each test group. Representative samples were examined in a scanning electron microscope (SEM, XL 30 CP, Philips, Kassel, Germany) with an acceleration voltage of 15 kV after sputtering using a gold alloy conductive layer of approximately 30 nm [12, 122, 123].

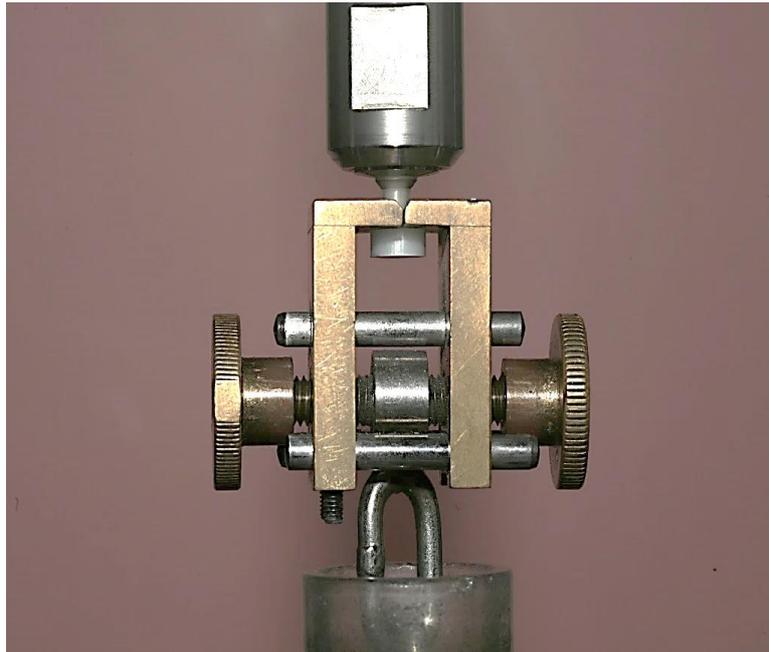


Figure 6: Self-aligning chain loop attachment

3.11. Statistical analysis

Shapiro-Wilk test verified normal distribution of the collected data and then the data were analyzed to determine the effect of aging and surface treatment on the tested variables. One-way analysis of variance (ANOVA) followed by t-test were used for the BFS analysis. In addition, Kruskal-Wallis and Mann-Whitney-U tests were used to analyze the hardness measurements and the TBS.

The significance level was set to $p \leq 0.05$. All calculations were made with statistical software (IBM SPSS FOR Windows; Version 20.0, IBM SPSS, Inc., Chicago, IL, USA).

4. Results

4.1. Aging and phase analysis

XRD analysis showed that both aging and different surface treatments result in different amounts of monoclinic phase. The highest values of the monoclinic phase ratio (vol.%) were found within groups A-SIN, A-1B, and A-2.5B. While groups N-1B and N-2.5B showed a lower monoclinic phase ratio. Both polished groups (N-POL and A-POL) and group N-SIN exhibited almost no monoclinic phase (Table 4). Typical XRD spectra for all groups are shown in Figures 7 and 8.

Table 4. Mean and standard deviation (SD) of monoclinic phase ratio of the zirconia measured by XRD and roughness measured by laser scanning microscopy

Group	Aging	Monoclinic Phase	Ra Mean \pm SD	Rz Mean \pm SD
		Ratio Mean \pm SD (vol.%)	(μm)	(μm)
SIN	Non-aged (N)	0.0 \pm 0.0	0.449 \pm 0.096	2.321 \pm 0.422
	Aged (A)	39.9 \pm 0.7	0.502 \pm 0.055	2.434 \pm 0.221
1B	Non-aged (N)	7.5 \pm 2.4	0.531 \pm 0.051	2.738 \pm 0.176
	Aged (A)	41.5 \pm 0.3	0.434 \pm 0.032	2.241 \pm 0.136
2.5B	Non-aged (N)	10.4 \pm 1.5	0.528 \pm 0.020	2.845 \pm 0.092
	Aged (N)	38.5 \pm 2.8	0.485 \pm 0.044	2.480 \pm 0.150
POL	Non-aged (N)	2.1 \pm 0.6	0.006 \pm 0.001	0.034 \pm 0.012
	Aged (A)	2.1 \pm 0.5	0.002 \pm 0.001	0.014 \pm 0.003

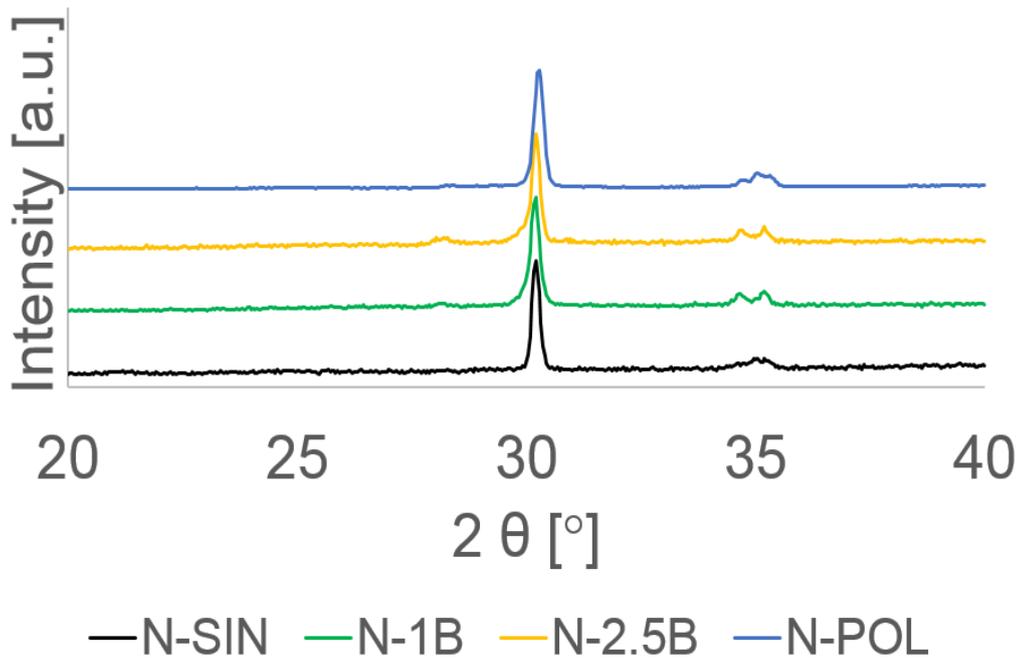


Figure 7. Representative XRD spectra of all non-aged groups with different surface treatments. As sintered (SIN); particle air-abrasion with 50 μm Al_2O_3 at 1 bar (1B); particle air-abrasion with 50 μm Al_2O_3 at 2.5 bar (2.5B), and polished down to 1 μm (POL)

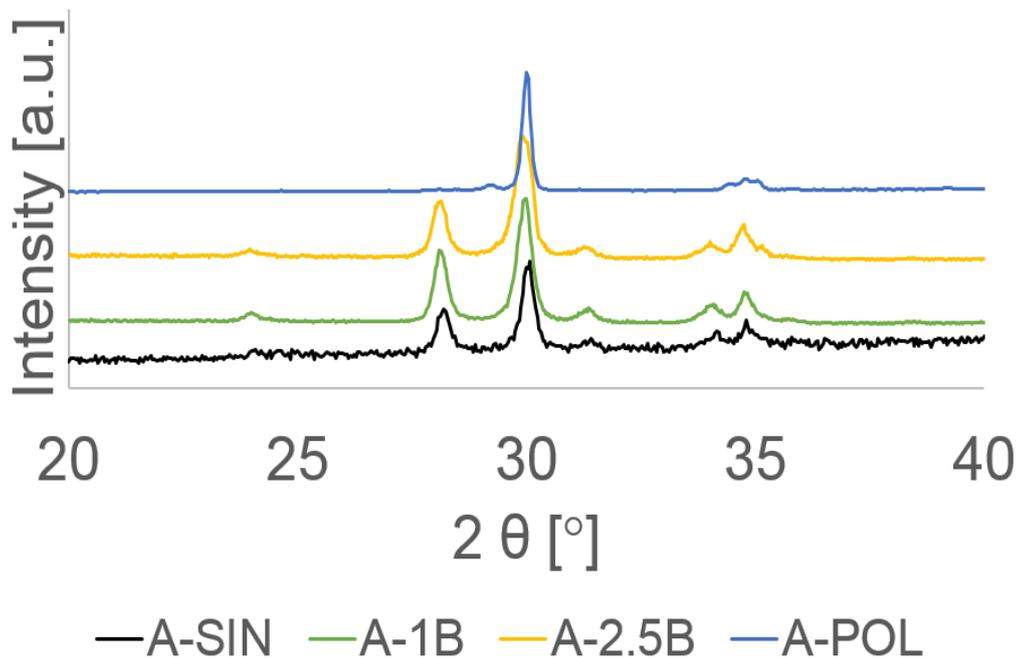


Figure 8. Representative XRD spectra of all aged groups with different surface treatments. As sintered (SIN); particle air-abrasion with 50 μm Al_2O_3 at 1 bar (1B); particle air-abrasion with 50 μm Al_2O_3 at 2.5 bar (2.5B), and polished down to 1 μm (POL)

4.2. Surface roughness

The polishing groups either aged or non-aged represented the lowest mean R_a and R_z values. The descriptive statistics is shown in Table 4.

4.3. Biaxial flexural strength

The mean BFS values increased with aging and particle air-abrasion, as shown in groups A-SIN, N-1B and N-2.5B. Likewise, both groups A-1B and A-2.5B showed comparable BFS mean values. While the BFS mean value decreased within both polished groups either aged or non-aged as well as in N-SIN group (Table 5).

Table 5. Mean and standard deviation of BFS (MPa) of all test groups

Groups	Aging	
	Non-aged groups Mean \pm SD	Aged groups Mean \pm SD
SIN	720 \pm 37 C, α	1064 \pm 27 A, β
1B	1153 \pm 92 A, α	1110 \pm 76 A, α
2.5B	1137 \pm 89 A, α	1105 \pm 74 A, α
POL	894 \pm 96 B, α	888 \pm 86 B, α

Within the same column, mean with the same capital letter are not statistically different ($p > 0.05$). Within the same row, mean with the same Greek letter are not statistically different $p > 0.05$.

4.4. Vickers hardness

The Vickers hardness ranged from 1346 HV5 (A-POL) to 1446 HV5 (N-SIN). The aging had no effect on the Vickers hardness. Likewise, with the polishing treatment both polished groups (N-POL and A-POL) reported a lower hardness than both as-sintered groups (N-SIN and A-SIN), as shown in Table 6.

Table 6. Median, mean and standard deviation of hardness (HV5)

Groups	Aging			
	Non-aged groups		Aged groups	
	Median	Mean \pm SD	Median	Mean \pm SD
SIN	1446 A α	1413 \pm 127	1436 A α	1428 \pm 98
POL	1347 B α	1340 \pm 21	1346 B α	1346 \pm 18

Within the same column, mean with the same capital letter are not statistically different ($p > 0.05$). Within the same row, mean with the same Greek letter are not statistically different $p > 0.05$.

4.5. Fracture pattern

SEM micrographs of typical samples of each group are shown in Figure 9. In most cases, failure of these samples was initiated from a surface crack. Within the particle air-abrasion treated specimens either aged or not and A-SIN, the SEM micrographs showed many subsurface cracks connected with the surface cracks.

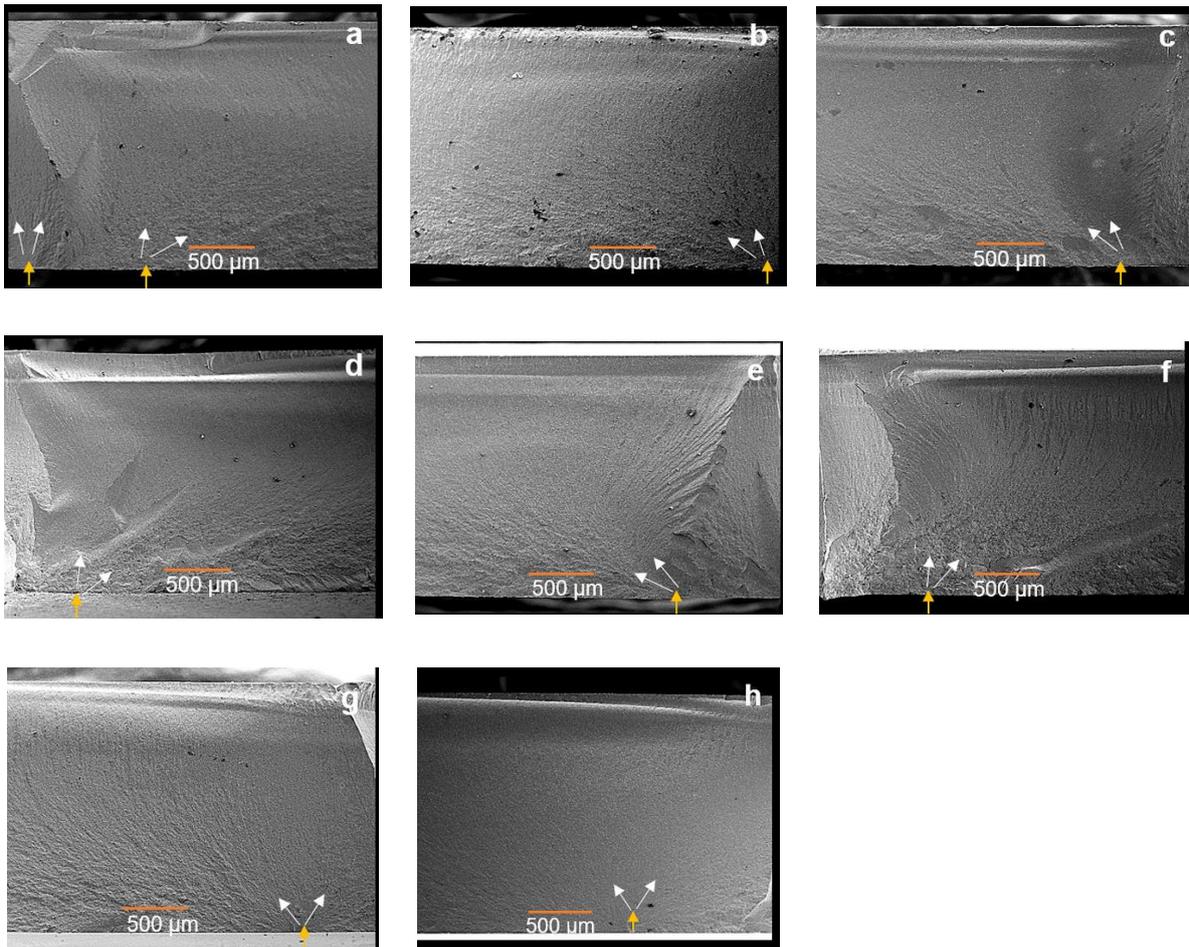


Figure 9. Representative SEM micrographs of typically fractured specimens; (a) and (b) as-sintered specimens; (c) and (d) particle air-abrasion with 50 μm Al_2O_3 at 1 bar; (e) and (f) particle air-abrasion with 50 μm Al_2O_3 at 2.5 bar; (g) and (h) polished down to 1 μm , exhibiting the origin of failure (yellow arrow on the treated surface). Thin white arrows follow the hackle lines in the direction of crack propagation. In most specimens, the failure was initiated from a surface crack then going deeper through the connected subsurface cracks.

4.6. Tensile bond strength and failure mode

The medians of tensile bond strength (TBS) for all groups are shown in Table 7. After 3 days of water storage, 20 hours aging showed no effect on the TBS except for group A-SIN-SA, in which TBS decreased significantly in comparison to the non-aged groups. The particle air-abrasion cause a significant increase in TBS for all test groups. However, the used different pressure showed no statistical significant effect, group N-1B-V5 showed a lower statistically significant effect in TBS compared to group N-2.5B-V5. Groups N-1B-SA and A-1B-SA showed no statistical significance before and after the thermocycling, although most of the groups showed a significant decrease in TBS after long-term storage in water with thermal cycling. So specimens of group V5 which were abraded with 1 bar showed a significant decrease in TBS in comparison with 2.5 bar after thermocycling. Figures 10 and 11 show a boxplot for TBS after 3 days storage and 150 days storage with thermocycling.

Table 7. Tensile bond strength of groups in MPa, medians, (n=8). Statistically different medians ($p \leq 0.05$) are indicated by different upper case letter (within a column for the same grouping factor), or by different lower case letter (within a column for the same surface treatment), or by different greek letters (within a row)

Main group	Group with Code	3 Days	150 Days
		Median	Median
Panavia SA, No-aging (Gr factor 1)	N-SIN-SA	10.8 B, a, α	0.0 B, a, β
	N-1B-SA	29.5 A, a, α	25.1 A, ab, α
	N-2.5B-SA	37.0 A, a, α	27.0 A, a, β
Panavia V5, No-aging (Gr factor 2)	N-SIN-V5	12.6 C, a, α	0.0 C, a, β
	N-1B-V5	27.3 B, a, α	11.7 B, c, β
	N-2.5B-V5	38.7 A, a, α	22.8 A, ab, β
Panavia SA, 20 h Aging (Gr factor 3)	A-SIN-SA	6.4 B, b, α	0.0 B, a, β
	A-1B-SA	32.5 A, a, α	27.7 A, a, α
	A-2.5B-SA	32.4 A, a, α	23.2 A, ab, β
Panavia V5, 20 h Aging (Gr factor 4)	A-SIN-V5	7.5 B, ab, α	0.0 B, a, β
	A-1B-V5	29.5 A, a, α	15.4 A, bc, β
	A-2.5B-V5	34.0 A, a, α	20.1 A, b, β

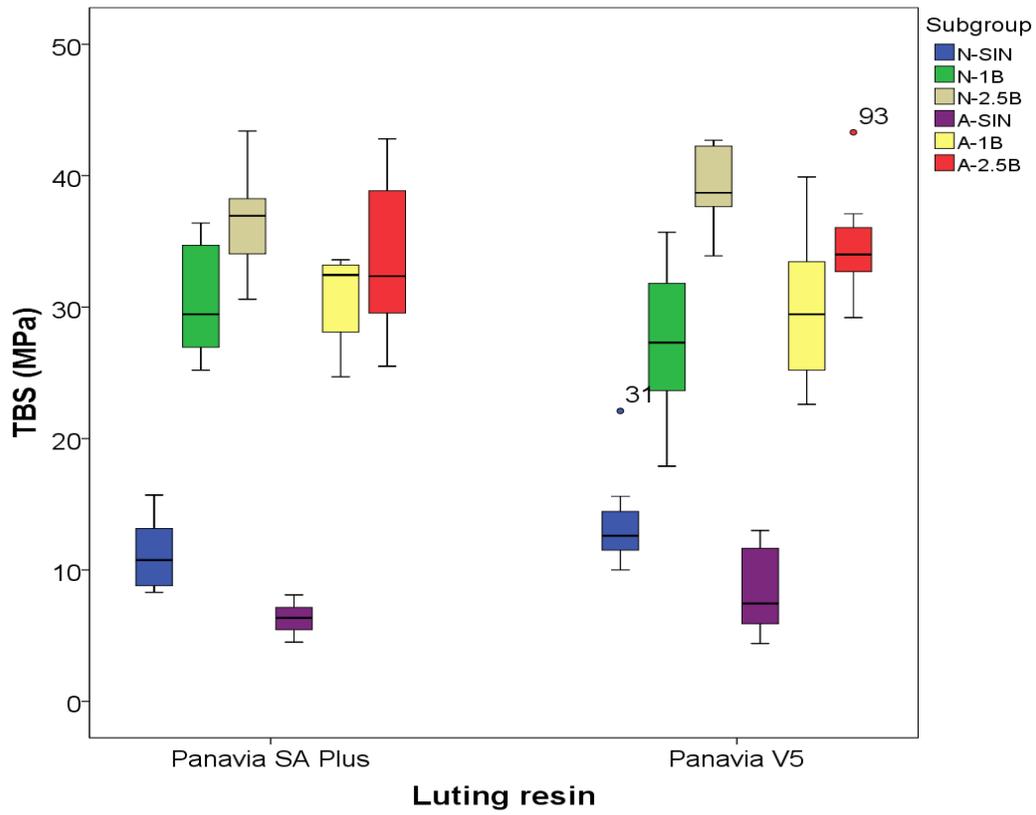


Figure 10: Boxplot of TBS after 3 days of storage

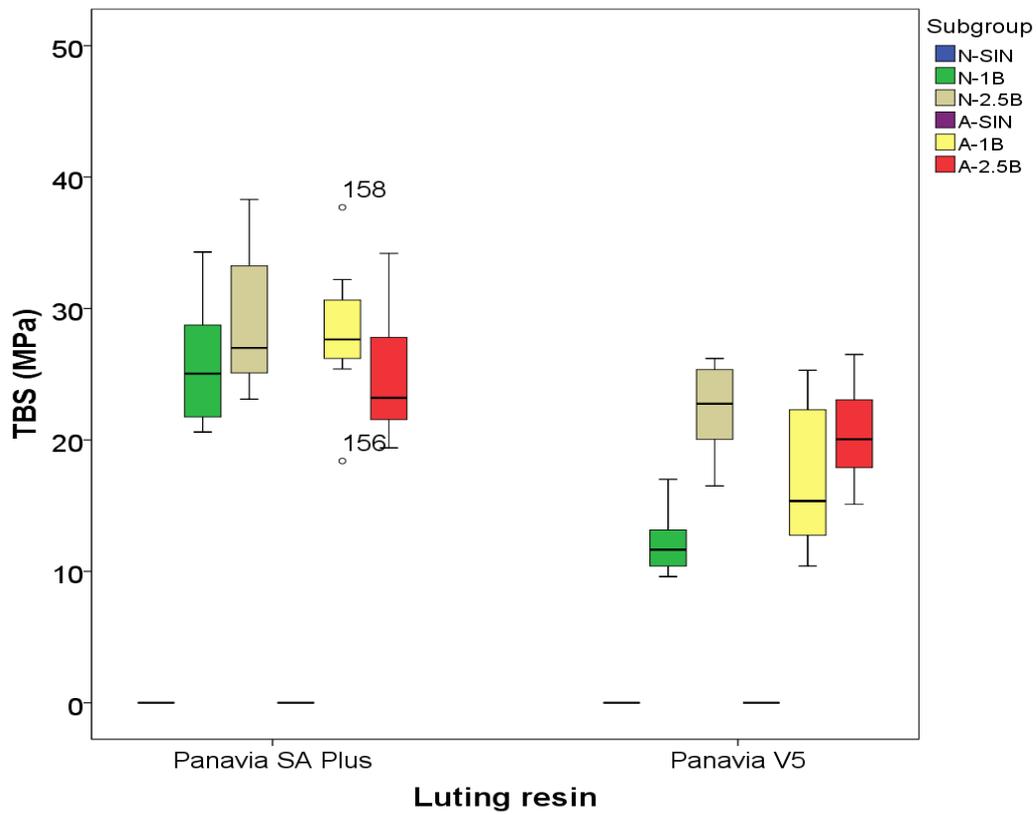


Figure 11: Boxplot of TBS after 150 days of storage with thermocycling

The mode of failure of all test groups is illustrated in Figures 12 and 13. Most specimens tested after 3 days storage showed a predominantly cohesive failure within the luting and tube resin except for aged groups, which received no surface treatment. These groups showed almost complete adhesive failure. After thermal cycling, groups A-1B-V5 and N-1B-SA shifted from cohesive to adhesive failure, while the other groups except all SIN groups, still had a predominantly cohesive mode of failure. All SIN groups debonded spontaneously during thermal cycling with a pure adhesive mode of failure, as shown in the SEM representative photographs (Figures 14-16).

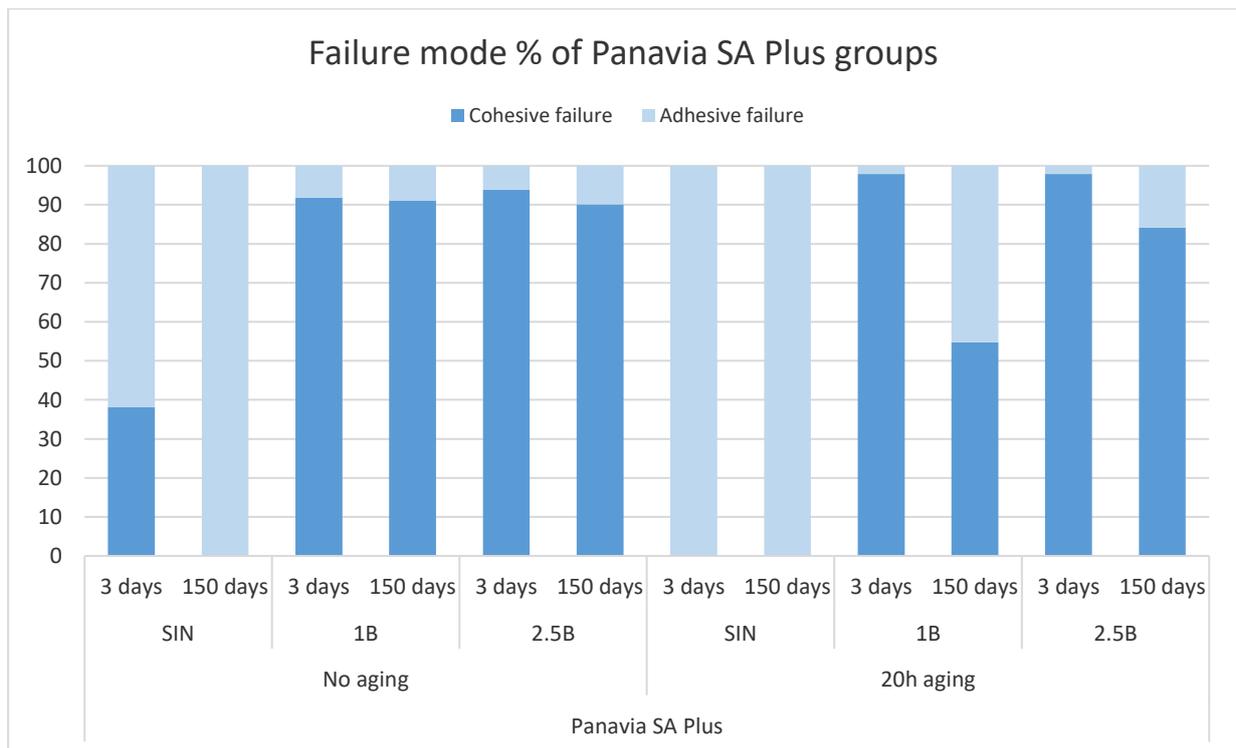


Figure 12: Type of bonding failure mode as identified with a light microscope at 30x magnification and calculation in % of bonding area for all test groups bonded with Panavia SA Plus

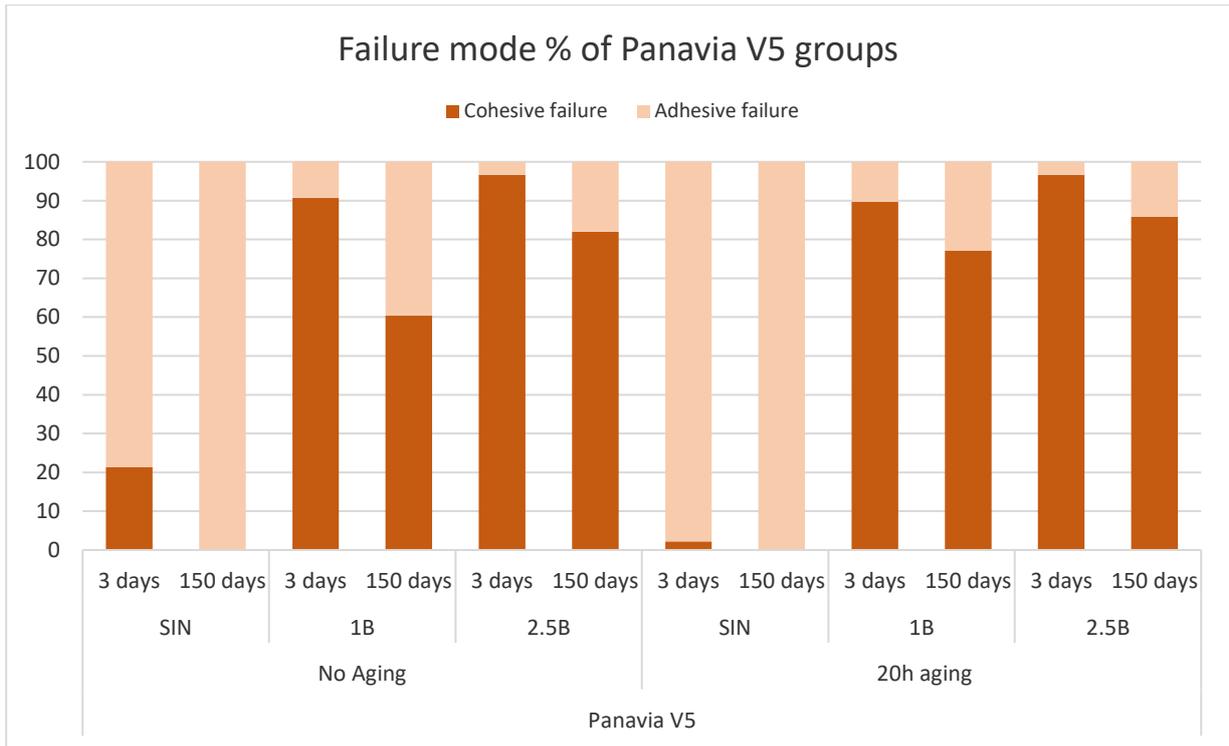


Figure13: Type of bonding failure mode as identified with a light microscope at 30x magnification and calculation in % of bonding area for all test groups bonded with Panavia V5

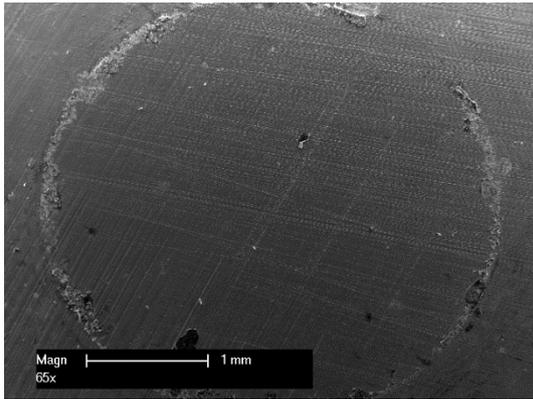


Fig. 14a. SEM of representative sample of subgroup N-Sin-SA after 150 days of storage with thermocycling presenting a complete adhesive failure

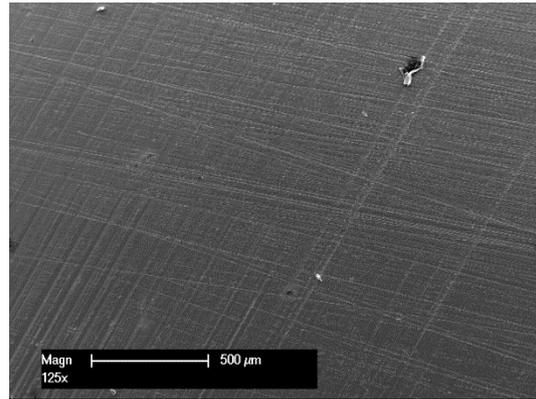


Fig. 14b. Detailed magnification of Fig. 1a, showed a complete adhesive failure at the zirconia surface

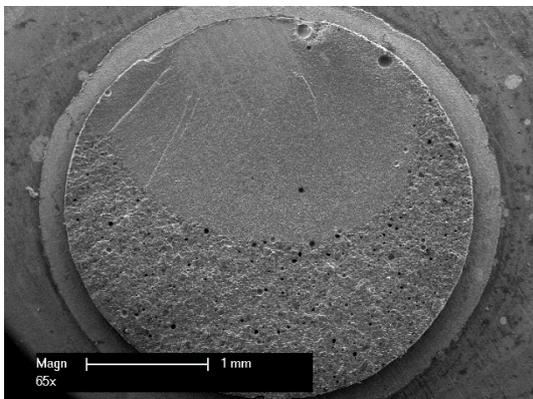


Fig. 15a. SEM of representative sample of subgroup N-1b-V5 after 3 days of storage presenting a predominantly cohesive failure mode. Only a small of area at the top failed adhesively

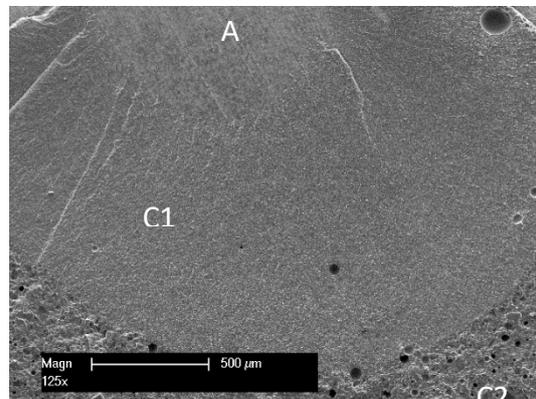


Fig. 15b. Detailed magnification of Fig. 2a. A, a small area of adhesive failure at the zirconia surface; C1, cohesive failure in Panavia V5 luting resin; C2, cohesive failure in the tube resin

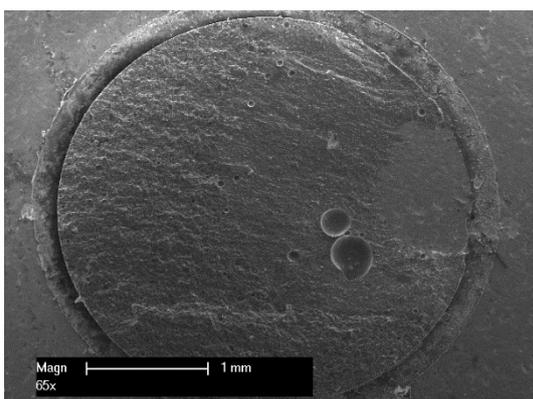


Fig. 16a. SEM of representative sample of subgroup N-1b-SA after 150 days of storage presenting a predominantly cohesive failure mode. Only a small of area at the right side failed adhesively

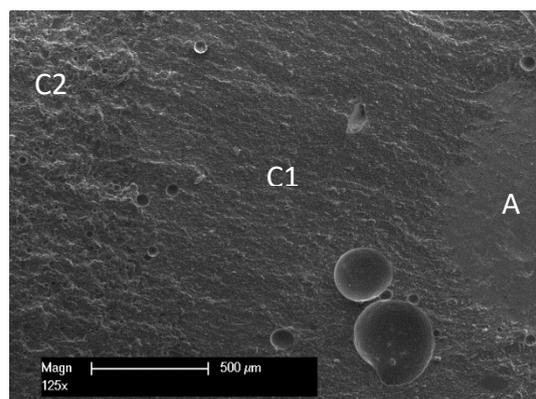


Fig. 16b. Detailed magnification of Fig. 3a. A, a small area of adhesive failure at the zirconia surface; C1, cohesive failure in Panavia SA plus luting resin; C2, cohesive failure in the tube resin

5. Discussion

Many studies have been performed to predict the behavior of dental zirconia ceramics in the oral environment regarding the aging ability as well as the mechanical properties [5, 41, 90]. In this concern, the literature shows conflicting results of the effect of aging and surface treatment on the mechanical properties of zirconia. For that reasons the current study was carried out to evaluate the influence of aging and surface treatment on the biaxial flexural strength, measuring the hardness and the tensile bond strength to a high translucent dental zirconia.

The aging protocol used in this study was in an autoclave at 134 °C, under 2 bar, over a period of 20 h, which was consistent with previous studies [27, 58]. These studies showed that this protocol promotes an extensive t→m phase transformation. Thus the aging performed in our study should be sufficient to observe significant changes induced by different surface treatments.

In the current study, XRD analysis showed a different percentage of phase transformation. The highest monoclinic phase ratio (38.5 ± 2.8 vol.% to 41.5 ± 0.3 vol.%) was found within groups A-1B, A-2.5B and A-SIN. These findings show that both aging and alumina particle air-abrasion produced a significant t→m phase transformation. These results are consistent with other studies [44, 47, 89, 90]. In contrast, both non-aged specimens with 1 or 2.5 bar particle air-abrasion showed a lower monoclinic phase ratio (7.5 ± 2.4 vol.% to 10.4 ± 1.5 vol.%). These results indicate that the differing pressures during particle air-abrasion treatment produced a comparable amount of monoclinic phase on the surface of zirconia. In addition, both polished groups (N-POL and A-POL) and group N-SIN showed almost no vol.% of monoclinic phase ratio revealing almost no t→m phase transformation on zirconia surface. These results may have been due to that neither sintering nor polishing treatment probably promote any monoclinic phase transformation as shown with groups N-SIN and N-POL. These findings also confirm other studies, which showed that polishing produced no t→m phase transformation [3, 53, 85]. It can be assumed that the previously transformed monoclinic layer (20 μm thicknesses) caused by aging on the surface of zirconia within group A-POL was removed during the polishing procedure.

BFS data demonstrated that groups with monoclinic phase ratio ranging from 7.5 ± 2.4 vol.% to 41.5 ± 0.3 vol.% showed the highest mean BFS. However, this increase in the BFS was independent on the percentage of the phase transformation. The increase in the BFS might be explained by the toughening mechanism, as the t→m phase transformation lead to volumetric

expansion at a localized area around the superficial defects resulting in compressive stresses concentrated around these defects and consequently stopping crack propagation [5, 45, 87], requiring more force to bring a sample to fracture. The SEM micrographs show many subsurface cracks connected with the surface cracks within the particle air-abrasion groups. This indicates, that there were already some microcracks inside the material, which were stopped by phase transformation. This phase transformation induced some compressive stress resulting in a higher BFS as described above. Groups with a lower BFS do not show these many subsurface cracks as there were no microcracks that were stopped by phase transformation. In contrast to the current study, other studies showed that neither aging nor particle air-abrasion affected the strength of 3Y-TZP zirconia [39, 42, 88, 121].

Regarding the effect of the different used pressures during the particle air-abrasion on the strength of zirconia, the current results revealed no significant differences in BFS. In this regard, another study also concluded that there were no significant differences in the BFS of zirconia after particle air-abrasion treatment at different pressures [14]. Likewise, both polished groups (aged or non-aged) and group N-SIN showed a reduced BFS, which might be a result of no monoclinic phase on the surface of zirconia. In addition, group N-SIN showed a lower BFS in comparison to both polished groups either aged or non-aged. These results may have been due to the different surface roughness of the as-sintered and polished specimens. As the as-sintered specimens showed a rougher surface than the polished specimens, and the surface defects and flaws might later on act as a stress concentration sites, which led to a significant decrease in the strength of zirconia [76, 119].

Hardness results of the current study showed that the aging had no effect on the hardness which is in consent to the finding of others [31]. In contrast, some studies showed a strong correlation between the reduction of hardness and the increase of monoclinic phase. They reported 41% hardness reduction in femoral head of zirconia with 78% monoclinic phase content [29] and 30% hardness reduction with 48 ± 1 wt.% monoclinic phase fraction [22]. However, in the current study the monoclinic phase content was 39.9 ± 0.7 vol.% with the as-sintered aged specimens, which showed no effect on the hardness. One explanation for this might be that the $t \rightarrow m$ phase transformation percentage was lower than that in previous studies.

Likewise, the findings concerning the effect of polishing treatment on hardness, both as-sintered groups (aged or non-aged) showed a lower hardness than the polished groups (aged or non-aged), which might be an artefact of the measurement, as the rougher surface of the as-sintered samples makes it difficult to identify the exact margins of the indentations and to obtain

reliable results. However, the null hypothesis concerning the hardness is confirmed by the results of the study.

TBS between the luting resin and zirconia in this study was measured with universal testing machine. TBS test was chosen due to its uniform stress distribution and to avoid the inaccurate complex forces outcome that is produced by shear tests [33]. It was also chosen over a microtensile bond test to avoid its technique sensitivity and exclude all the micro/movements and vibrations produced during specimen preparation [98]. Regarding the weak point of the tensile bond test which is the adjustment of the composite tubes on the ceramic specimens, it had been solved using a paralleling bonding device that ensures their perfect adjustment in 90° on the ceramic surface [111].

According to the ISO specification thermocycling should be done between 5 °C and 55 °C, with a minimum of 500 cycles and a dwell time more than 20 s. However, several scientific studies proposed more cycles or longer dwell times to simulate more time in the oral cavity. In this study the thermocycling protocol chosen was adopted by several studies before testing TBS of luting resin to zirconia. This protocol was applied as it uses the largest number of cycles (37,500 cycles) in the literature therefore simulating more time in the oral cavity and it ensures complete water saturation of the bonded specimens [10, 38, 65].

Our results showed that 20 hours aging in an autoclave significantly decreased TBS only for group A-SIN-SA in comparison to the non-aged groups after 3 days storage. A previous study [86] showed that aging of a 3Y-TZP zirconia for 5 hours in an autoclave decreased significantly the bond strength after 24 hour storage in distilled water at 37° C without thermocycling. These specimens were received no surface treatment and were bonded with RelyX Unicem resin and conditioned with MDP-containing primer. These results considered clinically not reliable as the tested TBS was only for short period of storage and without thermocycling.

Alumina particle air-abrasion caused a significant increase in TBS for all test groups; these findings are consistence with several previous studies [99, 107, 116]. However, the used different pressure showed no statistical difference, the group N-1B-V5 showed a significantly lower TBS in comparison to group N-2.5B-V5. These finding are in concent with a previous study [107], which showed that the bond strength increased significantly within the groups using a pressure more than 1 bar during particle air-abrasion. In contrast, another study [68] reported that there was no significant differences in the bond strength at different used pressure when either 1, 2, 4 or 6 bar were used. The specimens which were abraded with 1 bar and bonded with Panavia V5 cement (group V5), showed a reduced bond strength in comparison to Panavia

SA plus (group SA). No bond strength reduction was observed for specimens abraded with 2.5 bar. These results are in a good accordance with a recent study ^[108], which showed that the TBS of Panavia V5 groups increased with an increased pressure during particle air-abrasion for short-term water storage conditions (1 day and 30 days, without thermocycling), while Panavia SA plus groups did not vary with the used pressure. That might be due to the effect of primer which applied before luting might has enhanced the bond strength to a greater extend, which might be same case in our study. In general increasing the pressure during particle air-abrasion treatment results in an increased surface roughness and bonding surface area, which enhances the surface wettability and enhance the bond strength.

In the current study, most groups showed a significant decrease in bond strength after long-term storage in water with thermocycling compared with only 3 days water storage. Similar results were reported in several previous studies ^[38, 65, 120]. A previous study ^[86] investigated the effect of aging in autoclave for 5 hours on the resin bond strength to zirconia without thermocycling. That is in contrast to the current study, where the specimens were subjected to 150 days of thermocycling. Furthermore, some studies ^[56, 102, 110] showed that pretreatment of surface zirconia with an MDP-containing primer enhances the bond strength and its durability. Groups N-1B-SA and A-1B-SA showed no significant decrease in TBS. This finding was in a good accordance with previous studies ^[65, 120, 129], which showed that because of MDP containing luting resin there was no reduction in bond strength due to water storage.

In the present study, it was also observed that after 3 days storage most test groups showed a predominantly cohesive failure. These finding were previously observed in other studies ^[38, 95]. In addition, it was also observed that after long-term storage in water with thermal cycling most test groups still had a predominantly cohesive failure mode except groups which received no surface treatment. These groups debonded spontaneously during thermocycling with an almost completely adhesive mode of failure. These findings are in a good accordance with previous studies ^[38, 65, 123]. It can also be seen from our results that particle air-abrasion treatment which lead to formations of micro irregularities are essential to achieve a durable bond strength to luting resin as shown with the as-sintered groups, which completely debonded during thermal cycling. This was also consistence with previous studies ^[62, 65, 123].

Translucent 3Y-TZP zirconia showed promising results concerning the effect of aging and surface treatment on the mechanical behavior and on the bonding durability. The durability of resin bonding to aged zirconia plays an important role during repair of a fractured restoration. Both aging and alumina particle air-abrasion caused a significant increase in BFS, while the hardness was not affected. The used pressure during the alumina particle air-abrasion did not

affect BFS and hardness of zirconia significantly. Regarding the TBS, 20 hours aging revealed almost no effect on the TBS even with long-term storage in water with thermocycling. That is in contrast to the results of a previous study^[86], which showed that the 5 hours aging of a 3Y-TZP zirconia without thermocycling decreased the TBS significantly. This reduction of bond strength of aged zirconia might be as a result of the presence of monoclinic phase on zirconia surface, which might result in surface alteration that compromise the bond durability. In general in this study alumina particle air-abrasion improved TBS, while the used pressures reported variable results depending on the type of used bonding system.

Although the current study showed promising results regarding the mechanical behavior of 3Y-TZP ceramics, it should be considered that the fatigue behavior of this zirconia needs to be investigated under thermal and mechanical loading in aqueous environment over long time periods. Also for confirming the bond strength results, specimens should be subjected to mechanical fatigue in addition to the thermal stress. This would better simulate the mechanical loading and thermal conditions in the oral environment, which were not applied in the current study. These and other aspects should be considered in future studies.

6. Conclusions

Within the limitations of this laboratory study the following conclusions are drawn:

- 1- Both aging and particle air-abrasion demonstrated a significant t→m phase transformation which leads to a significant increase in BFS of high translucent 3Y-zirconia, while polishing caused no significant monoclinic phase transformation.
- 2- Aging leading to a ratio up to 40 vol.% of monoclinic phase seems to have no effect on the hardness.
- 3- The varied pressures during particle air-abrasion had no influence on the tested mechanical and crystallographic properties.
- 4- The positive response of this zirconia to aging and surface treatment seems to provide promising mechanical properties of this material during the repair in case of chipping or partial fractures under clinical conditions.
- 5- Twenty hours of aging in the autoclave has almost no influence on the resin bond strength.
- 6- For Panavia V5 luting resin (primer/resin system) a higher pressure during particle air-abrasion might be necessary to optimize the surface area of zirconia for bonding and to achieve a long-term durable bond strength to zirconia.

7. Summary

The aim of this study was to evaluate the influence of aging and surface treatment on biaxial flexural strength (BFS), Vickers hardness (VHN) and the tensile bond strength (TBS) of high translucent dental zirconia.

For the BFS test, one surface of 80 disc-shaped zirconia specimens (1.2 mm thickness and 12 mm diameter) was polished down to 1 μm . Half of the specimens were aged in an autoclave at 134 °C, under 2 bar (group A) for 20 h, the other half were not aged (group N). The specimens of each group were randomly distributed according to the surface treatment: no treatment (SIN), particle air-abrasion with 50 μm alumina particles at 1 bar (1B) or 2.5 bar (2.5B) or polishing down to 1 μm (POL). The phase transformation, surface roughness, BFS and VHN were evaluated using X-ray diffraction, a laser scanning microscope, a universal testing machine and an indentation tester, respectively. For the TBS test, half of 192 disc-shaped zirconia specimens (3.2 mm thickness and 8 mm diameter) were aged in an autoclave for 20 h (134 °C, 2 bar). For each group, a different surface treatment was applied: as-sintered (group SIN), alumina particle air-abrasion either with 1 bar (group 1B), or 2.5 bar (group 2.5B). Discs were later bonded to Plexiglas tubes filled with composite resin using two different luting resins, one with included phosphate monomer in the resin itself, one with a separate phosphate monomer containing primer. All specimens subjected to tensile bond strength test before and after thermocycling.

No monoclinic phase was detected for three groups (N-SIN, N-POL, and A-POL). Other groups showed monoclinic phase ratio ranging from 7.5 ± 2.4 vol.% (N-1B) to 41.5 ± 0.3 vol.% (A-1B). BFS increased significantly with aging and particle air-abrasion, ranging from 720 ± 37 MPa (N-SIN) to 1153 ± 92 MPa (N-1B). Aging had no significant effect on the hardness. It ranged from 1346 HV5 (A-POL) to 1446 HV5 (N-SIN). Before thermocycling, 20 hours aging showed no effect on the TBS except for group A-SIN-SA, in which TBS decreased significantly. Alumina particle air-abrasion caused a significant increase in TBS for all test groups. A variation in pressure showed no statistical effect, except for the use of phosphate monomer containing primer, where TBS after particle air-abrasion with 1 bar was lower than after using 2.5 bar. After thermocycling, TBS of most groups decreased significantly. Specimens of the primer group which were abraded at 1 bar, showed a significant decrease in TBS in comparison with particle air-abrasion at 2.5 bar.

A certain amount of monoclinic phase at the surface strengthened the high translucent dental zirconia ceramic, while hardness and roughness were not influenced. The used pressure during particle air-abrasion did not influence significantly the evaluated properties. Twenty hours

aging in the autoclave had almost no influence on the bond strength of the test groups. For the primer/resin bonding system higher pressure during particle air-abrasion might be advantageous to obtain a higher and more durable bond strength to zirconia ceramic.

Zusammenfassung:

Ziel dieser Studie war es, den Einfluss der Alterung und Oberflächenbehandlung auf die biaxiale Biegefestigkeit (BFS), die Vickers-Härte (VHN) und den Klebeverbund zwischen Kompositkunststoff und hochtransluzentem Zirkoniumoxid zu untersuchen.

Für den BFS-Test wurde eine Oberfläche von 80 scheibenförmigen Zirkoniumoxidproben (1,2 mm Dicke und 12 mm Durchmesser) auf 1 μm poliert. Die Hälfte der Proben wurde in einem Autoklaven bei 134°C unter 2 bar (Gruppe A) 20 Stunden lang gealtert, die andere Hälfte wurde nicht gealtert (Gruppe N). Die Proben jeder Gruppe wurden gemäß der Behandlung der zweiten Oberfläche zufällig verteilt: keine Behandlung (SIN), Korundstrahlen mit 50 μm Aluminiumoxidpartikeln bei 1 bar (1B) oder 2,5 bar (2,5B) oder Polieren auf 1 μm (POL). Die Phasenumwandlung, Oberflächenrauheit, BFS und VHN wurden unter Verwendung von Röntgenbeugung (XRD), einem Laser-Scanning-Mikroskop, einer Universalprüfmaschine bzw. einem Härteprüfgerät evaluiert. Für den Test des Klebeverbundes wurde die Hälfte von 192 scheibenförmigen Zirkoniumoxidproben (3,2 mm Dicke und 8 mm Durchmesser) in einem Autoklaven 20 h lang (134 ° C, 2 bar) gealtert. Für jede Gruppe wurde eine andere Oberflächenbehandlung angewendet: gesintert (Gruppe SIN), Korundstrahlen entweder mit 1 bar (Gruppe 1B) oder 2,5 bar (Gruppe 2,5B). Die Scheiben wurden dann unter Verwendung zweier Adhäsivsysteme mit Plexiglasröhren verklebt, die mit Kompositkunststoff gefüllt waren. Ein Adhäsivsystem beinhaltete ein Phosphatmonomer direkt im Kompositkleber, während das andere Adhäsivsystem einen zusätzlichen Primer mit Phosphatmonomer aufwies. Bei allen Proben wurden vor und nach dem Thermocycling der Klebeverbund mittels Zugfestigkeitstest evaluiert.

Bei drei Gruppen (N-SIN, N-POL, und A-POL) wurde keine monokline Phase entdeckt. Andere Gruppen zeigten ein monoklines Phasenverhältnis im Bereich von 7.5 ± 2.4 vol.% (N-1B) bis 41.5 ± 0.3 vol.% (A-1B). Die BFS stieg signifikant mit Alterung und Korundstrahlen im Bereich von 720 ± 37 MPa (N-SIN) bis 1153 ± 92 MPa (N-1B). Die Alterung hatte keinen signifikanten Einfluss auf die Härte, die zwischen 1346 HV5 (A-POL) bis 1446 HV5 (N-SIN) betrug. Ohne Thermocycling zeigten die 20 Stunden Alterung keinen signifikanten Einfluss auf den Klebeverbund außer bei der A-SIN-SA Gruppe, wo der Klebeverbund signifikant reduziert war. Das Korundstrahlen bewirkte eine signifikante Steigerung des Klebeverbundes für alle geprüften Gruppen. Die verschiedenen verwendeten Strahldrücke wiesen keinen statistisch signifikanten Einfluss auf den Klebeverbund auf außer bei Verwendung des Primer haltigen Adhäsivsystems, wo der Klebeverbund bei einem Strahldruck von 1 bar signifikant geringer war als bei Verwendung eines Strahldrucks von 2.5 bar. Die meisten Gruppen zeigten nach dem

Thermocycling einen signifikanten Abfall des Klebeverbunds. Die Proben der mit Primer haltigen Adhäsivsystem verklebten Proben zeigten nach Abstrahlen mit 1 bar einen signifikanten Abfall des Klebeverbunds im Vergleich zu den mit 2.5 bar gestrahlten Proben. Eine gewisse Menge von monokliner Phase an der Oberfläche verstärkte die hochtransluzente Zirkonoxidkeramik, während Härte und Rauheit nicht beeinflusst wurden. Der verwendete Druck während des Korundstrahlens hatte keinen signifikanten Einfluss auf die untersuchten Eigenschaften. Zwanzig Stunden Alterung im Autoklaven hatte fast keine Auswirkung auf die Haftfestigkeit der Testgruppen. Für das Primer enthaltende Adhäsivsystem scheint ein höherer Druck während des Korundstrahlens vorteilhaft zu sein, um einen hohen und dauerhaften Klebeverbund zu Zirkonoxid-Keramik zu erzielen.

8. References

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10. Appendix

Material compositions

Name	Lot.No.	Composition	Manufacture
Katana HT10	DPQAY (BFS) DLUTX/DMRFH (TBS)	ZrO ₂ + HfO ₂ + Y ₂ O ₃ > 99.0%, yttrium oxide (Y ₂ O ₃) > 4.5% – ≤ 6.0%, hafnium oxide (HfO ₂) ≤ 5.0%, and other oxides ≤ 1.0%	
Clearfil FII New Bond	Base paste	2F0016	
	Catalyst past	2A0015	
Panavia V5	990010	Bisphenol A diglycidylmethacrylate 5-15%, triethylene glycol dimethacrylate <5%, silanated barium glass filler, silanated fluoroalminosilicate glass filler, colloidal silica, surface treated aluminum oxide filler, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, dl camphorquinone, initiators, accelerators and pigments	Kuraray Noritake, Tokyo, Japan
Clearfil ceramic primer plus	9B0006	Ethanol >80%, 3- trimethoxysilylpropyl methacrylate <5% and 10- Methacryloyloxydecyl dihydrogen phosphate (MDP)	
Panavia SA plus	7U0058	MDP, Bis-GMA, TEGDMA, Hydrophobic aromatic dimethacrylate, HEMA, Silanated barium glass filler, Silanated colloidal silica, dl, Camphorquinone, Peroxide, Catalysts, Pigments, Hydrophobic aliphatic dimethacrylate, Surface treated sodium fluoride, Accelerators	
Oxyguard II	1B0039	Glycerol 50-70% , polyethyleneglycol, catalysts, accelerators, dyes	

Dedication

To my father with his endless love and sacrifices.

To the soul of my mother, that always encouraging and pushing me in the way of success.

To my supportive aunts, sisters and brother without their love, care, help, patience and prayers, I would not have accomplished this work.

To all my friends whom encouraged and gave me a reason to go on and get this work out, wish them all the best forever.

To my lovely country

Yemen,

May ALLAH give y an overwhelmed lasting peace.