

BONDING ORTHODONTIC RESIN CEMENT TO ZIRCONIUM OXIDE UNDER ORTHODONTICS LOAD AND THERMOCYCLING EFFECT

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ABSTRACT

DOI: 10.25241/stomaeduj.2018.5(2).art.4

Purpose: Evaluate the microshear bond strength (μ SBS) of orthodontic resin cement to monolithic zirconium oxide ceramic (MZ) under orthodontic load (OL) and thermocycling (TC) effect.

Materials and Methods: Glazed MZ blocks (Zenostar, Ivoclar Vivadent) were tested after air abrasion with 30- μ m silica coated aluminum oxide (Al_2O_3) particles (CoJet, 3M ESPE). The specimens were randomly divided into 4 groups ($n = 15$): G1, OL with TC; G2, OL without TC; G3 no OL with TC; and G4, no OL, no TC (control). Orthodontic cement cylinders (Heliosit Orthodontic, Ivoclar Vivadent) were bonded to the primed samples (Monobond Plus, Ivoclar Vivadent) using the Ultradent SBS system and light cured (SmartLite Max, Dentsply Sirona, 1400 mW/cm², 40 s). G1 and G2 were subjected to 70 ± 15 N load perpendicular to the cylinder axis, G1 and G3 were thermo-cycled (5000 cycles 5-55°C, 90 s/cycle). G2 and G4 were stored in distilled water at $37 \pm 1^\circ C$. The specimens were subjected to μ SBS test (crosshead speed 0.5 mm/min). Data were analyzed using two-way ANOVA, and one-way ANOVA and Tukey test (HSD).

Results: Two-way ANOVA for μ SBS values (MPa) showed significant ($p = 0.0004$) load effects, but not thermal effect ($p = 0.2455$) with significant load/thermocycling interactions ($p < 0.0001$). The ranking of the single groups by Tukey test ($\alpha = 0.05$) showed that G1 exhibited the highest μ SBS (8.4 ± 2.8 MPa), G4 (6.3 ± 1.1 MPa) and G2 (5.8 ± 1.1 MPa) as a group was second, and G2 and G3 (4.7 ± 1.1 MPa) as a group was the lowest.

Conclusion: G1, which is the closest to clinical reality, yielded the best results.

Keywords: orthodontics, dental materials, orthodontic resin cement, monolithic zirconium oxide ceramic, microshear bond strength test.

1. Introduction

Due to the patients' increased esthetic demands and the development of technology in dental materials, porcelain fused to metal (PFM) crowns and bridges are being replaced by glass based ceramic materials and monolithic Zirconium oxide (MZ) [1]; they are more esthetic, biocompatible, resistant to wear, show low thermal conductivity, and are color stable [2]. However, despite these advantages, the brittle nature of these materials restricts their use [3].

Therefore, there is a need for new materials which have the same esthetic properties as glass based all ceramic materials and a strong framework like PFM for fixed dental prosthesis (FDPs). The introduction of zirconium oxide fulfilled these requirements [4]. The advantages of zirconium oxide include high fracture resistance and high flexural strength (> 1000 MPa), which allowed for thinner restorations. Furthermore the material can be stained which allows better esthetic results [5]. These properties make it a very good candidate for aesthetic FDPs. The tetragonal zirconia polycrystals (TZP), especially 3 mol % Y_2O_3 stabilized zirconia (3Y-TZP) has been used as a material for dental and medical

restorations [6]. Previous studies have reported that the life expectancy of (3Y-TZP) zirconium oxide when compared to PFM FDPs for posterior indication, is shorter because of the delamination and chipping of veneering ceramic; to overcome this problem, the monolithic zirconium oxide (MZ) was introduced to the dental market [1,2,4,7].

Full contour MZ FDPs are produced using CAD/CAM technologies. The restorations are milled from blocks which can be used either glazed or polished for better esthetic results [8]. Although polishing may provide sufficient esthetic appearance by decreasing the surface roughness, technicians like to glaze the ZrO_2 surface to improve the esthetic properties. With this process, the glass will infiltrate the zirconium oxide [9]. Since the number of adult patients who have been seeking orthodontic treatment is increasing [9], it means that the orthodontist will sometimes apply orthodontic brackets on dental restorations rather than on enamel. In a clinical situation, the orthodontist may not know the composition of all the ceramic crowns. Since different ceramics require different bonding procedures and the lack of bonding protocols for

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Peer-Reviewed Article

Citation: Hussein HS, Abdulhameed N, Shen C, Dolce C, Roulet J-F. Bonding of orthodontic resin cement to zirconium oxide under load and thermocycling. Stoma Edu J. 2018;5(2):102-108.

Academic Editor: Diana Ducea, DDS, PhD, Professor, "Iuliu Hatieganu" University of Medicine and Pharmacy Cluj-Napoca, Cluj-Napoca, Romania

Received: May 28, 2018

Revised: June 06, 2018

Accepted: June 11, 2018

Published: June 12, 2018

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these new materials, these situations may lead to early debonding of orthodontic brackets [4]. Furthermore, in orthodontics the goal is not a maximum bond strength, but one that is adequate to withstand orthodontic forces. Finally, the bond should be reversible i.e. it should be easy to remove brackets without damaging the enamel or the restored teeth.

Currently, there are several studies about the different surface conditioning protocols for orthodontic bonding to porcelain materials. They enhance the adhesion either by mechanical conditioning such as "roughness by airborne particle abrasion" or by chemical conditioning such as the use of hydrofluoric acid etch of glass-based ceramics to increase the bond strength and/or silane coupling agents or oxidic primers which change the wettability of the surface, or by combination of both mechanical and chemical surface treatments [10-12].

Although there is increased use for MZ crowns in dental practice, there is not enough information available about how to bond orthodontic brackets on MZ [13]. The most commonly method used to evaluate the performance of orthodontic bonding systems and the bonding technique is by measuring shear bond strength [14]. Doing this, one should consider the effect of orthodontic forces applied and the stress induced by water storage and thermocycling on the bond strength. This should be simulated in vitro as an accelerated ageing process [13]. In shear bond strength testing, the ideal direction of pull is parallel to the loading interface. It has been recognized that the direction of the debonding force will affect the results [15]. In clinical orthodontic practice, bonding the brackets and placement of arch wire might be done in the same visit. Hence, force could be applied to the bracket within the first hour after bonding and regardless of the relatively low magnitude of the force, it could have an adverse effect on the bond strength. It was reported that the polymerization of adhesives should quickly reach a minimum value to enable the adhesive to resist bonding failure when tying in initial arch wires [16].

The objective of this study was to evaluate the thermo cycling effect accompanied by orthodontic force on the micro shear bonding strength of orthodontic resin cement on glazed monolithic zirconium oxide surface conditioned by air abrasion with silica coated alumina particles.

The following null hypotheses were tested: 1) The thermo cycling (TC) does not influence the shear bond strength, 2) The orthodontic load does not influence the shear bond strength, and 3) The orthodontic load with TC does not influence the shear bond strength.

2. Material and methods

The types, brands, manufacturers and chemical composition of the material used in this study are listed in Table 1.

2.1. Specimen Preparation

One monolithic zirconium oxide (MZ) material was tested in this study (Zenostar, Ivoclar Vivadent, Schaan, Liechtenstein). The specimens were received in nonsintered blocks. They were cut into squares approximately (9 mm × 9 mm × 4 mm). They were

Table 1. Types, brands, manufacturers and chemical compositions of the material used in this study.

Type and Brand	Manufacturer	Chemical composition
Zenostar	Wieland, Ivoclar Vivadent	Zirconium dioxide (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 %, aluminum oxide (Al ₂ O ₃) + other oxides ≤ 1.0 %
Glaze spray	IPS e.max Ceram	Isobutane 30-60%, propan-2-ol 25-40%
Acratray Acrylic Powder (blue)	Henry Schein; Melville NY, USA	Poly Methyl methacrylate (PMMA), calcium carbonate, titanium dioxide, benzoyl Peroxide
Acrylic Liquid (Self cure)	Henry Schein	Methyl methacrylate (MMA), benzophenone, hydroquinone
CoJet Sand	3M ESPE; St Paul, MN, USA	30-µm Al ₂ O ₃ SiO ₂
Monobond Plus	Ivoclar Vivadent; Schaan, Liechtenstein	Ethanol, 3-trimethoxysilylpropyl methacrylate, methacrylate phosphoric acid ester, disulfide methacrylate
Heliosit Orthodontic Adhesive	Ivoclar Vivadent	Bis-GMA 50-100% urethane dimethacrylate 10 < 20% 1,10-decandiol dimethacrylate 10 < 20%
Orthodontic wire	ORTHO TECHNOLOGY	SS Straight Lengths 0.14.
Orthodontic brackets	Dentsply GAC international, INC Bohemia, NY, USA	Stainless Steel.

then sintered in a furnace at 1530°C (Sintramat S1 High Temperature Furnace, Ivoclar Vivadent) with a heating rate of 8°C/min and a holding time of two hours.

All the specimens were glazed with (IPS e.max Ceram glaze spray, Ivoclar Vivadent) according to the manufacturer's directions (770°C). The glazing material was applied in an even layer on the specimen in the usual manner and then all the specimens were fired according to the manufacturer's direction in a furnace (Programat EP 5000, Ivoclar Vivadent). After completion of the firing process the samples were removed from the furnace and allowed to cool to room temperature in a place protected from draft.

All specimens were then embedded in autopolymerizing acrylic resin (powder and liquid, Acratray Blue, Henry Schein, Melville, NY, USA). First, the specimens were held in place on a smooth surface with a piece of two-sided adhesive tape. Then, powder and liquid of acrylic resin was mixed (1:3) and poured into the molds to produce cylinders measuring 2.5 cm in diameter and 2.3 cm in length (Ultradent Products, South Jordan, UT, USA). After autopolymerization started, the mold was placed in a container with cold water to decrease the polymerization temperature.

After polymerization, the cylinders were removed from the mold and the two-sided adhesive tape was removed. The specimen surfaces were then cleaned with ethanol (Table 1). The cylinders were ground with 120-grit silicon carbide abrasive paper under running water for about 1 min to ensure a parallel surface to the bottom surface of the clamp into which the cylinders were placed during

bonding.

The specimens (N = 60), were randomly divided in two subgroups. Half of the specimens with orthodontic load (n = 30) and the other half without orthodontic load (n = 30). The orthodontic load and the non-orthodontic load specimens were further randomly divided into two subgroups: thermo cycling (TC) group and non-thermo cycling (non-TC) group (n = 15 per group) (Fig. 1).

2.2. Surface Conditioning Methods

All the specimen's surfaces were conditioned using air abrasion with an intraoral air-abrasion device (Microetcher, Danville Engineering, San Ramon, CA, USA) with 30 μm silica-coated Al₂O₃ (CoJet Sand, 3M ESPE, St Paul, MN, USA), perpendicular to the surface from approximately 10 mm for 20 s in circling motions at 2.8 bar. After air abrasion, the specimen surfaces were air blown to remove the remnants of the powder.

2.3. Bonding Procedures

Specimen surfaces were coated with a thin layer of Universal Primer (Monobond Plus, Ivoclar Vivadent) that was left for 60 seconds to allow it to react, and then the remaining excess was removed with a strong stream of air. Each specimen was fixed to a bonding clamp with a special mold (Ultradent Shear Bond Test, Ultradent Products, Inc., South Jordan, UT, USA) to assure flat substrate surfaces and to standardize the diameter (2.3 mm) of the resin composite. Orthodontic resin (Heliosit Orthodontic, Ivoclar Vivadent) was applied to the surface using the bonding mold. Composite was applied in the mold and light cured (SmartLite Max, Dentsply Sirona, York, PA, USA, 1400 mW/cm², 40 s) (Fig. 2). All specimens were stored in distilled water at 37 ± 1°C.

As next step, orthodontic brackets were bonded to the acrylic next to the embedded specimens of the load groups. The position was selected to apply a force of approximately 70 ± 15 g (0.69 ± 0.14 N) with an orthodontic wire (SS 0.14) (Fig. 3) to the bonded composite cylinders. The force was measured by using a Dontrix gauge (TP Orthodontics, Inc., La Porte, IN, USA). The orthodontic load group and the non-orthodontic load group were further randomly divided into two subgroups (n = 15): the thermo cycling (TC) group and the non-TC group (Fig. 1). Before testing the microshear bond strength all the specimens of the TC group were thermocycled in a Chewing Simulator device (CS-4SD Mechatronic GmbH, Feldkirchen, Westerham, Germany) for 5000 cycles between 5°C and 55°C with a dwell time of 30 seconds with the mechanical load component of the machine turned off. At the same time, all the specimens of the non-TC group were stored in distilled water at 37 ± 1°C. The position of the brackets to be bonded to the resin block is marked on both sides of the composite/MZ sample. The specimens were subjected to μSBS test using an universal machine (Instron 1125, Norwood, MA, USA, Fig. 4) (crosshead speed 0.5 mm/min).

2.4. Statistical Analysis

Means and standard deviations of the shear bond strength were calculated for all groups [9]. Microshear bond strength data (MPa) were submitted to a two-way ANOVA (SAS 9.4). Multiple comparisons were made using the Tukey's Studentized Range (HSD) Test (α =

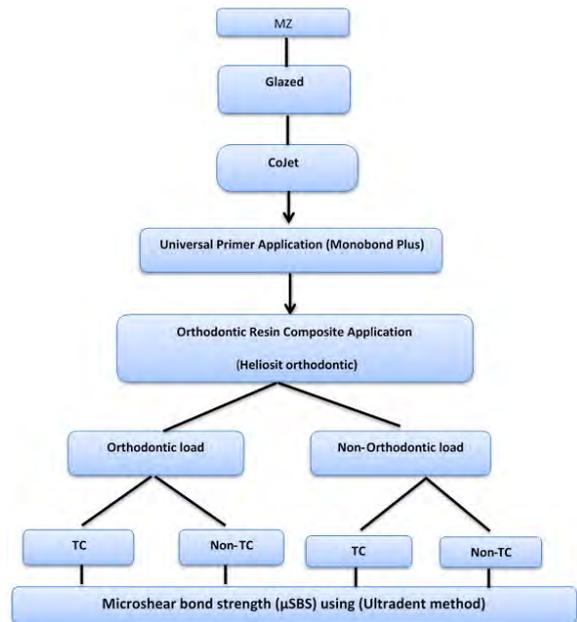


Figure 1. Experimental sequence, MZ= Monolithic Zirconium oxide ceramic, TC= Thermocycling effect.

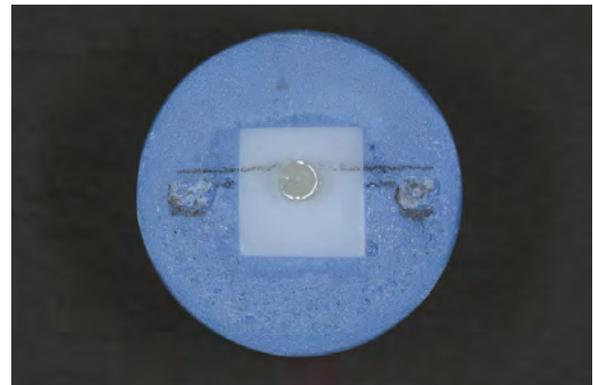


Figure 2. The Pencil line is tangent to the composite cylinder and it is 0.5 mm higher than the position of the bracket on the both side of MZ, in order to provide 70 ± 15 g (0.69 ± 0.14 N) load force by the orthodontic wire.

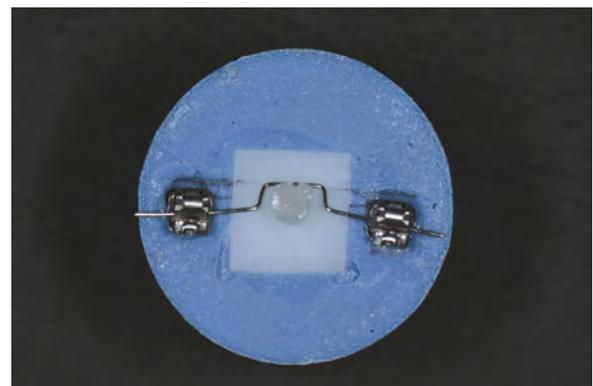


Figure 3. Top view showing the final design after adding orthodontic wire to composite cylinder.

0.05) for shear used to determine significant differences between the group dependent on the variable with and without application of load and/or TC.

3. Results

Two-way ANOVA for μSBS values (MPa) showed highly significant (p = 0.0004) effects, however highly significant load/thermocycling interactions were found (Tab.

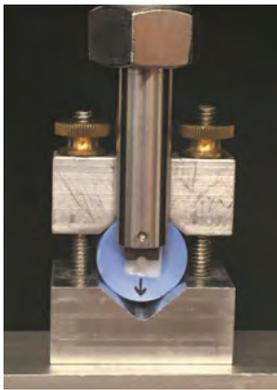


Figure 4. Sample loaded in universal Instron machine.

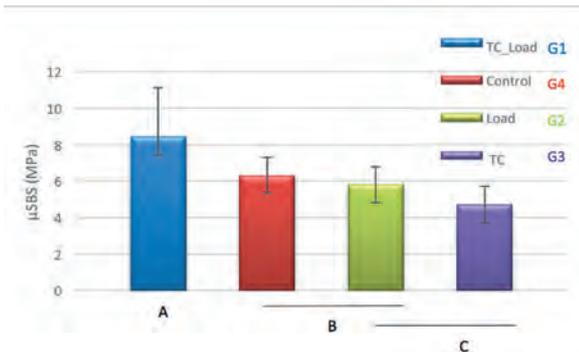


Figure 5. Means and standard deviations of μ SBS values between the TC and non-TC group with or without the orthodontic load. Bars connected with a line are in the same statistical group (Tukey test, $\alpha=0.05$).



Figure 6. The principle of CoJet treatment: the silica coated alumina particles create micro roughness of the surface due to the kinetic energy and leave silica embedded inside the surface, so it can react chemically with a primer (Silane) [21].

Table 2. Two-way ANOVA for μ SBS values (MPa). Note that the 2-way ANOVA showed highly significant differences (Model $p<0.0001$). However there were highly significant interactions (Load x Thermal).

Source	DF	Anova SS	Mean Square	F Value	Pr > F
Model	3	110.1178850	36.7059617	13.25	<.0001
Error	56	155.1204133	2.7700074		
Corrected Total	59	265.2382983			
Load	1	38.86540167	38.86540167	14.03	0.0004
Thermal	1	3.81528167	3.81528167	1.38	0.2455
Load +Thermal	1	67.43720167	67.43720167	24.35	<.0001

Table 3. Means and Standard deviations of the 4 tested groups. Same letters in the Tukey Grouping column mean no statistically significant difference.

TRET	N	Mean	STD	Tukey Grouping
TC_Load (G1)	15	8.4320	2.76480	A
Control (G4)	15	6.3180	1.09754	B
Load (G2)	15	5.8073	1.06452	B C
TC (G3)	15	4.7020	1.04792	C

2). Therefore, no main effects could be shown. The subsequent one-way ANOVA and the Tukey test ($\alpha = 0.05$) showed that thermal cycling with load (G1) had the greatest shear bond strength (8.4 ± 2.8 MPa) while the thermal cycling alone (G3) had the least shear bond strength (4.7 ± 1.1 MPa) among the groups. Comparing the results of μ SBS values for the adhesive system with and without the application of TC eliminates the effect of the load (G3, G4) and showed that there was significant difference between them and there was a significant difference with load (G1, G2). The result shows a nonsignificant reduction in μ SBS values between the load without TC effect (G2) and TC without load (G3) (Fig. 5).

4. Discussion

The first and third null hypotheses had been rejected, the second one had been accepted.

The conditioning technique used in this study (17), gritblasting with CoJet (Al_2O_3 silica coated sand) was selected for the following reasons: glass based ceramics can be etched with Hydrofluoric acid (HF) to achieve an excellent micromechanical surface topography for bonding. On the other hand, ZrO cannot be etched with HF at room temperature at all. Sandblasting with CoJet works well on glass-based ceramics as well. Since the orthodontists do not know which ceramic has been used, with the method used they are on the safe side. It would be the protocol of choice in the clinical routine [17].

Due to increased esthetics most MZ FDPs are glazed or stained, according to the study of Canigur et al. [4] "the CoJet yields higher bond strength values. The CoJet creates micro retentive sites by increasing surface area and roughness" [18-20] "silica coated particles not only roughen the surface, they also have a chemical effect: because of blasting pressure, the embedded silica and alumina particles can then chemically react with the silane coupling agent" (Fig. 6) [21]. "The improved chemical bonding with silane coupling agents in this approach is advocated to be the key factor for a higher resin bond strength."

Since the orthodontist does not know the details of the fabrication process of the crown where a bracket should be bonded to, it is better to consider the ZrO surfaces were all glazed. With the gritblasting the glaze may be partially or totally removed but using CoJet. A silica layer is deposited regardless of the composition of the underlying surface, thus allowing the use of Silane as a primer.

Spontaneous debonding of brackets is one of the most common clinical problems in fixed orthodontic therapy. There are two major interfaces that can be subjected to debonding: the enamel or restorative material/adhesive interface and the adhesive/bracket interface [22]. In this study, we tested the restorative material (ZrO)/adhesive interface and it was decided not to use brackets for the following reasons: The shear bond strength between orthodontic cement and the ceramic surface was the only topic of interest. If we had used brackets we would have had to deal with the bracket-cement interface as well, which was investigated in the past abundantly [23-25]. Furthermore, it was necessary to eliminate possible confounding factors such as geometry, mesh design of the bracket base, or bracket material, all of

which may influence the test results [23-25]. Therefore, we opted for a shear bond test which is the closest to the clinical reality. We assume that the main reason for bracket failure is shear. The shear bond strength test was performed with an orthodontic resin composite cylinder in order to eliminate tilting moments as much as possible. However, we understand elastic and plastic deformation of the cylinder may have some negative effects on the shear bond strength measured. We are well aware that in orthodontic treatment brackets transmit forces to the teeth in all directions, however unexpected debondings usually occur under shear. The brackets used in this study were used to apply shear forces to the bonded cylinders and were attached to the resin-embedding material (Fig. 3).

Thermocycling is a standard procedure for accelerated ageing in bond strength tests in vitro. In this study, all the specimens of the TC group were thermocycled in a chewing simulator device for 5000 cycles between 5°C and 55°C with a dwell time of 30 seconds with the mechanical load component of the machine turned off. At the same time, all the specimens of the non-TC group were stored in distilled water at $37 \pm 1^\circ\text{C}$. Extensive water storage and thermal cycling seem to be important parameters to simulate intraoral conditions and to stress bonding interfaces. Literature data show that thermocycling had a much higher impact on the durability of the resin bond strength to zirconia than did water storage at a constant temperature alone [26]. Loading the samples represented the clinical reality. In clinical orthodontic practice, bonding of brackets and placement of the arch wires can be done in the same visit, particularly after rebonding of debonded brackets. Hence, force could be applied to the bracket within the first hour after bonding. This force could affect polymerization of the orthodontic adhesive and subsequently its bond strength.

The force magnitude used for orthodontic tooth movements varies depending on the type of movement [16]. In this study, $70 \pm 15 \text{ g}$ ($0.69 \pm 0.14 \text{ N}$) was applied. This force is considered the optimal orthodontic force. In the clinical situation the average force transmitted to a bracket during mastication was reported to be 40 to 120 N, the surface area of the bracket is approximately 11.9 mm^2 and therefore it should be able to resist stress values between 6 and 8 MPa [16], during fixed orthodontic treatment for clinical success [27]. The mean bond strength values of brackets bonded to natural teeth are significantly lower than those obtained for surfaces other than enamel, especially when a chemical promoter such as silane is used during bonding [28]. These results may imply that universal primer application alone, prior to bonding, would already enhance the bond strength of the orthodontic resin composite tested. However, it is known that in vitro bond strength values are often higher than in clinical situations, and biodegradation of resin composites in the oral environment over time may even decrease these values [29,32]. In addition, aging procedures have a detrimental effect on the bond strength values of resin composites when compared to non-aging test conditions. [22,30,31].

As reported previously, long-term (two-year) water storage [25] or 6000 thermocycles [20] decreased the

bond strength of bis-GMA resin composites, regardless of air abrasion or silica coating and silanization. Hence, because no long-term aging was performed in this study, the results should be carefully and critically evaluated, considering that bonding in orthodontics is semi-permanent. However, at least 24-month water storage should be preferred for orthodontic bond strength testing [32], since it is usually the average period for complete orthodontic treatment with fixed appliances, and significant decreases may be expected under such prolonged aging conditions compared to shorter durations of only a few days to several months [30,33]. The bond strength is affected by aging only when a mechanical pre-treatment is not applied prior to an MDP-containing primer [34]. Wegner and Kern [25] also showed that the bond strength of an MDP-containing resin composite did not change significantly after aging [25]. For this reason, the use of universal primers containing methacrylate phosphoric acids and silane should be preferred after air-abrasion protocols. The results (Fig. 5) were not as anticipated. All samples of the present study were stored in water for the same time (2 weeks); the only difference is that some groups were subjected to additional stress (Load or TC) of the interface before being subjected to shear stress to failure. One could assume that additional stress would weaken the interface. This only happened for TC, which confirms the trend in other studies. However direct comparisons are difficult or problematic, because of the different methods used in different studies. The Kiel group has extensively looked into the bond strength of composites to ceramics, especially zirconium oxide ceramic. However they use tensile strength as a testing method and very long water storage (up to 2 years) and high numbers of TC (37,500) [35-38]. As a general trend they found for most adhesive techniques that water storage with or without TC has a negative effect on bonded interfaces.

There are a few studies that use shear or microshear bond strength. When looking at the bond strength of surfaces that have been silicized (Rocatec, CoJet or similar procedures) water storage and TC also decreased the shear bond strength [20,39]. On the other hand, Lüthy et al. [40] found for some bonding procedures no significant differences between TC and water storage and water storage alone).

Since the load applied corresponded to the load that is usually used in the clinic, one can expect that it has little effect on the bond strength, which confirms the clinical observations (few debondings of brackets). This explains that the control and load group showed the same shear bond strength. Since there are no other publications that have subjected samples to orthodontic load, it is not possible to compare the results of the present study with others.

It was surprising to see that the group with orthodontic load in combination with TC showed the best results, which is difficult to explain. It seems that there is a synergetic effect of load and TC. It is known that, due to polymerization shrinkage, stress will be build up at the interface. This stress can be slightly increased in one direction by the orthodontic load. Under TC cyclic dimensional changes are induced, which may damage the interface by creating additional stress

and thus crack induction points. One may hypothesize that under slight load the internal stress relaxation that happens within the material is enhanced and thus stress induction points may be reduced. In a clinical situation the crown material is not known to the orthodontist. The procedure used will work with any ceramic. Therefore, the proposed protocol is a safe approach.

5. Conclusion

Based on the results of this in vitro study obtained with Cojet, Monobond Plus and Heliosit Orthodontic and considering the limitations of in vitro results one can conclude that

- Under OL and TC, the highest bond strength was observed.
- Load or TC alone yielded similar or lower shear bond strengths.
- Sandblasting with CoJet and using Monobond Plus as primer is a good protocol for bonding to ZrO crowns in orthodontic therapy.

Therefore, it is recommended for future in vitro tests of adhesives for orthodontic purposes to test under OL and TC.

Author Contributions

HSH: Performed experiment, wrote manuscript. NA: Performed experiment, proofread manuscript. CS: Data analysis, proofread manuscript. CD: Consulting of orthodontic aspects of experiment, proofreading manuscript. JFR: Idea, experimental design, proofread manuscript.

Acknowledgments

The authors declare no conflict of interest related to this study. There are no conflicts of interest and no financial interests to be disclosed.

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Questions

1. Hydrofluoric acid is used to increase the surface roughness for bonding for the following ceramics?

- a. Zirconium Oxide;
- b. Lithium disilicate ceramic only;
- c. All glass based ceramics;
- d. Leucite reinforced ceramic only.

2. The treatment with Co-jet sand provides the following?

- a. Roughens the surface;
- b. Roughens the surface and deposits a layer of silica;
- c. Cleans the surface;
- d. Polishes the surface.

3. Which surfaces can be primed with silane?

- a. Base metals;
- b. Gold alloys;
- c. Oxide ceramics e.g. Zirconium oxide;
- d. Surfaces containing silica, e.g. glass based ceramics.

4. Which is the ideal clinical protocol to bond to an all ceramic crown made out of an unknown ceramic?

- a. Roughen surface with Co-jet, prime with Silane and use resin based orthodontic cement;
- b. Etch with hydrofluoric acid, prime with silane and use resin based orthodontic cement;
- c. Etch with phosphoric acid, use universal primer and use resin based orthodontic cement;
- d. Roughen surface with Aluminumoxide sand, use silane and cement bracket with glass ionomer cement.