The effect of elapsed time following alumina blasting on adhesion of CAD/CAM resin block to dentin

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The aim of this study was to evaluate the time elapsed bond strength of the CAD/CAM resin block (CRB) to bovine dentin after alumina blasting. CRB (KATANA AVENCIA BLOCK) slices were ground with #600-SiC paper and divided into three groups according to alumina blasting pressure —0.1 MPa, 0.2 MPa or untreated— and then divided into two subgroups according to the time elapsed after alumina blasting —same-day or one-week “dry-storage” at controlled laboratory conditions before cementation. The CRB slices were then cemented to bovine dentin with Panavia V5 (Kuraray Noritake Dental), and divided into two subgroups —light curing or chemical curing. After 24 h storage in distilled water at 37°C, the specimens were then subjected to micro-tensile bond strength (µTBS) testing. One-week group showed a significant decrease in µTBS. The µTBS values showed that CRBs must be cemented with light curing immediately after alumina blasting at 0.1 or 0.2 MPa to obtain a stable adhesion.

Keywords: CAD/CAM, µTBS, Alumina blasting, Resin cement, Dentin

INTRODUCTION

At the present time, indirect dental composites are widely used. Manually operated restorations are carried out extraorally for greater polymerization than direct composites with little unreacted units1-4, and they are incrementally built to avoid polymerization shrinkage5. Recently, computer-aided design computer-aided manufacturing (CAD/CAM) manufacturers launched industrial CAD/CAM resin blocks (CRB) with new polymerization techniques and different compositions. These industrial techniques require a higher temperature (HT) of 180°C and/or a higher pressure (HP) of 300 MPa6 to improve the mechanical properties of composite and to overcome many issues that are associated with the polymerization of composite in comparison to manually fabricated direct and indirect composite7. Besides the HT/HP technique, the manufacturers developed a new technique (the filler press and monomer infiltration) for CRB to obtain appropriate mechanical properties8. This CRB has densely packed silanated nanofillers, alumina filler (20 nm), and silica filler (40 nm), with fillers that load up to 62% by weight. The degree of conversion is extremely high in all CRBs. Therefore, the unreacted methacrylate groups are minimal or zero, so the bonding of CRB to resin cement will be difficult8. However, there are no specific recommendations regarding the bonding of CRB to the tooth substrate and between the CRB and cement as well. Creating stable bonding of the adherend surfaces and the cement depends on mechanical, physical, and chemical adhesion properties of the resin cement to the adherend surface9. In general, roughening the adherend surface leads to an increase of the surface area and the bonding accordingly10. However, the CRB interface with resin cement requires a chemical bonding as well and can be enhanced by using different adhesive systems in addition to sandblasting12.

A bonding assessment can be evaluated by using the micro-tensile bond strength (µTBS) test. Few studies have investigated the µTBS of CRB and the resin cement13-15. In addition, there have been limited studies about the adhesion between CRB and the tooth substrate, because the detachment might have occurred on either side (dentin-cement interface or CRB-cement interface).

Moreover, some manufacturer recommend sandblasting in addition to silanization before cementation. However, chairside CAD/CAM and/or an adjustable sandblasting machine are not available in all dental clinics. In such situations, the milling and sandblasting procedures of the CRB have to be completed at a dental laboratory and might be stored for up to two weeks before the patient’s appointment.

Therefore, this current study was carried out to conduct an investigation on the bonding performance of dentin and CRB interface by inspecting the impact of elapsed time between alumina blasting and cementation.
Materials used in this study
The material compositions used in this study are listed in Table 1. KATANA AVENCIA BLOCK, Kuraray Noritake Dental, Tokyo, Japan) is a CRB and PANAVIA V5 (PV5, Kuraray Noritake Dental) is a resin cement with its tooth primer. Forty percent phosphoric acid (K-etchant gel, Kuraray Noritake Dental) was used to clean up the CRB surface. Clearfil Ceramic Primer (Ceramic Primer, Kuraray Noritake Dental) was used as a silane coupling agent for the CRB surfaces.

Specimen preparations
The experimental procedures are schematically illustrated in Fig. 1. Recently extracted bovine incisors, stored frozen, were used as bonding substrates. The labial surfaces of the teeth were ground using a model trimmer under water lubrication to expose the dentin surface; they were then sectioned in the middle of the crown by using a low-speed diamond-blade saw (Isomet, Buehler, Lake Bluff, IL, USA) under water irrigation, exposing areas of middle-depth dentin. The exposed dentin surfaces were wet ground with #600-grit SiC paper (Sankyo Rikagaku, Saitama, Japan) to create flat surfaces with a standard smear-layer before cementation.

On the other hand, the CRB was cut into 24 slices with dimensions of approximately 14.5×14.5×2 mm. The slices were then wet ground by #600 SiC paper. After that, they were randomly distributed into three groups: untreated, and blasted by air-particle abrassion with 50-μm Al2O3 particles at 0.1 or 0.2 MPa (20 s, 10 mm distance) by using a blasting machine (Basic Master, Renfert, Hilzingen, Germany). All CRB slices were cleaned with 99% ethanol for 3 min in an ultrasonic bath. Then according to the time elapsed after alumina blasting, the slices were divided into two subgroups: same-day (S) and one week (W) “dry storage” at controlled laboratory conditions before cementation, where the temperature was set to 23.0±0.5°C while the relative humidity (RH) was 50±5%.

Bonding procedures
The adhesive surfaces of the slices were treated with 40% phosphoric acid (K-etchant) for 20 s, rinsed with water for 10 s, and air dried. The surfaces were then treated with a silane coupling agent by using ceramic primer for 20 s and air dried gently. All exposed bovine dentin surfaces were then treated with tooth primer for 20 s and air dried. After that all CRB slices were cemented to on µTBS and the appropriate curing methods of the dual cure resin cement. The null hypotheses of this study were that (a) the time elapsed after alumina blasting and (b) the curing mode of the dual cure resin cement show no influence on the bond strength between the CRB and the dentin.

Table 1 Composition of the materials used in the study and their applications procedure

| Material          | Lot No | Composition                                                                 | Application procedures                                      |
|-------------------|--------|----------------------------------------------------------------------------|-------------------------------------------------------------|
| KATANA AVENCIA BLOCK | 000320 | Mixed filler with colloidal silica (40 nm) and aluminum oxide (20 nm), cured resins consisting of methacrylate monomer (Copolymer of Urethane dimethacrylate and other methacrylate monomers), pigments, filler content 62 (wt%) | —                                                           |
| PANAVIA V5         | 6Q0035 | Paste A: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, initiators, accelerators, silanated barium glass filler, silanated fluoroaluminosilicate glass filler, colloidal silica | Apply it by auto-mix syringe on the CRB surface, then light cure for 60 s or chemical cure for 30 min in dark. |
| PANAVIA V5 Tooth Primer | 5Q0027 | 10-MDP, HEMA, hydrophilic aliphatic dimethacrylate, accelerators, water. | Apply on the dentin surface for 20 s and air-dry gently.     |
| K-etchant gel      | B80016 | 40% phosphoric acid, water, colloidal Silica, dye.                          | Apply on the CRB surface for 20 s, rinsing with water for 10 s and air-dry gently. |
| Clearfil Ceramic Primer Plus | 1V0001 | 3-trimethoxysilylpropyl methacrylate, MDP, ethanol                         | Apply on the CRB for 20 s and air-dry gently.               |

Manufacturer: Kuraray Noritake Dental, Tokyo, Japan.

10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate; Bis-GMA: bisphenol-A-diglycidyl methacrylate; TEGDMA: triethyleneglycol dimethacrylate.
bovine dentin using PV5 resin cement. In accordance to the curing methods of the resin cement, the specimens were divided into two subgroups: light curing groups (LC) using a halogen light curing unit (Optilux 501, Demetron, Danbury, CT, USA) at 600 mW/cm² of light intensity for 60 s, and chemical curing groups (CC) by keeping the specimens in the dark at the laboratory for 30 min. All the specimens were then stored in distilled water for 24 h at 37°C.

µTBS test
After the specimens were stored for 24 h, they were perpendicularly sectioned with the low-speed diamond saw at the adhesive-dentin interface into approximately 1×1 mm beams. A total of 12 beams for each subgroup were tested (n=12). The beams were individually fixed to a jig with a cyanoacrylate glue (Model Repair II Blue, Dentsply-Sankin, Tokyo, Japan) and then subjected to µTBS test using a universal testing machine (EZ-Test, Shimadzu, Kyoto, Japan) at a crosshead speed of 1 mm/min.

Failure mode analysis
After the debonding of the specimens, the beams were mounted with carbon adhesion tape (Nisshin EM, Tokyo, Japan) on the specimen holders and fixed with cyanoacrylate glue (Model Repair II Blue, Dentsply-Sankin). They were then gold sputter-coated and inspected by scanning electron microscopy (SEM; JSM-5310LV, JEOL, Tokyo, Japan) at 100× magnification. The failure modes were classified into four categories: (A) adhesive failure between CRB and resin cement, (B) adhesive failure between dentin and resin cement, (C) mixed failure including adhesive failure between CRB and resin cement or between dentin and resin cement in addition to cohesive failure within dentin, and (D) cohesive failure within dentin.

SEM observation of CRB surface after surface treatment
In order to observe the CRB surface texture, the CRB surfaces were ground with #600 SiC paper, blasted with alumina at (0.1 and 0.2 MPa) and examined by SEM at ×350 and ×10,000 magnification. For SEM observation, the samples were mounted with carbon adhesion tape on a specimen holder and coated with gold, and SEM observation was operated at 15 kV.

Statistical analysis
There were five samples in each group, based on the pilot study using PASS 15 software (Power Analysis and Sample Size Software, NCSS, LLC, Kaysville, UT, USA). A factorial design with three factors at 2, 2, and 3 levels has 12 groups (treatment combinations). A total of 144 subjects are required to provide 12 subjects per group. This design achieves 100% power when an F test is used to test time at a 5% significance level and the effect size is 0.400, achieving 100% power when an F test is used to test curing mode at a 5% significance level; the effect size is 0.400, achieving 99% power when an F test is used to test blasting pressure at a 5% significance level, and the effect size is 0.400 and achieves 90% power when an F test is used to test the time*curing*blasting pressure interaction at a 5% significance level and the effect size is 0.300. The µTBS indicated normal distribution by the Shapiro-Wilk test in each group (p>0.05). The µTBS values of the different curing methods, time factor, and the pretreatment methods were analyzed by three-way analysis of variance (ANOVA), while the values of each group were subjected to a two-way ANOVA with Bonferroni’s correction at a 95% confidence level. All statistical procedures were carried out using SPSS ver. 23.0 (IBM, Chicago, IL, USA).
RESULTS

μTBS test
The mean μTBS values were shown in Table 2. Three-way ANOVA indicated a significant effect for the all three parameters: alumina blasting pressure, time elapsed, and type of curing (p<0.01). By using 2-way ANOVA, most of the groups showed significant reduction in μTBS after one-week dry storage while chemical groups showed greater reduction compared with light-cured ones. However, in the S group at 0.2 MPa, results showed no significant differences between the LC and CC, as well as LC at 0.2 MPa after one-week. In the W untreated group, there was no significant difference between LC and CC. In the untreated group S (CC) after one-week dry storage W (CC) there was no significant difference, however in comparing to the untreated group, S (LC) showed a significant drop in μTBS after one week W (LC).

Failure mode analysis
The results of the failure mode analysis after debonding were summarized in Table 3. The typical fractured specimens on the CRB side (A, C) or dentin side (B, D) were displayed in Fig. 2. Most of the groups showed

Table 2 Microtensile bond strengths of the CRB to dentin (MPa)

|       | CC       | LC       |
|-------|----------|----------|
| Untreated |          |          |
| S       | 28.9 ± 8.6 (8.6) | 42.5 ± 7.4 (7.4) |
| W       | 27.6 ± 5.3 (3.3)  | 26.9 ± 4.2 (4.2)  |
| 0.1 MPa |          |          |
| S       | 39.3 ± 8.9 (8.9)  | 47.3 ± 7.1 (7.1)  |
| W       | 29.1 ± 5.3 (5.3)  | 41.0 ± 7.7 (7.7)  |
| 0.2 MPa |          |          |
| S       | 40.6 ± 13.1 (13.1)| 44.4 ± 14.6 (14.6)|
| W       | 29.1 ± 5.4 (5.4)  | 39.4 ± 13.1 (13.1)|

n=12.
Data are shown as mean (standard deviation).
Values with same superscript letters were no statistically significant differences (p>0.05).
Small letters show the horizontal significance and capital letters show the vertical significance for each group.

Table 3 types of failure mode

|       | CC       | LC       |
|-------|----------|----------|
| Untreated |          |          |
| S       | [11/0/0/0] | [9/0/2/1] |
| W       | [12/0/0/0] | [9/0/2/1] |
| 0.1 MPa |          |          |
| S       | [12/0/0/0] | [11/0/2/1] |
| W       | [4/7/1/0]  | [5/6/1/0]  |
| 0.2 MPa |          |          |
| S       | [10/0/1/1] | [6/0/4/2] |
| W       | [4/8/0/0]  | [10/1/0]  |

Numbers in square brackets are the number of specimens classified into four fracture modes [A/B/C/D]: [A] adhesive failure between CRB and resin cement, [B] adhesive failure from dentin, [C] mixed failure, including adhesive failure between CRB and resin cement or between dentin and resin cement in addition to cohesive failure within dentin, [D] cohesive failure within the dentin.

Fig. 2 Micrographs of the failure types, magnification of 100×.
(A) adhesive failure between CRB and resin cement, (B) adhesive failure between dentin and resin cement, (C) mixed failure, including adhesive failure between CRB and resin cement or between dentin and resin cement in addition to cohesive failure within dentin, (D) cohesive failure within the dentin. DT: Dentinal tubule.

Fig. 3 SEM observation of CRB after polishing by SiC #600 and prior to cementation.
(A) Untreated surface, many scratches caused by the #600 SiC paper were observed on the surface at 350× magnification, (B, C) Alumina-blasted surfaces by 0.1 and 0.2 MPa respectively at 350× magnification, after alumina blasting, many irregular micro-dimples were observed and the scratches disappeared, (D) at 10,000× magnification of single dimple after alumina blasting at 0.2 MPa, many homogeneity fillers exposed.
predominant adhesive failure between CRB and resin cement (type A) with the exception of the blasted one-week groups, which showed adhesive failure between dentin and resin cement (type B) alongside the adhesive failure between CRB and resin cement (type A). Furthermore, there was a low rate of mixed failure (type C) and cohesive failure within dentin (type D) reported among the one-week and same-day groups.

**SEM observation of CRB surface after surface treatment**

Figure 3 shows the SEM images of the CRB surface morphology. The untreated group (A) showed many scratches caused by #600 SiC paper. With 0.1 MPa (B) or 0.2 MPa (C) blasting pressure, the CRB surface showed many micro-dimples and irregular structure while mostly all the scratches disappeared. At high magnification of CRB surface after alumina blasting at 0.2 MPa (D), the CRB surface showed many nano-fillers densely packed in the dimples.

**DISCUSSION**

Bonding approaches of CRB resemble those of the indirect composite. However, the degree of polymerization of CRB is higher than the manually polymerized composite\textsuperscript{16-18}. The reduced amount of unreacted monomers resulted from the industrial polymerization; the operator is required to pretreat the CRB surface properly prior to cementation. Consequently, the initiation of chemical adhesion with resin cement reduced as the free carbon double bond reduced\textsuperscript{16,17}.

In this study, the µTBS results showed that the highest bonding strengths were obtained after alumina blasting at 0.1 or 0.2 MPa with the light curing method in the S group. The SEM images after alumina blasting prior to cementation (Fig. 3) showed how much the surface changed after sandblasting at 0.1 or 0.2 MPa to become a rougher surface with many dimples and irregular structure. However, a few residual scratches remained on the CRB surface at the 0.1 MPa group, which implies that the sandblasting at this pressure was inadequate to achieve a uniform surface. Air-abrasion with alumina blasting (Al\textsubscript{2}O\textsubscript{3}) with a 50-µm particle size was reported as the efficient method of surface roughness to increase the surface energy, expose the cleaned fresh surface, and enhance the micromechanical interlocking\textsuperscript{17}. However, the composite is a very yielding material with a lower Vickers hardness than either dental ceramic or zirconia\textsuperscript{19}. Cracks inside the resin matrix were reported after sandblasting at 0.2 MPa among other CRBs, and the length of crack was ranging from 1–10 µm. Therefore, the blasting pressure should not exceed 0.2 MPa\textsuperscript{19}.

The silane coupling agent is a bifunctional molecule and reacts chemically with exposed composite fillers\textsuperscript{17}. However, CRB has to be pretreated initially to facilitate the retention of silane molecules to promote the wettability of resin cement to CRB surfaces. Nonetheless, silanization only without alumina blasting or alumina blasting only without silanization showed inferior bonding strength\textsuperscript{20} compared to combination of them\textsuperscript{12,20}.

Poskus et al.\textsuperscript{21} found that the treatment of an indirect composite surface by a silane coupling agent after sandblasting and cleaned with 96% ethanol for 5 min increased the bonding strength significantly. In this study, 99% ethanol ultrasonication cleaning for 3 min was used to remove the residual alumina particles attached to the blasted surfaces of CRB and to expose fillers by partially dissolving the organic matrix, so the silanization could reach more fillers effectively. In fact, subsequent to sandblasting and ultrasonication, the nano-filler particles were exposed, which is an ideal condition to achieve silanization process effortlessly\textsuperscript{22}.

Generally, according to the manipulation instructions of the CRBs, some companies indicated that the sandblasting should have done it just prior to cementation. However, in reality most of the clinicians or ceramic technicians do not consider this as a crucial concern. In this study, the time elapsed after alumina blasting was shown to influence the adhesion negatively between the CRB and the dentin, regardless of the curing method used, through which the hypothesis (a) was rejected. Observations in this study showed that if the silanization is carried out shortly after alumina blasting it might produce optimum results but leaving the CRB exposed to ambient air leads to the reduction of the µTBS, as a result of water sorption (Fig. 4)\textsuperscript{23-25}. In this study, the lab environment was controlled at 23.0± 0.5°C, while the RH was 50±5%. However, the µTBS decreased significantly for both LC and CC groups after one-week dry storage. The failure mode analysis points out that the rate of adhesive failure between cement and dentin (type B) appears alongside the adhesive failure between cement and resin (type A) after one-week storage of either LC or CC sandblasted groups. An exception was the W 0.2 MPa (LC) group, which showed a lower rate of (type B) failure than the other W 0.1 MPa (LC) group, and this might be because more fillers were exposed to ambient air after alumina blasting at 0.2 MPa, which may weaken the CRB-cement interface. These results...
suggest that the ambient air affected the cement layer after one-week of dry storage. In spite of that, the W (CC) 0.1 and 0.2 MPa groups registered a higher rate of (type B) failure because the water diffusion of dentin to the adherend surface as a result of slow rate of chemical polymerization.

The chemical curing (0.1 and 0.2 MPa) groups showed more reduction of the µTBS than the (LC) groups. However, the untreated (CC) group was not influenced by the one-week dry storage as much as the (LC) group, since the (CC) group is water-inclusive. The slow rate of polymerization of the CC group gives the opportunity for water diffusion from the dentin, and it might obstruct the chemical adhesion. Nonetheless, the situation is more complicated in the case of the W (CC) as water droplets existed in both adherend surfaces. The untreated S (LC) group showed a significant improvement of the µTBS over the S (CC) group, while the W (LC) group exhibited a significant reduction after one-week because the water droplets contaminated the cement layer.

Lab conditions may interact readily with the exposed silicon dioxide and/or polymeric matrix. The quantity of absorbed water by resin composite is dependent on the hydrophilicity of resin matrices and the filler particles. However, by monitoring mass variance of resin composite at lab conditions, it is speculated that the composite resin was sensitive to ambient air and has the ability to absorb water after few days even at 50% RH or at low humidity ~22%. Accordingly, Asay and Kim stated that the water adsorbed layer on the silicon dioxide surface can increase when the RH increases, as the adsorbed layer composes an ice-like layer at 0–30% RH; from 30–60% RH begins to form a transitional layer and later morph into liquid form at more than 60% RH.

In the present study, the µTBS values significantly increased by light curing except for the W groups (untreated). The µTBS of LC (0.1 or 0.2 MPa) groups showed stable performance after one week. The overall µTBS results of this current study suggest that the chemical cure alone still is insufficient to obtain stable µTBS of this current study suggest that the ambient air affected the cement layer over chemical curing mode, which corresponds with the manufacturers’ claims that the dual cure resin cement is produced to work devoid of any light source.

Within the limitations of the current study, investigation of the bonding performance of CRBs with bovine dentin concluded that the bond strength of CRBs influenced by the time elapsed after alumina blasting. Furthermore, further studies should clarify whether bonding performance of CRB/CRB interface might be influenced by “dry storage” at lab conditions, however, observation of mass changes of composite resin at laboratory conditions and/or inspection of short-term storage, e.g. three days should be considered in further studies as well.

CONCLUSION

The one-week “dry storage” after alumina blasting will reduce the bonding strength, while the light curing mode minimized the negative impacts of time elapsed after alumina blasting.

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