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# The Effect of Different Decontamination Methods on the Micro-Tensile Bond Strength of CAD/CAM Resin-Based Blocks to Resin Cement

## Keywords

*Resin Cement*
*Resin Blocks*
*CAD/CAM*
*Decontamination Methods*
*Micro-tensile Bond Strength*
*Saliva and Blood Contamination*

## Authors

Carlos Soler-Tornero *
*(DDS, PhD)*

Mustafa M. Mutluay § ‡
*(DDS, PhD)*

## Address for Correspondence

Carlos Soler-Tornero *
Email: carlos.soler.tornero@uef.fi

- * *Department of Cariology and Endodontics, Institute of Dentistry, University of Eastern Finland, Kuopio, Finland*
- § *Department of Prosthodontics and Clinical Dentistry, Institute of Dentistry, University of Eastern Finland, Kuopio, Finland*
- ‡ *Department of Restorative Dentistry and Cariology, Adhesive Dentistry Research Group, Institute of Dentistry, University of Turku, Turku, Finland*

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## ABSTRACT

*Introduction: This study evaluates the impact of decontamination methods on Micro-tensile bond strength (μTBS) between resin cement and resin blocks. Methods: Twenty CAD/CAM resin blocks from two manufacturers were wet-polished, sandblasted, and ultrasonically cleaned. After blood and saliva contamination, blocks were divided into subgroups: control, water rinsing, acid etching, alkaline cleaning paste, or 10-MDP containing cleaner. Resin-based cement was then applied. After 24 hours, the blocks were sectioned to obtain bars for testing. Half of the specimens were tested immediately for μTBS, and the other half underwent artificial aging. The surfaces of the blocks were inspected with a scanning electron microscope (SEM). Three-way ANOVA was performed for μTBS values ($\alpha$=0.05). Results: In one of the substrates, the positive control subgroup obtained the highest value (56,01 MPa, SD:6,96) followed by 10-MDP cleaner and universal cleaning paste, when immediately tested, with significant differences respect to the water rinsing ($p<0.041$) and acid etching ($p<0.048$) groups. After thermocycling, higher values were found in the 10-MDP cleaner (47,57 MPa, SD:8,15), but differences were not significant. In the other substrate group, the 10-MDP cleaner subgroup showed highest bond strengths (64,46 MPa SD: 10,92) at the initial test. After thermocycling, 10-MDP cleaner (58,66 MPa, SD: 9,93) gave the highest μTBS value. Significant differences between water rinsing group and the rest of subgroups ($p<0.001$), and between 10-MDP cleaner and the positive control group ($p<0.006$) were observed. Conclusion: Cleaning after contamination improves bonding. 10-MDP containing cleaner can help to restore initial μTBS value and maintain it in the long-term.*

## INTRODUCTION

Chairside computer-aided design/computer-aided manufacturing technology (CAD/CAM) is more accessible to the clinician, leading to an increased use of indirect restorations in general practice.[1,2] CAD/CAM blocks can be fabricated from various materials, including lithium disilicate reinforced glass, leucite-reinforced glass, feldspathic glass, aluminum oxide, yttrium tetragonal zirconia polycrystal, and titanium.[3,4] Aesthetic demands in dentistry have increased, and now aesthetic restorations are not only

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limited to the anterior teeth but also to the molars. This is one of the main reasons, along with the reduced cost, why resin-based CAD/CAM blocks have been introduced.[4,5]

Despite the ongoing improvement of resin-based materials, ceramic restorations continue to be the preferred choice in dental applications, since they have better aesthetic, optical and mechanical properties.[3,6] On the other hand, resin-based materials exhibit properties that closely resemble natural teeth in terms of flexural and compression properties, wear on opposing enamel, and resistance to fracture.[1,7,8] From an economic and time-saving perspective, there are some advantages, such as the short milling times, possibility of placing the restoration in a single visit[9] and the longer service life of the burs used to mill the block.[10] Other characteristics make resin-based materials appealing. Resin-based materials are not only easier to polish and repair but also exhibit improved milling damage tolerance, enabling faster milling speeds and better marginal quality.[9,11]

However, despite the mentioned desirable properties of resin-based CAD/CAM materials, they present some disadvantages. Due to the high degree of resin polymerization, it could be challenging to achieve a strong bond between the tooth and the restoration.[4,12] The indirect restoration also increases the number of surfaces to bond to, being one interface at the tooth structure and the other at the surface of the restoration.[1] Sandblasting, surface roughening, and silane application are some of the techniques that have been proposed for increasing the bonding strength. While there seems to be a consensus that sandblasting has a positive impact on the bonding process,[13-15] the benefits of silane use are still unclear, and it seems to have some drawbacks.[15]

Moreover, aside from the complexity of resin adhesion, the need for a try-in before final cementation adds another step that can interfere with the quality of the bond, since fluid contamination is difficult to avoid.[16] Saliva, blood, exudate, plasma, temporary bonding or sealing material can contaminate the bonding surfaces, affecting the bond strength negatively.[17-19] The positive effect of acid etching after saliva contamination has been tested before.[20,21] Also, other cleansing techniques, such as additional airborne particle abrasion or an extraoral cleaning paste (Ivoclean, Ivoclar Vivadent AG, Schaan, Liechtenstein), had been widely studied for the decontamination of the restorations and accepted as effective cleaning methods.[22] A new product, the KATANA cleaner (Kuraray Noritake, Tokyo, Japan), containing MDP salt, is an alternative to those, and it has the advantage that it can be used intraorally.[5,23]

Given the limited research in this domain, the effectiveness of KATANA cleaner in restoring bond strength after saliva and blood contamination of resin-based indirect restorations remains unclear. The purpose of the present study was to assess the immediate and long-term effects in the bonding strength of different cleaning techniques on CAD/CAM resin blocks. Since not all the methods are suitable for intraoral use, we focused on their influence on the restoration surface-cement interface. The bond strength was assessed using the micro-tensile bond strength (μTBS), since many authors have previously suggested that is one of the most reliable tests when measuring bond strength in laboratory studies, and it should be preferred over other similar tests.[14,24] The null hypotheses were: (1) Blood and saliva contamination would not influence the obtained bond strength (μTBS) values; (2) the type of decontamination method would not affect the results of the bond strength test (μTBS); (3) and thermocycling does not have an impact in bond strength.

## MATERIAL AND METHODS

Twenty CAD/CAM resin blocks were selected for this study and divided into two groups: one of them consisting of ten Tetric CAD for CEREC and inLab MT A3/C14 (Ivoclar Vivadent AG, Schaan, Liechtenstein) blocks (T), and the conformed of ten KATANA Avencia Block Cerec A3 12 (Kuraray Noritake, Tokyo, Japan) blocks (K). Table 1 shows the manufacturers and composition of the materials used in the study.

The contact surface of each block was wet-polished using 360 and 600-grid silicon carbide abrasive paper (CarbiMet, Buehler Ltd, Lake Bluff, IL, USA) on a polishing machine (Buehler EcoMet 30, Buehler Ltd, Lake Bluff, IL, USA). After the initial polishing, the samples were sandblasted with 50 μm alumina particles in a dust cabinet (RØNVIG Dental Mfg. A/S, Daugaard, Denmark) at 0,15 MPa pressure for 20 seconds at 10 mm from the surface. The specimens were then ultrasonically cleaned in an ultrasonic unit (BioSonic UC 125, Coltène/Whaledent GmbH + Co. KG, Langenau, Germany) with ethanol for 5 minutes then rinsed with water and air dried using a three-way syringe.

Before the application of the Multilink Automix (Ivoclar Vivadent AG, Schaan, Liechtenstein) cement, the blocks were subdivided into five groups (2 blocks of K group and 2 blocks of T group per treatment) and treated in one of the following ways:

- T1 positive control group: no contamination.
- T2 negative control group: contamination by immersion in a mixture consisting of saliva and blood for 60 s, rinsed with water for 10 s using a 3-way syringe, and air dried for 10 s.
- T3 acid etching control group: by immersion in a mixture consisting of saliva and blood for 60 s, rinsed with water for 10 s, air dried for 10 s, etched with 37 % phosphoric acid (Total etch, Ivoclar Vivadent AG, Schaan, Liechtenstein) for 20 s, rinsed with water for 10 s using a 3-way syringe and air dried for 10 s.
- T4 Ivoclean treated group: by immersion in a mixture consisting of saliva and blood for 60 s, rinsed with water for 10 s, air dried for 10 s, the entire bonding surface of the restoration was covered with a layer of Ivoclean (Ivoclar Vivadent AG, Schaan, Liechtenstein) using a micro brush and after 20 s was rinsed with water for 10 s and air dried for 10 s.

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**Table 1.** Materials used in the study.

| Material | Composition | Manufacturer |
|---|---|---|
| **Tetric CAD** | Bis-EMA, Bis-GMA, TEGDMA, UDMA, barium glass (<1 µm), silicium dioxide (<20 nm). | Ivoclar Vivadent |
| **KATANA avencia** | Mixed filler with colloidal silica (Ø40 nm) and aluminum (Ø20 nm) oxide, Cured resins consisting of methacrylate monomer (Copolymer of Urethane dimethacrylate and other methacrylate monomers), Pigments. | Kuraray Noritake Dental |
| **Multilink Automix** | The monomer matrix is composed of Dimethacrylate and HEMA. The inorganic fillers include barium glass, ytterbium trifluoride and spheroid mixed oxide. The particle size is 0.25–3.0 µm. The mean particle size measures 0.9 µm. The total volume of inorganic fillers is approximately 40 % | Ivoclar Vivadent |
| **Multilink A and B primer** | Multilink Primer A is an aqueous solution of initiators. Multilink Primer B contains HEMA, phosphonic acid and methacrylate monomers. | Ivoclar Vivadent |
| **Total Etch** | 37% phosphoric acid | Ivoclar Vivadent |
| **KATANA cleaner** | Water, 10- MDP, Triethanolamine, Polyethylene glycol, Stabilizer, Dyes. | Kuraray Noritake Dental |
| **Ivoclean** | Zirconium oxide, Water, Polyethylene glycol, Sodium hydroxide, Pigments, additives. | Ivoclar Vivadent |

**Bis-EMA: bisphenol-A-polyetheylene glycol dimethacrylate; Bis-GMA: bisphenol-A-diglycidyl dimethacrylate; HEMA: 2-Hydroxyethyl methacrylate; TEGDMA: triethyleneglycol dimethacrylate; UDMA: urethandimethacrylate; 10-MPD; 10-Methacryloyloxydecyl dihydrogen phosphate.**

- T5 KATANA cleaner treated group: by immersion in a mixture consisting of saliva and blood for 60 s, rinsed with water for 10 s, air dried for 10 s, KATANA cleaner (Kuraray Noritake, Tokyo, Japan) was applied and rubbed in the entire bonding surface of the restoration using a micro brush during 10 s, rinsed with water for 10 s and air dried for 10 s.

A mixture consisting of saliva (10 ml) and blood (5 ml) in 2:1 proportion was used for the contamination of the surfaces. The saliva was non-stimulated and obtained from one healthy male donor (principal investigator) who had refrained from eating or drinking for two hours before the collection process. The blood was also from the same donor, and it was fresh blood obtained from the needle-punctured fingertip using a 28 G needle (Vitrex Sterilance Press II, Vitrex Medical A/S, Herlev, Denmark). The specimens were immersed in the saliva and blood mix at 37°C for 60 s. According to the Committee on Health Research Ethics at the University of Eastern Finland, specific approval was not necessary since blood and saliva were obtained only from the main researcher and their purpose was no other than only simulate a laboratory agent.

After the contamination and cleaning of the blocks, and prior to cement build-up, Multilink A+B primer (Ivoclar Vivadent AG, Schaan, Liechtenstein) was mixed on a 1:1 proportion and applied to the surface of the block by scrubbing it for 30 s followed by air drying until no mobile liquid was visible, according to the product's instructions for use. Then, the resin-based cement was built up to 6 mm in 2 mm increments following the manufacturer's instructions and cured using Elipar DeepCure-S LED Curing Light (3M Oral Care, St. Paul, MN, USA) with a light intensity of 1,470 mW/cm$^2$ (-10%/+20%). After that, the blocks were stored in distilled water at 37°C for 24 h in an incubator (MELAG INCUBAT 85, MELAG Medizintechnik oHG, Berlin, Germany). Figure 1 summarizes the steps involved in the bonding surface preparation, contamination, cleaning, and luting procedures.

The next day, each block was divided into beams of 0,7 mm x 0,7 mm x 10 mm (+- 0,2 mm) by slow-speed water-cooled saw (IsoMet, Buehler Ltd, Lake Bluff, IL, USA) using a 102 x 0,3 mm 15 HC diamond wafering blade (Buehler Ltd, Lake Bluff, IL, USA), obtaining about 49 beams with an area of approximately 0,5 mm$^2$ at the bonding surface. The cut started at the cement side through the resin block perpendicular to the bonding surface. Then, the block was rotated 90° and again cut. Finally, to obtain the slabs, the block was cut at their base, parallel to the bonded surface.

The 98 resulting microbars of each surface treatment (49 x 2) were visually inspected, and the specimens with defects at the interface level or those clearly defective (cracks, big size variations, bubbles affecting the cement or the interface, etc.) were discarded. The beams from the outermost sides of the blocks were also excluded. From the remaining specimens, 24 beams were randomly selected for the initial µTBS group (I) and immediately tested, and 24 were put in the thermo-cycled µTBS group (TC). They were later subjected to tests after 5000 thermo-cycles between 5 – 55°C, with 30 seconds dwell time, using a thermo-cycle tester (Proto-tech, Portland, OR, USA). The specimen number was determined based in previous studies using similar methodology.[4,5,25]

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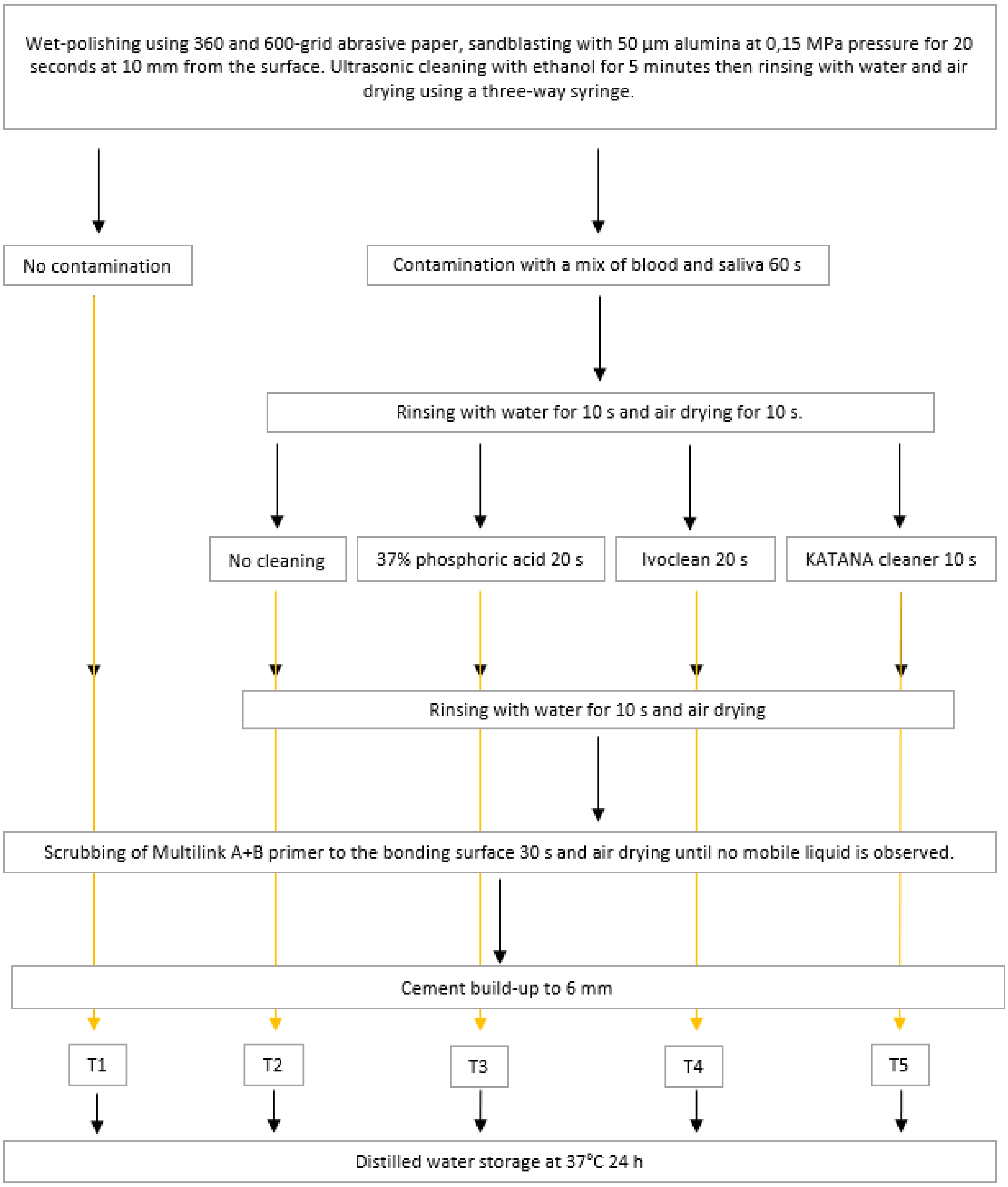

**Figure 1:** block pre-treatment, contamination, cleaning, and luting procedures.

For the measure of the micro-tensile bond strength, first, the exact thickness and width of the bonded area of each specimen were measured using a digital caliper. Then the specimens were glued with cyanoacrylate (Loctite 416, Henkel AG & Co. KgaA, Düsseldorf, Germany) to a stainless-steel jig, and tested using a universal testing machine and a compact force gauge (Mecmesin CFG+ 500N, PPT Group UK Ltd, West Sussex, United Kingdom) with a cross-section speed of 1 mm/min until fracture. The µTBS value was expressed in megapascals (Mpa) and was obtained individually for each microbar dividing the imposed force (N) at the time of fracture by the bonded area ($mm^2$). The failure modes were evaluated at 2x magnification using loupes (Optergo Ultralight flip-up, Merident, Lohja, Finland). The color of the luting agent was selected to improve the contrast of the resin-cement interface, thus leading to an easier identification of the fracture location. They were classified as an adhesive failure at the interface (A), cohesive failure within the cement (B) or the resin block (C).

Also, a 2 mm thickness slice from the remaining material of each block was cut and again wet-polished using 360 and 600-grid silicon carbide abrasive paper, sandblasted with 50 µm alumina particles, ultrasonically cleaned and rinsed with water and air dried using a three-way syringe. The slices were also contaminated and cleaned following the protocol described before. Finally, samples were gold sputter-coated and examined under scanning electron microscope (SEM) at different magnifications using Phenom ProX (Phenom-World, Eindhoven, The Netherlands) operating at 15 kV to evaluate the surface of the material at 1000x, 2000x, 5000x and 10000x magnification. Figures 2 and 3 illustrate the experimental procedures of the study.

For the statistical analyses, SigmaPlot for Windows version 14 (Systat Software Inc, Palo Alto, CA, USA) was used. Three-way variance analysis (ANOVA) followed by multiple comparisons using One-way ANOVA were used to analyze the µTBS tensile strength mean values ($p<0.05$).

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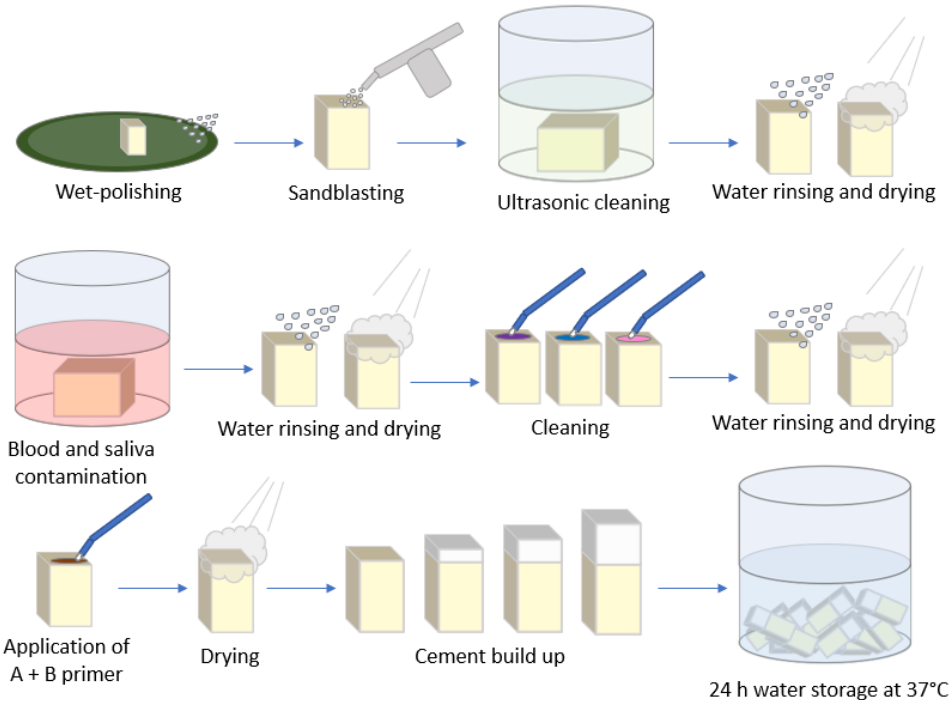

**Figure 2:** illustration of the bonding surface preparation, contamination, cleaning, and luting procedures.

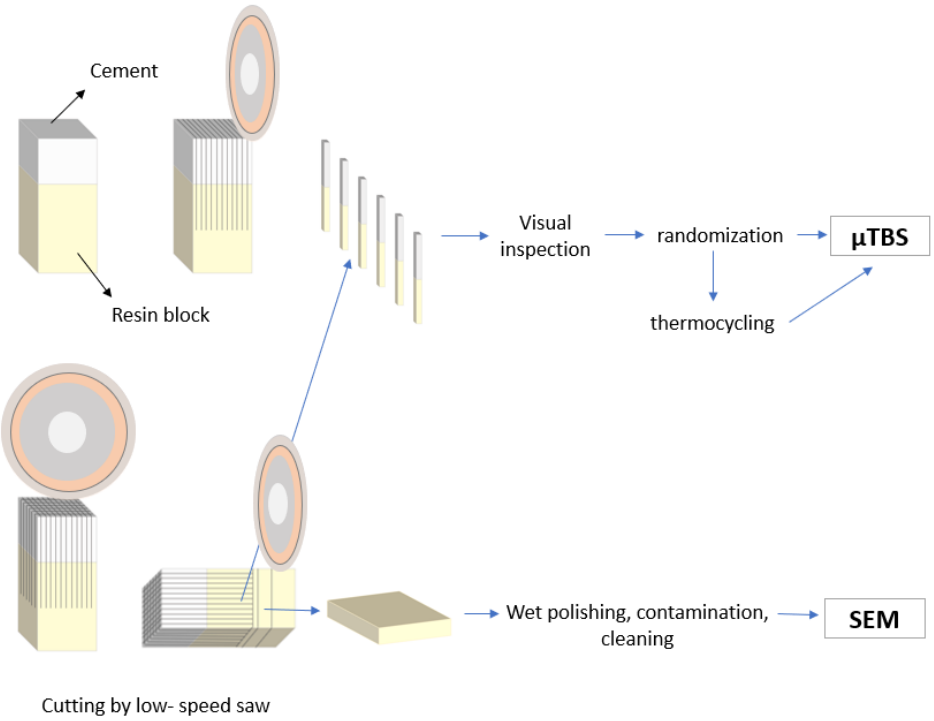

**Figure 3:** cutting of the blocks using low-speed saw to obtain beams for the µTBS test and pieces for SEM analysis.

# RESULTS

## SURFACE OBSERVATION

SEM revealed that scars and debris were found after resin blocks were polished and sandblasted *(Figure 4)*. Sandblasting removed the most part of the debris and increased surface roughness. The surface of T group specimens showed a more homogeneous surface, with small micropores, compared to those of the K group. In addition, the presence of debris was more common in the K group. No major morphological differences between blocks of different treatment groups of the same substrate were noticeable.

## MICRO-TENSILE BOND STRENGTHS

The mean µTBS and standard deviations are illustrated in Figure 5. Data passed the normality test Shapiro-Wilk (p= 0,517) and the equal variance test Brown-Forsythe (p= 0,193). The results of the

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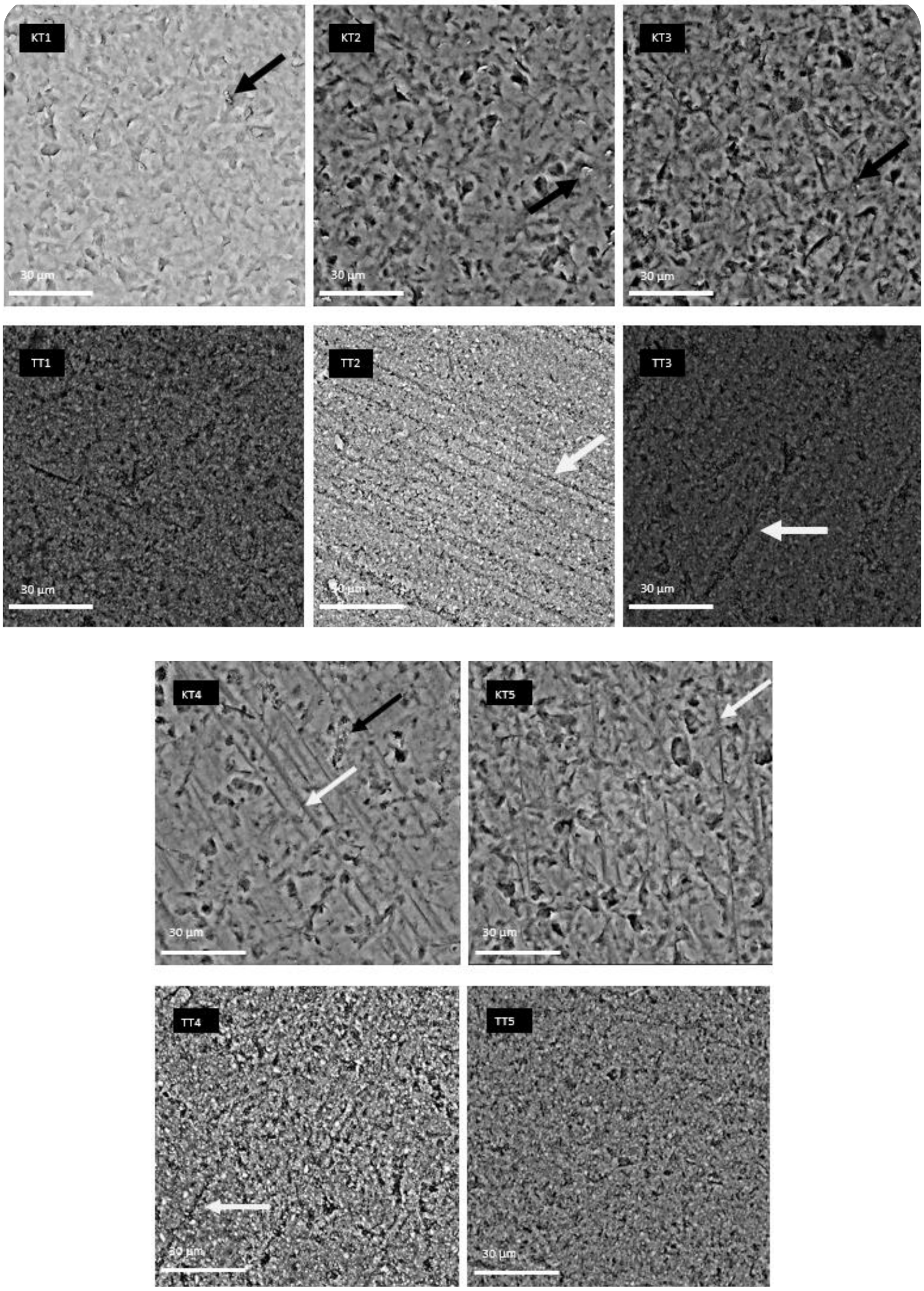

**Figure 4:** 2500x representative SEM images of the surface of the specimens after the surface treatment, contamination, and cleaning protocols. Microstructures differences between substrates can be noticed. The presence of small debris (black arrows) after polishing and sandblasting procedures is more frequently seen in the K group. Polishing scars (white arrows) can be appreciated in both groups. KT1: KATANA avencia control subgroup, KT2: KATANA avencia no cleaning subgroup, KT3: KATANA avencia acid etching subgroup, KT4: KATANA avencia Ivoclean subgroup, KT5: KATANA avencia KATANA cleaner subgroup. TT1: Tetric CAD control subgroup, TT2: Tetric CAD no cleaning subgroup, TT3: Tetric CAD acid etching subgroup, TT4: Tetric CAD Ivoclean subgroup, TT5: Tetric CAD KATANA cleaner subgroup.

three-way analysis of variance are shown in Table 2. Three-way ANOVA revealed a significant effect for the parameters “aging” ($p<0.001$, $F=109$), “substrate” ($p<0.001$, $F=87$) and “treatment” ($p<0.001$, $F=27$). The effect of “aging” does not depend on “substrate” ($p = 0{,}865$), but there is a statistically significant interaction between “aging” and “treatment” ($p= 0{,}003$). There is also a statistically significant interaction between the type of “substrate” used and the “treatment” ($p<0{,}001$). In the T2 group, the type of substrate does not have an impact on the bond strength ($p= 0{,}063$). Micro-tensile bond strength values in the T group were significantly higher than those in the K group ($p<0.001$). After thermocycling, all subgroups but KT5 presented significantly lower µTBS values ($p<0{,}05$).

Multiple comparisons using one-way ANOVA revealed that, in the K group, KT1 achieved the highest micro-tensile bond strength at the initial measurement (56,01 MPa), followed by KT5 (51,29 MPa), while KT2 and KT3 showed the lowest values

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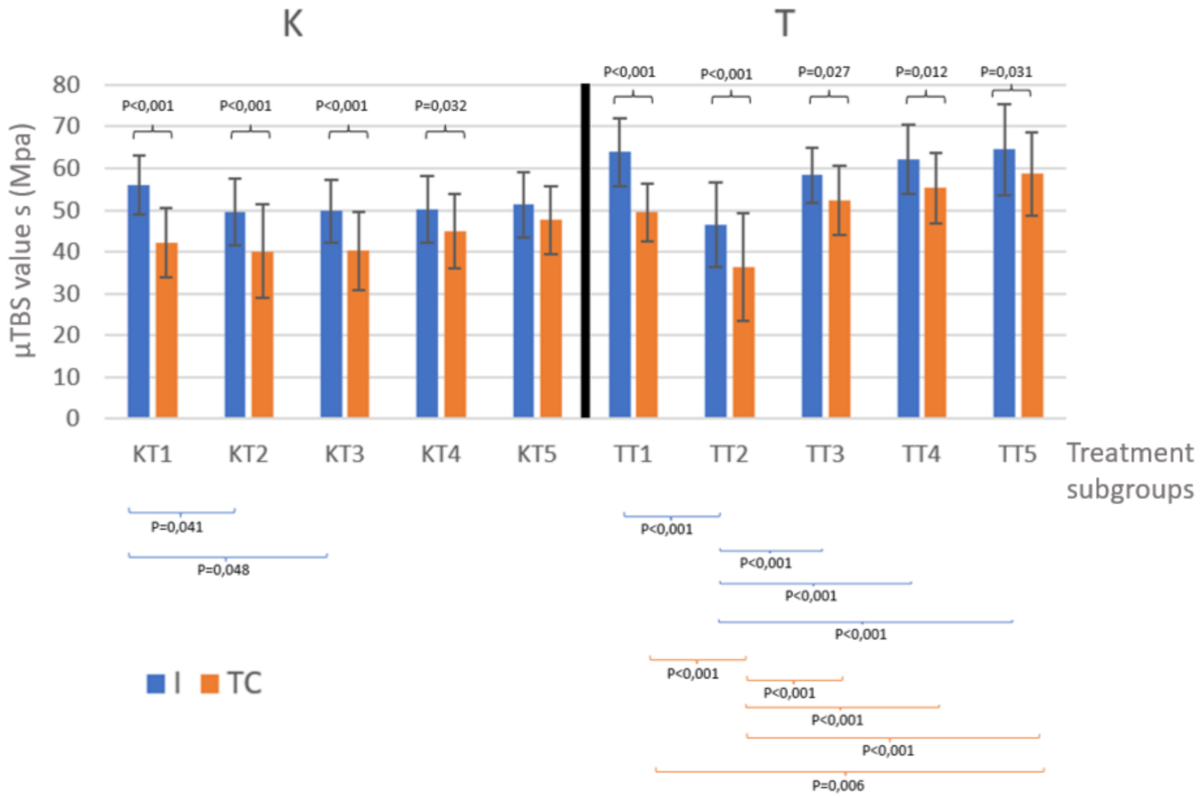

**Figure 5:** µTBS results of the KATANA Avencia (K) and Tetric CAD (T) groups and their standard deviations. Columns connected by a brace indicate a significant difference (a<0.05). I: initial, TC: after 5000 cycles of thermocycling. K: KATANA avencia group, T: tetric CAD group. KT1: KATANA avencia control subgroup, KT2: KATANA avencia no cleaning subgroup, KT3: KATANA avencia acid etching subgroup, KT4: KATANA avencia Ivoclean subgroup, KT5: KATANA avencia KATANA cleaner subgroup. TT1: Tetric CAD control subgroup, TT2: Tetric CAD no cleaning subgroup, TT3: Tetric CAD acid etching subgroup, TT4: Tetric CAD Ivoclean subgroup, TT5: Tetric CAD KATANA cleaner subgroup.

**Table 2.** Results of the three-way ANOVA test. DF: degrees of freedom; SS: sum-of-squares; MS: mean squares; F: F ratio; P: p-value.

| Source of Variation | DF | SS | MS | F | P |
|---|---|---|---|---|---|
| **Aging (A)** | 1 | 8632,468 | 8632,468 | 109,293 | <0,001 |
| **Substrate (B)** | 1 | 6887,879 | 6887,879 | 87,206 | <0,001 |
| **Treatment (C)** | 4 | 8670,206 | 2167,552 | 27,443 | <0,001 |
| **A*B** | 1 | 2,292 | 2,292 | 0,029 | 0,865 |
| **A*C** | 4 | 1307,912 | 326,978 | 4,14 | 0,003 |
| **B*C** | 4 | 3872,571 | 968,143 | 12,257 | <0,001 |
| **A*B*C** | 4 | 114,913 | 28,728 | 0,364 | 0,834 |
| **Residual** | 460 | 36332,81 | 78,984 | | |
| **Total** | 479 | 65821,05 | 137,413 | | |

(49,54 MPa and 49,75 MPa respectively). Significant differences in micro-tensile bond strength were observed between the KT1 and KT2 (p=0,041), and KT1 and KT3 (p=0,048). After aging, higher values were observed in KT5 (47,57 MPa) and KT4 (44,87 MPa) subgroups, but the differences were not significant (P= 0,065).

In the T group, TT5 and TT1 achieved better bond strength values at the initial measurement (64,46 and 63,94 MPa), with TT2 and TT3 being the subgroups with lower µTBS values (46,53 and 58,31 MPa). There were significant differences between TT5 and TT2 (p<0,001), TT4 and TT2 (p<0,001), TT3 and TT2 (p<0,001), and TT1 and KT2 (p<0,001). After thermocycling, TT5 and TT4

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obtained higher bond strength values (58,66 and 55,35 MPa). The lowest value was found in TT2 (36,43 MPa), followed by TT1 (49,44 MPa). Significant differences between TT5 and TT2 (p<0,001), TT4 and TT2 (p<0,001), TT3 and TT2 (p<0,001), and TT1 and KT2 (p<0,001) and TT5 and TT1 (p=0,006) were found.

## FAILURE MODE ANALYSIS

The fracture mode distribution of the initial and thermocycling groups is shown in Figure 6. Representative SEM images are presented in figure 7. In all groups, most failures were seen at the adhesive interface. No cohesive failures within the resin block were observed in the experiment. In the K group initial test, KT5 and KT1 showed more cement cohesive failures than the rest of subgroups (30%). After thermocycling, the rate of fractures at the cement level slightly decreased in all subgroups except KT5 (33%). In the T group, TT1, TT5 and TT4 presented similar rate of cement cohesive failures (20-25%) before thermo-cycling. After artificial aging, those values increased in all subgroups but TT1, and this increase was more significant in the TT5 (46%) and TT4 (37%).

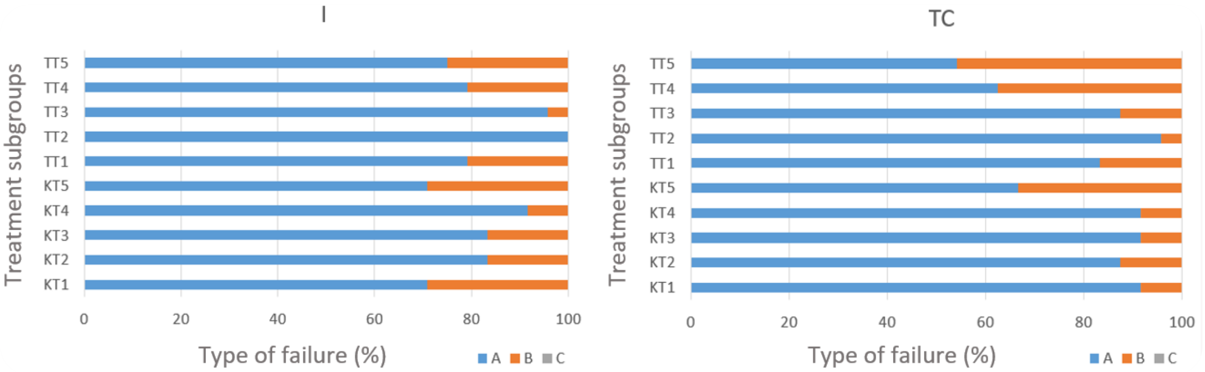

**Figure 6:** distribution of failure mode in the initial (I) and thermocycled (TC) groups. I: initial, TC: after 5000 cycles of thermocycling. KT1: KATANA avencia control subgroup, KT2: KATANA avencia no cleaning subgroup, KT3: KATANA avencia acid etching subgroup, KT4: KATANA avencia Ivoclean control subgroup, KT3: KATANA avencia KATANA cleaner subgroup. TT1: Tetric CAD control subgroup, TT2: Tetric CAD no cleaning subgroup, TT3: Tetric CAD acid etching subgroup, TT4: Tetric CAD Ivoclean control subgroup, TT3: Tetric CAD KATANA cleaner subgroup.

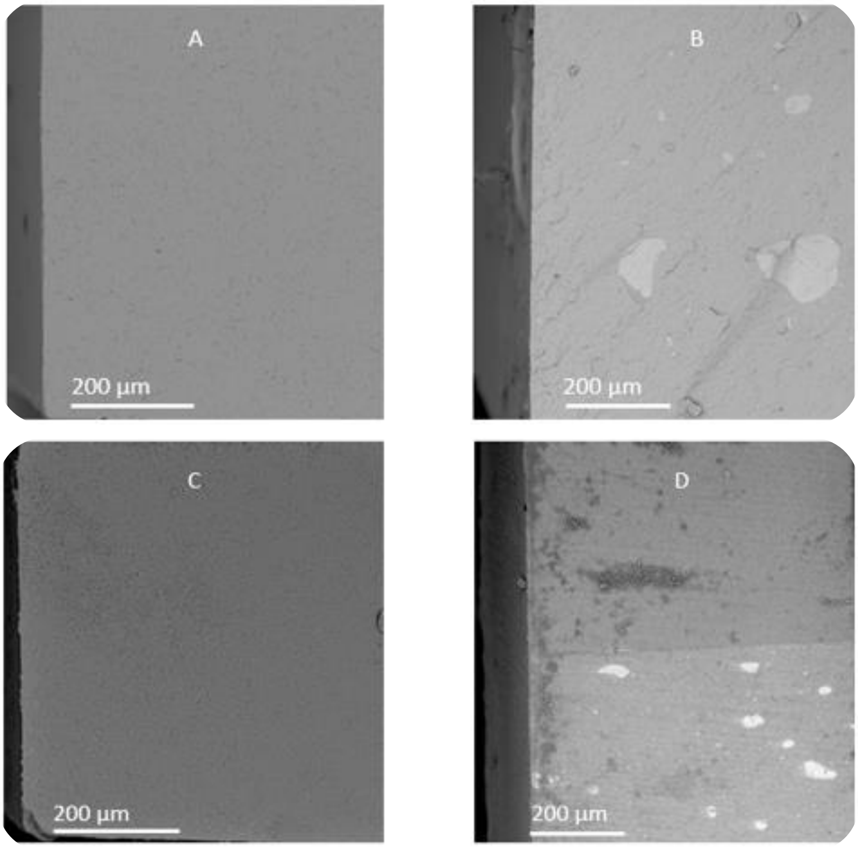

**Figure 7:** SEM images obtained from fractured specimens after µTBS test. A: specimen from KATANA avencia group fractured at the interface. B: failure of a specimen from KATANA avencia group at the cement level. C: failure at the interface, Tetric CAD group. D: side view of a Tetric CAD group specimen after cement cohesive failure.

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# DISCUSSION

Blood and saliva contamination affected the μTBS values negatively in all groups (p<0,05) but in the aged K group, the blood and saliva contamination did not influence the obtained bond strength and the first hypothesis was partially rejected. The adhesive bond to the resin cement after the try-in of the indirect restoration deteriorates by some inhibitors like saliva, blood, or sulcus exudate.[5,17,26] In the present study, fresh capillary blood and non-stimulated saliva were utilized to simulate an actual clinical situation. In laboratory experiments, artificial saliva is often chosen over human saliva because its use does not require permission from ethical committees, and it is easier to standardize. But it also has some drawbacks since it only contains inorganic components and no salivary proteins. This could lead to the obtention of results that cannot always be extrapolated to the clinical practice5. Human fresh saliva use in laboratory experiments is supported by many authors and seems to be an acceptable contamination agent.[27-30.] Dietrich *et al*., stated that fresh capillary blood should be used for blood contamination in laboratory studies, because the addition of an anticoagulant can interfere with the bonding process, masking the results.[31]

The cement manufacturer´s instructions for the pretreatment of the restoration were followed for conditioning the surface of the blocks. This procedure consisted in an initial step where the blocks were sandblasted with 50 μm alumina particles and a following step with ultrasonic cleaning with ethanol. The instructions also recommend the use of the cement's own primer (Multilink A and B primer) on the restoration prior to bonding. Air abrasion seems to be an essential method to obtain acceptable bonding strengths when using CAD/CAM composite resin blocks.[13-15,32] However, Yoshihara *et al*., demonstrated that sandblasting could lead to damage of the resin, especially if the pressure exceeds 0,2 MPa. In that study, all the materials showed an irregular microstructure and cracks. Each kind of block was affected differently by air abrasion.[33] Tekçe *et al*., measured the bond strength of composite blocks to luting agent after 15, 30 and 60 seconds of sandblasting, and the results advocate for a treatment no longer than 30 seconds to prevent damage of the blocks and obtain a better adhesive interface.[34] As stated above, the instructions provided by the manufacturer were used for the air-abrasion protocol: sandblasting with 50 μm alumina particles for 20 seconds at 0,15 MPa. This sandblasting protocol seems to be in line with the studies mentioned before. The objective of the sandblasting is to improve the final bond strength, assuring the creation of a better surface for bonding. This is achieved by producing micro-retentive areas for better adhesion, removing possible contaminants, and exposing filler particles.[33] According to recent literature, the sandblasting technique used in the present study should be safe, roughening the surface of resin-based materials but not creating important defects on it. This also brings another important factor to consider when conditioning the surface of a resin-based restoration, since clinicians could, for example, use airborne particle abrasion for cleaning the restoration after the try-in in mouth, leading to a possible damage of the bonding surface if the total time exceeds 30 seconds. Therefore, the use of a cleaning agent instead of sandblasting after try-in contamination becomes even more important if the restoration has been previously sandblasted. For the present work, two types of blocks were used. The surface analysis with SEM exposed the differences in the microstructure of the surfaces between the two kinds of blocks after sandblasting. In both groups, sandblasting created a microporous surface optimal for bonding, which likely had a positive influence on the results of the μTBS tests. The T group exhibited a more regular structure with smaller pores and less sharp zones than the K group. This, combined with the fact that debris was more frequently present in the K group, probably due to the increased size of the micropores created by airborne particle abrasion, may explain why the T group achieved better values in μTBS tests. In addition, the presence of unnoticed microfractures and cracks or other defects in the K group could have had a negative impact. The accuracy of the surface treatment method for conditioning Tetric CAD material seems evident. However, it can be questioned whether a shorter sandblasting time, the use of smaller particles, or less pressure could be beneficial when conditioning KATANA Avencia material. Another factor that could have an impact on this difference was the different composition of the blocks used. According to the results obtained in a study carried out by Hagino *et al*., the filler content of the material can affect the bond strength when a methacrylate-containing primer is applied, as it adheres to the exposed resin matrix. Depending on the block, the filler content of resin blocks changes and is usually not specified by the manufacturer.[25] Laborie *et al*. published a paper clarifying the filler content of different types of resin blocks: the KATANA Avencia blocks had a filler content of 62%, whereas it was 71% for the Tetric CAD blocks.[8] The results of the present work showed that bond strength values were lower in the K group, the material with more resin matrix content. Therefore, there should be a different mechanism involved. Chuenjit *et al*., found that the impact of surface treatments and their combinations on the surface roughness and energy of CAD/CAM materials varied based on the specific type of CAD/CAM materials used which can contribute to an improved or decreased adhesion[35] Plus, the methacrylate monomers of the Multilink primer or cement could have bonded or penetrated better the Tetric CAD material due to the more regular surface or differences in the contained monomers of the resin matrix of the block. Additionally, applying a ceramic primer including silane to the blocks could improve bonding to KATANA Avencia blocks. This step is indicated according to the instructions for use of the KATANA Avencia blocks. However, in this trial, ceramic primer was not applied in an effort to standardize the luting protocol across all groups and because its use was not recommended by the manufacturer of the luting agent. Many

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authors have found that resin primer application is superior to silane conditioning[36] and better than the combination of both.[37,38] Contrary to this, others have suggested that silane could help maintain the quality of the bond over time.[39] KATANA Avencia blocks contain larger silica particles, which may help create chemical bonding to the luting agent when silane is applied. Another explanation for the better performance of the T group could be the fact that Tetric CAD/CAM blocks and Multilink cement are from the same manufacturer, so they could have been developed, tested, and produced considering the properties of both materials simultaneously to ensure optimal bonding. Small differences in opacity between the materials could be the cause of variation, if the light does not reach the center of the block, being polymerization purely chemical in that area. The actual cause of the better bonding of Multilink cement to Tetric CAD blocks is difficult to determine without further chemical and physical experimentation. For this trial, and as the last procedure of the pretreatment, the blocks were ultrasonically cleaned after air abrasion following the luting agent instructions, but according to the current literature, the effects of ultrasonic cleaning are still unclear.[40]

Decontamination using Ivoclean or KATANA cleaner led to a recovery of the initial bond strength in both K and T groups before and after thermocycling ($p>0.05$). The use of phosphoric acid was able to restore the bond strength in all groups ($p>0.05$) but the initial K group ($p=0,048$). Water cleaning (T2) failed to restore the initial bond strength in all groups except the K group after thermocycling, thus, the assumption of the second null hypothesis – that the type of decontamination method would not affect the results of the bond strength test (μTBS) – have been also rejected in part by the results of this study. Contamination affected the KATANA avencia blocks less compared to the Tetric CAD/CAM blocks. As mentioned above, a possible explanation for this is the irregular microtopography of the KATANA avencia blocks after sandblasting, which could lead to a weaker interface due to microcracks and sharp zones in the surface material, or an incomplete removal of the debris and contaminants, as seen in the SEM analysis. In clinical practice, contamination of the restoration is common and sometimes unavoidable. Following contamination, an organic coating on the surface of the material is created, and it cannot be removed by water rinsing.[4,5] Multiple options have been recommended to clean the bonding surface, but in practice not all alternatives are available. Sandblasting has demonstrated its efficacy in removing contamination;[4,21] therefore, its positive impact was assumed and not included as a cleaning method in this study. Water rinsing is probably the most common cleaning method and is usually the most accessible. Despite this, numerous authors who previously investigated the effects of decontamination, use air-dried contaminated restoration as negative control instead of rinsing with water. For the purposes of this study, blocks after water rinsing were used as negative control groups. There were two main reasons for this: one considers that water cleaning is routinely used by dental practitioners after the try-in of restoration, even if the contamination is not assured; the second assumes that the clinician will not cement a restoration in presence of saliva or blood, which are often detectable, so the restoration must be cleaned at least using water spray if the access to other options is limited. Furthermore, there seems to be no need to study the negative outcomes of contamination with saliva and blood, as they have already been extensively studied and confirmed.[4,30] In this study, blocks decontaminated by water rinsing showed lower μTBS values before and after thermocycling with respect to the control groups, and the differences were statically significant in all groups except the K group after artificial aging. In contrast, the repercussions of phosphoric acid cleaning remain uncertain. Acid-based cleaners may dissolve the contaminants and make their removal easier. While certain studies claim that the application of phosphoric acid removes organic contaminants,[41] others defend that it is not able to remove them completely.[21] According to the results of the present trial, phosphoric acid is an effective decontaminant and seems to help in restoring the initial μTBS after contamination in all groups except in the K group before thermocycling, but this difference is in the limit of being not significant respect with the positive control group ($p=0.048$). Ivoclean contains zirconium oxide and sodium hydroxide, it is hyper-saturated in zirconia particles and highly alkaline (PH=13). Due to this, its use is exclusively extra-oral. It has been suggested that the mechanism of action is based on the fact that, in presence of a high concentration and size of zirconia particles, like in the Ivoclean solution, the phosphate contaminants are more likely to bind to it rather than to the zirconia surface. It also increases surface energy of the zirconia.[21] In the present study, Ivoclean was effective in restoring the μTBS to levels near the uncontaminated group. Since no zirconia blocks were utilized in this experiment, its mechanism of action should be other than the one mentioned above. Sulaiman *et al.*, analyzed the bonding efficiency of a resin cement before and after contamination with saliva. In their study, they applied different cleaning agents to contaminated zirconia surfaces and used the notched-edge shear bond strength test to evaluate their performance. ZirClean (BISCO, Inc., Schaumburg, IL), which is an alkaline (PH> 13) cleaning gel composed of potassium hydroxide and not including zirconia particles, obtained similar results than Ivoclean, thus indicating that the high alkalinity of the paste could be the main mechanism of action rather than the phosphate group absorption. Alkaline solutions break the ionic bond between the contaminant and restorative surface, leaving a clean surface for bonding.[42] KATANA cleaner is a slightly acidic cleaning solution that contains 10-methacryloyloxdecal dihydrogen phosphate salt (10-MDP) and can be used both intra- and extra-orally. MDP salt has a surfactant effect, penetrating organic substances and reducing surface tension.[5] The hydrophobic group of the MDP salt bonds to the contaminant, exposing the hydrophilic phosphate group and allowing it to be rinsed with water. MDP can in addition improve the chemical bonding to dentin and the interactions between dentin and resin cement.[5] In this experiment, KATANA cleaner restored the bond strength to acceptable levels and was able to maintain them even better than the positive control groups after artificial aging. This is in line with the results obtained by Takahashi *et al.*, who measured the microtensile bond

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strength of a resin-based cement (PANAVIA V5) to resin blocks (KATANA avencia) and concluding that the use of KATANA cleaner is beneficial to improve the bond strength after contamination. In that study, μTBS test was performed to analyze the quality of the bonding obtained by different cleaning techniques including phosphoric acid, Ivoclean, and KATANA cleaner.[5] However, results may vary in that samples were contaminated with artificial saliva and only a single type of block and cement were used.

When bonding restorations, the long-term quality of the bonding is crucial to demonstrate clinical efficacy. As a result, when evaluating adhesion, measurements of long-term bond strength are required. Artificial aging can be induced in laboratory studies through techniques such as water storage or thermocycling. When using thermocycling methods, a minimum of 5000 cycles are necessary. Increasing the number of cycles beyond that does not appear to provide any additional advantages.[43] Thermocycling reproduces *in vitro* hydrothermal aging.[44] In the present study, artificial aging of the specimens using the thermocycling method had the greatest impact on μTBS among all the parameters studied (F= 109, p<0.001). This difference was significant except in KT5, which partially rejects the third null hypothesis that thermocycling does not affect bond strength. In all subgroups, μTBS values decreased following artificial aging. In both K and T groups, the values after thermocycling decreased, with the results after aging uniformizing among the K subgroups. Subgroups cleaned with KATANA cleaner were able to maintain the bond strength even better than the positive control subgroups, showing no significant difference in μTBS before and after aging in the K group (p>0.05). When substrate type was not considered, although not statistically significant, the difference between T5 and T1 was still noticeable (p=0.052). Temperature oscillations caused by thermocycling generate mechanical stress at the interfaces between different materials due to expansion and contraction.[45] Also, during artificial aging, post-curing and hydrolysis processes are responsible for the changes in the micro-tensile strengths. If the mechanical stress and hydrolysis processes are balanced with post-curing processes, the μTBS may remain unchanged. However, if one process is dominant, the bonding strength can increase or decrease. There are reports of cases in which the bonding strength increased after thermocycling.[46] In the present experiment, the artificial aging of specimens using the thermocycling method significantly impacted micro-tensile bond strength (μTBS), so it can be concluded that hydrolytic and mechanical degradation had more influence than possible post-curing processes. After thermocycling, an increased number of fractures at the cement level were observed in multiple groups, exceeding the 35% threshold in the KT5, TT4, and TT5 subgroups. This indicates that the actual bonding strength at the interface possibly could have been even higher if the cement was applied in a thin layer between restoration and tooth.

The role of MDP in bonding to tooth structure has been extensively studied, and while its positive outcomes have been confirmed, the exact processes that lead to them are still being discussed. Many different mechanisms of action had been proposed as reasons for the efficacy of MDP in improving bonding strength and durability: the creation of an acid-base resistant zone that protects the bonding interface from the acidic oral environment; ionic bonding to the calcium of hydroxyapatite and formation of insoluble MDP-Ca salt within the bonding interface; nano-layering by the self-assembly of the MDP-Ca that can defend the formed hybrid layer against biodegradation or strengthen the hybrid layer, possibly preventing collagen degradation; free MDP and/or MDP-Ca salts might inhibit the activity of proteases or improve the stability of collagen, etc.[47] Therefore, there may be unexplained mechanisms of action. In this study, the previously mentioned ways of action are excluded by the fact that dentin was not used as a substrate for bonding. Valente *et al*. stated that the vinyl group of the MDP molecule could facilitate polymerization and provide chemical coupling with unsaturated carbon links in the resin matrix of the substrate, while the phosphate group could promote adhesion with hydroxyapatite or metal oxides such as alumina or zirconia.[48] Nagaoka *et al*. studied the chemical interactions between 10-MDP and zirconia and proposed different mechanisms, but the actual process has not yet been elucidated.[49] In the present study, resin blocks were used as substrate. The application of a cleaner containing MDP seems useful to restore and maintain bond strength. In the KT5, no statistical difference was observed in μTBS before and after aging. In the TT5, the difference was significant but still performed better than the control group. Also, the subgroups treated with Ivoclean improved the results after thermocycling in K and T groups, while acid cleaning seemed to help in the K group, but the differences were not significant. A possible explanation for this could be, rather than the use of the decontaminating agent, the action of rinsing the sample multiple times, which was performed in all groups but the positive control group. Even with the surface of the blocks being free from organic contaminants, debris from polishing and sandblasting could have remained, making the adhesive interface more prone to degradation from thermocycling than those rinsed multiple times, even with them having a low quantity of organic contaminants.

The main limitations of this study are related to the lack of teeth in the experimentation, because bonding to dentin and cavity design are important factors that can affect the quality of the bonding. The focus of this experiment was the restoration-cement interface because not all decontaminating methods used during the study are suitable for intraoral use. Perhaps the efficacy of the cleaning agents differs more when tooth structure is taken into account. The use of only one cement type, and its build-up to 6 mm when it is supposed to be used in thin layers, may have influenced the outcomes of the study too. Additionally, resin block compositions and structures differ among manufacturers, introducing another

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source of variation. Mechanical fatigue was not included in the design of the study and can affect the bonding in alternative ways. Further research is needed, including the use of human teeth, mechanical and chemical testing, and a variety of resin blocks and cements.

## CONCLUSIONS

Within the limitations of the present study, it can be concluded that the bond strength between resin blocks and resin based luting cements is influenced by contamination with blood and saliva and artificial aging. Decontamination after try-in of indirect restoration either with Ivoclean or KATANA cleaner improves bonding. Specifically, when KATANA cleaner was applied after contamination, it not only effectively restored the initial bond strength between KATANA avencia blocks and Multilink cement but also maintained this strength over time, surpassing the results of the positive control group.

## CONFLICT OF INTERESTS

All authors declare no conflict of interest.

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