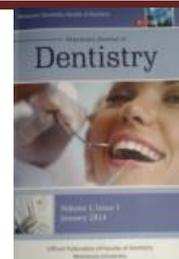




## *Influence of Micromechanical and Chemical Conditioning on Bonding to Machinable Composite*



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### **Abstract:**

**Purpose:** To evaluate the influence of micromechanical and chemical conditioning on bonding to hybrid ceramic (machinable composite MZ100 blocks). **Materials and methods:** A total of 64 machinable composite discs (MZ100 paradigm) were fabricated using CAD/CAM technology according to the desired dimensions (10 mm width and 4 mm thickness). The discs were divided in two main groups (n=32): as milled group (AM) and sandblast group (SB) used Al<sub>2</sub>O<sub>3</sub> (50-µm). Each main group was subdivided into two subgroups (n=16) according to method of chemical conditioning: conventional silane (SI) and multipurpose primer (MP). Composite resin discs were fabricated according to desired dimensions (4.5 mm width x 3.5 mm thickness). Surface treated MZ100 discs were bonded to the composite resin discs according to the types of adhesive resin cements, multistep adhesive resin cements or self-adhesive resin cements. One hour after bonding, all specimens were stored in water bath at 37°C for 5 months followed by thermocycling for 10,000 cycles. Following artificial aging all bonded specimens underwent shear bond strength testing using universal Instron testing machine. Assessment of the debonding failure mode was done by Binocular optical microscope and SEM. Statistical analysis was performed using a 3-Way ANOVAs. **Results:** In Three-Way ANOVAs test revealed statistically significant different in SBS between (SB) and (AM) groups {p=0.000} and between different resin cements used {p=0.006}. While there, was no statistically significant different when used different chemical conditioning methods {p=0.132}. **Conclusions:** Sandblasting of the MZ100 paradigm block surface is recommended when luting with the self-adhesive or multistep adhesive resin cements. The self-adhesive performed SBS values compared to multistep adhesive resin cement in all groups. The combination between micromechanical and chemical conditioning of MZ100 paradigm block surface are recommended over the chemical conditioning alone.

**Key words:** Machinable composite. Surface treatment. Resins. Bond Strength.

### **Introduction**

Hybrid ceramics such as CAD/CAM machinable composite blocks over CAD/CAM ceramic blocks in terms of resilience and less abrasion on the antagonist enamel,<sup>1</sup> because have elastic moduli comparable to those of natural tooth substance. In addition to enhanced milling accuracy, fewer composite chips at restoration margins and less crack formation during manufacturing by CAD/CAM technique, and no need for crystallization or additional curing cycles after CAD/CAM milling. Clinically, helped to preserve overall balance of the dentition and can also be easily adjusted or intraoral reparability, simple polishing procedure and lower cost.<sup>2-4</sup>

Many surface treatments before cementation are used nowadays to create a surface alteration of the esthetic restorations to enhance bonding to the tooth structure and have a significant role in the success of ceramic restorations and their longevity.<sup>5</sup> This stable bond is important for strengthening retentive forces, marginal adaptation and resistance to fractures. Intaglio surface treatment works on boosting surface energy, bonding area and wettability.<sup>6</sup>

Increased material surface roughness is critical step in improving bonding properties, air-particle abrasion and additional silane treatment should be used to improve the resin bond to CAD/CAM composites.<sup>7</sup> Alumina oxide particles propelled by compressed air to remove unfavorable oxides and contaminants, increase surface

energy, bonding surface area, and surface roughness. It is a most widely surface treatment used to specifically abrasion alloys, zirconia, and machinable composite CAD/CAM materials. The air abrasion systems rely on air-particle abrasion with different particles size ranging from 30 to 250 µm.<sup>8,9</sup>

The materials of choice for the adhesive cementation of machinable composite CAD/CAM is resin cements.<sup>10</sup> Cementation process involved surface roughness, priming, and bonding. Multistep adhesive resin cements are technique sensitive and time consuming. On the other side self-adhesive resin cements can be used as one step very simple procedure.<sup>11</sup> Currently, some universal adhesives may contain silane, which eliminates the silanization step when bonding to glass ceramics, hybrid materials, and resin composites.<sup>12,13</sup>

Therefore, the purpose of this in vitro study was to influence of micromechanical and chemical conditioning on bonding to machinable composite. The hypothesis of this study were that (1) there would be influence of sandblast as surface treatment leads to increase the bond strength comparable than untreated surface, (2) multipurpose primer would have improves bond strength more than conventional silane regardless of surface treatment, (3) the bond strength of multistep adhesive resin cement would be higher than self-adhesive resin cement.

## Materials and Methods

### Specimens preparation

A total of 64 CAD/CAM hybrid ceramic (paradigm MZ100 composite blocks) were selected for this study. All specimen surfaces were milled using Ceramill mikro CAD/CAM. Composite resin discs were fabricated according to desired dimensions (4.5 mm width x 3.5 mm thickness) used a specially designed Teflon mold with several round holes.

### Surface treatment

All specimens were undergoing to steam cleaning. Group I: (n=32) As milled no surface treatment applied. Group II: (n=32) Sandblast  $Al_2O_3$  (50- $\mu$ m). The intaglio surface of specimens were exposed to air particles abraded with 50- $\mu$ m aluminum oxide particles ( $Al_2O_3$ ) by sandblaster machine at a pressure of 0.2 MPa for 10 sec/cm<sup>2</sup> perpendicular to the machinable composite surface at a distance of 10 mm. Then ultrasonically cleaned with 95% ethyl alcohol for 5 minutes and drying with oil-free air.

### Chemical conditioning

Each main group was divided into two subgroups (n=16).

Subgroup (A): Conventional silane was applied to intaglio surface of specimens with a spiral brush and leaved it for 60 seconds in order to evaporate then oil-free air was applied for a period of 30 seconds.

Subgroup (B): A thin coat of multipurpose primer (Monobond N) was applied with a micro-brush to the intaglio surfaces for 60 seconds then dried with oil-free air for 30 seconds.

Each subgroup was divided into two divisions (n=8) according to type of adhesive resin cement used:

Division (SARC): Self-adhesive dual-cured resin cement RelyX U200 Automix (3M ESPE, St Paul, USA).

Division (MARC): Multistep adhesive resin cement Multilink N (Ivoclar-vivadent, Schaan, Liechtenstein).

### Bonding procedures

Two resin cements (RelyX U200 or Multilink N) were mixed with disposable automix tip based on the manufacturer's instructions and then placed on the intaglio surface of MZ100 specimens. After cement application the

composite disc was bonded to the treated intaglio surface of MZ100 specimens used cementation loading device under a static load of 1kg to create a uniform cement layer. The resin cement was light cured from all directions for 3 seconds to remove the excess cement by used a disposable brush. The final light curing was performed for 20 seconds at each surface. Under the static load, the bonded assembly was kept for 5 minutes.

### Artificial aging of the specimens

One hour after cementation the specimens were stored in distilled water at 37°C for 5 months. Then the specimens were subjected to thermal cycling for 10,000 cycles.

### Shear bond strength test

A universal testing machine was used to determine the shear bond strength. The application of shear force at a crosshead speed of 1 mm/min, a chisel-shaped mono-beveled metallic rod attached to the movable upper compartment of the testing machine was used. The load causing debonding of each specimen was registered in Newton. Shear bond strength values were determined by dividing the maximum load with the bonding area at failure (mm<sup>2</sup>) and recording the resulting pressure in MPa.

### Mode of failure

Assessment of the debonding failure mode was done by Binocular optical microscope and SEM to distinguish the failure modes that were defined as follows: (1) Adhesive failure pattern at MZ100 / resin cement interface. (2) Cohesive failure pattern within composite resin or resin cement. (3) Mixed failure pattern including cohesive and adhesive failure.

### Statistically analyses

Statistical data analysis was performed using version 25.0 of the International Business Machine (IBM) Social Package for Static Sciences (SPSS). Three-way ANOVA, Two-way ANOVA and One-way ANOVAs were used to conduct statistical analysis experiments, followed by the Post Hoc Tukey-HSD test ( $\alpha=0.05$ ).

### Results

Three-way ANOVAs test showed that there was statistically significant difference as regard to SBS between sandblasted groups and as milled groups ( $p=0.000$ ) also it showed statistically significant difference between different resin cements ( $p=0.006$ ). While there, was no statistically significant different when used primer agent protocols ( $p=0.132$ ). Statistical significant differences between groups are shown in Table 1.

**Table 1:** Summary of overall 3-way ANOVA: influence of different variables on shear bond strength.

Test group	Type III Sum of squares	f	Mean square	F	p-value
Surface treatment	740.896		740.896	94	0.000*
Primer agent	23.809		23.809	3	0.132
Resin cement	84.530		84.530	2	0.006*
Error	571.537	6	10.206		
Total	11587.048	4			
Corrected total	1603.692	3			

a. R Squared = .644 (Adjusted R Squared = .599)

**b. Computed using alpha = .05**

**Figure1:** Showing optical micrograph and SEM examination of group (SB-MP-MARC) with adhesion failure mode.

## Discussion

The current in vitro study, the strength of the shear bond was evaluated for hybrid ceramics and two different resin cements with or without air abrasion and using either a conventional silane or a multipurpose primer.

According to current study results, the first hypothesis that used sandblast as surface treatment leads to increase the bond strength was accepted. The second hypothesis that the used multipurpose primer improves bond strength more than conventional silane regardless of surface treatment was partially accepted, because only the groups that had multipurpose primer application in as milled group achieved higher bond strength to the resin cements than the conventional silane. Whereas the sandblast group revealed the conventional silane slightly superior bond strength compared to the used of multipurpose primer. The third hypothesis that the bond strength of multistep adhesive resin cement is higher than self-adhesive resin cement was rejected.

The results of this study in air-born particle abrasion group showed the highest shear bond strength value of (18.3±2.7 MPa) regardless of the types resin cement or primer agents compared to untreated group. Despite some variations in the study's design Elsaka S (2014)<sup>14</sup> have also found the sandblasting group major efficacy for both types

of CAD/CAM resin blocks were substantially greater than that of the untreated group. These results were in agreement with the results of several other studies,<sup>15,16</sup> Augusti D et al. (2015)<sup>15</sup> they concluded that the sandblasting increased of the SBS values compared to the untreated group. Also the Barutçigil K et al. (2019)<sup>16</sup> they showed, bond strength values were enhanced by sandblasting surface treatment methods compared to the untreated group.

The results of this study, as milled group revealed statistically significant different when used two different primer agents (conventional silane or multipurpose prime). The multipurpose primer (Monobond N) resulted in higher bond strength values (12.7±3.4 MPa), irrespective of cement types compared to the use of conventional silane (7.4±2.8 MPa). One probable explanation for these results is that the silane is capable of creating a solid bond to fillers within the resin composite, but at the same time, its bond to resin matrix is less strong.<sup>6</sup>

In contrast the sandblast group revealed statistically no significant different when used different primer agent protocols. The conventional silane resulted in slightly superior score (18.3±2.7 MPa) regarding the bond strength compared with the used of multipurpose primer (16.1±2.2 MPa) regardless of the cement types. This results

agreement with May M et al. (2021)<sup>17</sup> founded that, the conventional silane recorded the highest values in both surface treatment (SB or HF) groups compared to multipurpose primer.

The results of study that showed in two different resin cements when used multistep adhesive resin cement Multilink N or self-adhesive dual-cured resin cement RelyX U200 Automix there were revealed statistically significant different in SBS. We noticed the RelyX U200 outperformed the Multilink N in all groups. The RelyX U200 registered highest bond strength between the MZ100 discs and the composite resin discs with the maximum SBS value of (18.3±2.7 MPa) in (sandblast + conventional silane) group. While that, the Multilink N registered maximum SBS value of (15.6±2.5 MPa) in the same previous group.

This result agreement with Cinar et al. (2019)<sup>18</sup> they found the highest SBS for Lava ultimate (18.73±1.91 MPa) was obtained in group sandblast + silane with self-adhesive resin cement. Also the Sadighpour et al. (2018)<sup>19</sup> they showed self-adhesive resin cement (RelyX U200) higher than multistep adhesive resin cement (Panavia).

**Conclusion** within the limitations of this study:

1- Sandblasting of the hybrid ceramic surface is recommended when luting with the self-adhesive or multistep adhesive resin cements in order to significantly improve bond strength values.

2- RelyX U200 self-adhesive resin cement outperformed SBS values compared to multistep adhesive resin cement in all groups.

3- The combination between micromechanical and chemical conditioning (conventional silane or multipurpose primer) of MZ100 paradigm block surface are recommended over the chemical conditioning alone.

**Recommendation**

1- Further studies are necessary to access the long-term bond strength of different types of resin cement to resin-matrix ceramic CAD/CAM, and other surface treatments and adhesive approaches can also be evaluated.

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