INTRODUCTION

Over the last few years, computer-aided design/computer-aided manufacturing (CAD/CAM) has gained popularity and showed a dominance in dental applications. This was due not only to CAD/CAM systems having many advantages, including standardized manufacturing processes of dental restorations, but also to the advantages of CAD/CAM technology that led to increases in the adaptation of dental prostheses prepared by the milling of machinable restorative blocks, such as ceramics (e.g. yttrium tetragonal zirconia polycrystals, lithium disilicate glass ceramics) and metals (e.g. titanium, cobalt and chromium alloy). Due to increases in patient demand for esthetic restorations, dentists are focusing on ceramics, since ceramics have a successful natural-looking appearance, excellent mechanical and optical properties, chemical stability and biocompatibility.

However, ceramics require additional processing after milling, necessitating specialized equipment for firing and glazing.

New materials for CAD/CAM, namely resin-ceramic hybrid materials (CAD/CAM resins), have recently been developed, which do not necessarily require additional processing after milling and can be generally finished with a readily available armamentarium in the dental office and thus meet the requirements for one-day treatment.

Studies have been performed to investigate the bonding performance of a resin cement adhesive system consisting of an adhesive and a dual-curable resin cement to the corresponding CAD/CAM resin block by using the bonded specimens, which have been prepared by supplying the light energy from the top surface of the dual-curable resin cement placed on the CAD/CAM resin block. However, the degradation behavior of the bond strengths was strongly affected by the types of adhesives and initiator systems utilized. The adhesive consisting of a dimethacrylate monomer and redox-initiators enhanced the bonding performance of the dual-curable resin cement more effectively than the adhesive, which consists of a dimethacrylate monomer and photo-initiators or a γ-me thacryloxypropyltrimethoxysilane and a 10-methacryloyloxydecyl dihydrogen phosphate.

Keywords: Resin-ceramic hybrid material for CAD/CAM, Resin cement adhesive system, Amount of light energy transmitted, Resin primer, Silane primer

Resin-ceramic hybrid materials for computer-aided design/computer-aided manufacturing (CAD/CAM resins) have been developed. In this study, the effects of the amount of light energy transmitted through the four types of 1.5-mm-thick CAD/CAM resin blocks on the bond performance of corresponding resin cement adhesive systems consisting of an adhesive and a dual-curable resin cement were examined. The bond strengths of the four types of resin cement adhesive systems decreased with decreasing the amount of light energy transmitted through CAD/CAM resin block, due to a decrease in the light-curable ability of dual-curable resin cements. However, the degradation behavior of the bond strengths was strongly affected by the types of adhesives and initiator systems utilized. The adhesive consisting of a dimethacrylate monomer and redox-initiators enhanced the bonding performance of the dual-curable resin cement more effectively than the adhesive, which consists of a dimethacrylate monomer and photo-initiators or a γ-me thacryloxypropyltrimethoxysilane and a 10-methacryloyloxydecyl dihydrogen phosphate.

Keywords: Resin-ceramic hybrid material for CAD/CAM, Resin cement adhesive system, Amount of light energy transmitted, Resin primer, Silane primer

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and as a result, decrease the light-curable ability of monomer components employed in both adhesive and dual-curable resin cement16).

The aim of this study was therefore to determine the amount of light energy that passes through CAD/CAM resin blocks and to examine the effects of the light energy that reaches the resin cement adhesive system consisting of an adhesive and a dual-curable resin cement that exists under the CAD/CAM resin block on the bond performance. The null hypotheses tested were that: 1) the amount of light energy reaching the dual-curable resin cement that exists underneath the CAD/CAM resin block has no effect on the bonding performance, and 2) the type of adhesives for resin cement adhesive systems has no effect on the bonding performance of the dual-curable resin cement.

### Table 1

Four types of resin-ceramic hybrid materials for CAD/CAM (CAD/CAM resins) used and the corresponding resin cement adhesive systems, consisting of an adhesive and a dual-curable resin cement

| Manufactory                  | CAD/CAM resin          | Silica filler content [ ]: Particle size* | Resin cement adhesive system | Code of bonding system |
|-----------------------------|------------------------|------------------------------------------|------------------------------|------------------------|
| Kuraray Noritake Dental     | Katana Avencia Block   | 62 mass% [0.04 μm]                        | Clearfil Ceramic Primer Plus (CPP) | KA bonding system      |
| GC                          | Cerasmart270 (CE)      | 77 mass% [0.1–0.8 μm]                     | G-Multi Primer (GMP)         | CE bonding system      |
| Shofu                       | Shofu Block HC (HC)    | 61 mass% [1.2–9.8 μm]                     | HC Primer (HCP)              | HC bonding system      |
| Tokuyama Dental             | Estelite Block (ES)    | 75 mass% [0.2 μm]                         | Tokuyama Bondmer Lightless (TBL) | ES bonding system      |

( ): Abbreviation.

*: According to Abe et al.8) and Yoshihara et al.26).

### Table 2

Adhesives used and the components for each bonding system

| Bonding system | Adhesive | Category | Code | Components |
|----------------|----------|----------|------|------------|
| KA             | Silane   | CPP      | γ-MPTS, MDP, Ethanol |
| CE             | primer   | GMP      | γ-MPTS, MDP, MDTP, Methacrylic acid ester monomer, Ethanol |
| HC             | HCP      |          | UDMA, MMA, Photo-initiator*, Acetone and Other |
| ES             | Resin    | TBL      | Primer A: Bis-GMA, TEGDMA, HEMA, Phosphoric acid monomer, MTU-6, Acetone and Other |
|                | primer   |          | Primer B: Silane coupling agent, Borate-based catalyst, Peroxide, Acetone, Isopropanol, Water and Other |

γ-MPTS: γ-methacryloxypropyltrimethoxysilane; MDP: 10-methacryloxyloxydecyl dihydrogen phosphate; MDTP: 10-methacryloxyloxydecyl dihydrogen thiophosphate; UDMA: Urethane dimethacrylate; MMA: Methyl methacrylate; Bis-GMA: 2,2-bis[4-(2-hydroxy-3-methacryloxy-propoxy)-phenyl] propane; TEGDMA: Triethyleneglycol dimethacrylate; HEMA: 2-hydroxyethyl methacrylate; MTU-6: 6-methacryloxyhexyl 2-thiouracil-5-carboxylate.

*: According to the manufacturer20).

### MATERIALS AND METHODS

#### Materials

In this study, four types of CAD/CAM resin blocks and corresponding resin cement adhesive systems consisting of an adhesive and a dual-curable resin cement were used (Table 1). The four types of CAD/CAM resin blocks used were the Katana Avencia Block (KA; Lot No. 000632, Kuraray Noritake Dental, Osaka, Japan), the Cerasmart270 (CE; Lot No. 1702141, GC, Tokyo, Japan), the Shofu Block HC (HC; Lot No. 0317420, Shofu, Kyoto, Japan) and the Estelite Block (ES; Lot No. 0120Z7, Tokuyama Dental, Tokyo, Japan). The shade of each resin block was A3-LT.

Furthermore, the four types of resin cement adhesive systems used that corresponded to each CAD/CAM resin block were the KA bonding system (Kuraray Noritake Dental) consisting of Clearfil Ceramic Primer Plus (CPP, Lot No. 1J0023) and Panavia V5 (Universal,
Lot No. 880060), the CE bonding system (GC) consisting of G-Multi Primer (GMP, Lot No. 1701251) and G-Cem Linkforce (A2, Lot No. 1701181), the HC bonding system (Shofu) consisting of HC Primer (HCP, Lot No. 031603) and Block HC Cem (Ivory, Lot No. 021605), and the ES bonding system (Tokuyama Dental) consisting of Tokuyama Bondmer Lightless (TBL, Lot No. 003067) and Estecem (Universal, Lot No. A0083). The components of the four types of adhesives used are listed in Table 2. In accordance with previous studies\(^8,17,18\), we classified the adhesives used for the four types of dual-curable resin cements into two categories based on their monomer components. The CPP for the KA bonding system and GMP for the CE bonding system, which consists of a silane coupling agent, such as a \(\gamma\)-methacryloxypropyl trimethoxysilane (\(\gamma\)-MPTS) and an acidic component, such as a 10-methacryloyloxydecyl dihydrogen phosphate (MDP), are classified into a silane primer. The HCP for the HC bonding system and TBL for the ES bonding system, which consists of a dimethacrylate monomer (2,2-bis [4-(2-hydroxy-3-methacryloyloxy-propoxy)-phenyl) propane, triethyleneglycol dimethacrylate and/or urethane dimethacrylate) and photo- or redox-initiators, are classified into a resin primer.

The G-Light Prima (GC) was used as the LED light source. The intensity of LED light of the G-Light Prima, measured using a L.E.D.RADIOMETER (Kerr, Brea, CA, USA), was 1,200 mW/cm\(^2\).

**Methods**

1. Preparation of CAD/CAM resin plates

Four types of CAD/CAM resin (KA, CE, HC and ES) blocks were sectioned in plates of 2.5-mm thickness using a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). Both surfaces of each CAD/CAM resin block were ground using a sequence of 100-, 400-, 800- and 1000-grit silicon carbide papers under a running water, and the thickness of each resin block was then fixed to 1.50±0.01 mm. This is because manufacturers regulate the thickness of CAD/CAM resin crowns at the occlusal surface of molars to more than 1.5 mm to avoid the fracture of resin crowns seated on abutment teeth\(^19-21\). Thereafter, the sliced resin blocks were rinsed with distilled and deionized water for 20 min using an ultrasonic cleaner and were then dried at room temperature (24ºC) for 1 h.

2. Determination of the amount of light energy at 468 nm passing through the CAD/CAM resin block

The light intensity passing through each 1.5-mm-thick CAD/CAM resin block was measured under a safe-light (630 nm, Heiland LED Darkroom Light, LED-LGT, Heiland electronic, Wetzlar, Germany) as a function of the light irradiation time using a Power max PM3 (COHERENT, Santa Clara, CA, USA) equipped with a sensor to detect light intensity (Fig. 1a). The wave number of the sensor to detect the light intensity of the G-Light Prima was set at 468 nm, since that is the maximum absorbance of camphorquinone, which plays a key role in the photo-initiator system\(^13\). The light intensity was detected 10 times per second using a Lab max pro (COHERENT) and the irradiation time of the LED light was 20 s.

Considering clinical situations, the experimental groups were designed as shown in Fig. 1b.

**Fig. 1**  The experimental apparatus used (1a) and study designs for measuring the transmittance of LED light at 468 nm passing through 1.5-mm-thick CAD/CAM resin blocks (1b). ①: Exit window head for the LED light; ②: 1.5-mm-thick CAD/CAM resin block; ③: Acrylic plate; ④: 2.0-mm-thick spacer. Group 1: The exit window head for the LED light was positioned perpendicularly on top and in direct contact (0 mm) with the four types of 1.5-mm-thick CAD/CAM resin blocks; Group 2: The exit window head for the LED light was positioned at a distance of 2.0 mm from the upper surface of the four types of 1.5-mm-thick CAD/CAM resin blocks; Control: The exit window head for the LED light was positioned at a distance of 1.5 mm from the top surface of the Power max PM3 without any CAD/CAM resin block.
was directly contacted with the upper surface of the four types of 1.5-mm-thick CAD/CAM resin blocks (a distance of 0 mm). The light intensity at 468 nm that passed through the resin block was then measured as a function of the irradiance time (20 s).

Group 2: The exit window head for the LED light was positioned at a distance of 2.0 mm from the upper surface of the four types of 1.5-mm-thick CAD/CAM resin blocks (a distance of 2.0 mm). The light intensity at 468 nm that passed through the resin block was then measured as a function of the irradiance time (20 s).

As a control, the exit window head for the LED light was positioned at a distance of 1.5 mm from the top surface of the Power max PM3 without any CAD/CAM resin block. The light intensity at 468 nm was then measured as a function of the irradiance time (20 s).

The number of samples used for each experimental group was 6. The observation of light intensity was performed 1 time for each specimen. The amount of light energy that had passed through the four types of 1.5-mm-thick CAD/CAM resin blocks was determined by calculating the area under the obtained transmittance curve, respectively.

The reduction was then determined by using a following equation: Reduction=100×(1–the ratio of the light energy). There, the ratio of the light energy was calculated by dividing the amount of light energy transmitted through the four types of 1.5-mm-thick CAD/CAM resin blocks determined in Groups 1 and 2 by the amount of light energy, 9.44 J/cm² determined in the absence of any resin block as a control, assuming that the amount of light energy determined as a control represents the amount of light energy of 100%.

3. Preparation of bonded specimens for adhesion test

The four types of 1.5-mm-thick CAD/CAM resin blocks were cross-sectioned into a width of 10 mm and a length of 12 mm by a low-speed diamond saw under a stream of water and the corners of each cut resin block were then trimmed to a round shape by a carbide bur. One side of both polished surfaces of the four types of CAD/CAM resin blocks was alumina-blasted by using a laboratory sandblaster (Jet Blast III, Morita, Tokyo, Japan) and a COBRA 25 µm (Renfert, Hilzingen, Germany). The device was used at an air pressure of 0.15 MPa for 10 s and was positioned at a distance of 10 mm from the target surface. There, we used a COBRA 25 µm whose ds-50, ds-3 and ds-94 values are 29.2±1.5, 49 (maximum) and 16.5 (minimum) µm, since GC. recommends a use of alumina whose particle size is 25–50 µm\(^2\). Thereafter, the blasted surface of each CAD/CAM resin block was rinsed with distilled and deionized water for 10 min using an ultrasonic cleaner and was then dried at room temperature.

After 1 h, 80-µm-thick double-faced tape with a circular hole (internal diameter=\(\phi 3.2\) mm, Nichiban, Tokyo, Japan) was placed at the central portion of the blasted surface of each CAD/CAM resin block under a safe-light at room temperature. The adhesive for each resin cement adhesive system was applied to the blasted resin surface within the circular hole and the adhesive-coated surface was then air-dried with a high-pressure airflow for 10 s in accordance with manufacturer's instructions. After a 1-mm-thick silicone ring mold with a circular hole (internal diameter=\(\phi 3.2\) mm) was mounted on the double-faced tape placed, the corresponding dual-curable resin cement was immediately condensed on the adhesive-coated surface inside the hole through the mixing tip. The surface of the mixed resin cement was pressed by a glass plate after being covered with a matrix strip.

Immediately thereafter, the four types of dual-
curable resin cements were cured with or without LED light irradiation, as shown in Fig. 2.

Group 1: The exit window head for the LED light was directly contacted with the upper surface of the four types of 1.5-mm-thick CAD/CAM resin blocks. Immediately thereafter, the LED light was irradiated to the adhesive and corresponding dual-curable resin cement through the resin block for 20 s.

Group 2: The exit window head for the LED light was positioned at a distance of 2.0 mm from the upper surface of the four types of 1.5-mm-thick CAD/CAM resin blocks. Immediately thereafter, the LED light was irradiated to the adhesive and corresponding dual-curable resin cement through the resin block for 20 s.

Group 3: After the dual-curable resin cement was condensed within the hole of the silicone ring mold through the mixing tip, its surface was covered with a matrix strip and then pressed by a glass plate. Each sample was kept at room temperature under a safe-light for 10 min to self-cure the dual-curable resin cement by using its chemical-curable property (non-light irradiation group).

As a control, the exit window head for the LED light was positioned perpendicularly on top and in direct contact (0 mm) with the four types of dual-curable resin cements filled into the hole of the silicone ring mold. Immediately thereafter, the LED light was directly irradiated to the adhesive and corresponding dual-curable resin cement for 20 s.

In the light irradiation groups (Groups 1 and 2 and the control), the bonded specimens were kept at room temperature after light irradiation for 10 min under a safe-light. In the non-light irradiation group (Group 3), the bonded specimens were left at room temperature for 10 min under a safe-light after the dual-curable resin cement was immediately condensed on the adhesive-coated surface inside the hole through the mixing tip. After this, the mold and tape were removed and the bonded specimens were then immersed in water at 37°C for 24 h.

The ratio of the bond strength of each resin cement adhesive system to the corresponding CAD/CAM resin block was then determined by using the following equation: \( \text{Ratio}=100\times\left(\frac{\text{Bond strength of the Groups 1, 2 or 3}}{\text{Bond strength of the respective control}}\right) \), assuming that the bond strength determined as a control represents a bond strength of 100%.

4. Measurement of shear bond strength

After the bonded specimens were stored at 37°C in water for 24 h, the bonded specimen was adhered to the stainless steel rod (\( \phi 12 \times 15 \) mm) by using an instant adhesive (Aron Alpha, Toagosei, Tokyo, Japan). The specimen was set and held on our designed apparatus (Fig. 3). The shear bond strengths of the four types of resin cement adhesive systems to the corresponding CAD/CAM resin blocks were measured using a universal testing machine (TG-5KN, Minebea, Nagano, Japan). The cross-head speed was 1.0 mm/min. The number of specimens used for each experimental group was 15.

5. Determination of fracture type

To determine the fracture type of 15 specimens in each experimental group, the fractured surfaces of the CAD/CAM resin blocks and the corresponding resin cements were observed using a light microscope (Leica M60, Leica, Wetzlar, Germany). As shown in Fig. 4 (magnification: \( \times 32 \)), the fracture type of each sample was classified into five categories [0/1/2/3/4]. Consequently, a Category 0 is an interfacial failure where no resin cement remains on the resin block surface, a Category 1 is a mixed failure where less than 1/2 of the resin cement remains on...
the resin block surface, a Category 2 is a mixed failure where less than 1/2 of the resin block is fractured at the bonding interface, a Category 3 is a mixed failure where more than 1/2 of the resin block is fractured at the bonding interface, and a Category 4 is a cohesive failure where resin block is completely fractured at the bonding interface.

6. Statistical analysis
For statistical analysis, the assumption of normality was tested with Kolmogorov-Smirnov and Shapiro-Wilk statistics. The effects of the distance of the exit window head for the LED light from the upper surface of the 1.5-mm-thick CAD/CAM resin block and of the type of CAD/CAM resin blocks on the amounts of light energy transmitted were analyzed using one-way analysis of variance (ANOVA) and Bonferroni multiple comparison tests. The results of bond strengths were analyzed using two-way ANOVA and Bonferroni multiple comparison tests. Furthermore, statistical differences in the fracture types were investigated by chi-square tests.

Additionally, to clarify the effect of the amount of light energy transmitted on the degradation rate of the bond strengths of the four types of resin cement adhesive systems, the regression line between the reduction of the amount of light energy and the mean bond strength of each resin cement adhesive system was determined using a least-square method, respectively. The results of regression slopes of four types of bonding systems were compared using Bonferroni multiple comparison tests.

All statistical analysis was performed using the SPSS 12.0 package. The significance level was defined as \( p < 0.05 \).

RESULTS

Typical transmittance curves of LED light at 468 nm through the four types of 1.5-mm-thick CAD/CAM resin blocks by varying the distance of the exit window head of the LED light from the top surface of the resin blocks

Figure 5 shows the typical transmittance curves of light at 468 nm that passed through the four types of 1.5-mm-thick CAD/CAM resin blocks as a function of the irradiation time. As a control, we combined the typical curve of light intensity at 468 nm that had been determined in the absence of the resin block (black solid line in Fig. 5a).

The interposition of the four types of 1.5-mm-thick CAD/CAM resin blocks in the optical path for the LED light decreased the light intensity at 468 nm (colored solid lines in Figs. 5a and b), even the exit window head for the LED light unit was directly contacted with the resin block surface (a distance of 0 mm). An increase in the distance of the exit window head from the resin block surfaces to 2.0 mm allowed further decreases in light intensity, respectively (colored dotted lines in Fig. 5b).

To clarify the effect of the type of CAD/CAM resin blocks on the amount of light energy transmitted, we determined the amounts of light energy that passed through the four types of 1.5-mm-thick CAD/CAM resin blocks by integrating the transmittance curves of

![Fig. 5](image-url) Typical transmittance curves of LED light at 468 nm through the four types of 1.5-mm-thick CAD/CAM resin blocks (5a and 5b). Black: Without a CAD/CAM resin block (Control); Purple: With the KA block; Red: With the CE block; Orange: With the HC block; Blue: With the ES block. Solid lines: The distance of the exit window head of the LED light from the upper surface of the four types of CAD/CAM resin blocks is 0 mm; Dotted lines: The distance of the exit window head of the LED light from the upper surface of the four types of CAD/CAM resin blocks is 2 mm.
Table 3  The amount of light energy at 468 nm passing through the four types of 1.5-mm-thick resin-ceramic hybrid materials for CAD/CAM (CAD/CAM resins) (Unit: J/cm²) and their reductions of the amount of light energy (Unit: %)

| Control*  | CAD/CAM resin | Experimental group** |  |  |
|-----------|---------------|---------------------|---|---|
|           | Amount of light energy | Reduction***  |  |  |
|           | Group 1: 0 mm |  | Group 2: 2 mm |
| KA 9.44 (0.12)a | 0.90 (0.01)bA | 90.5 | 0.76 (0.01)cA | 91.9 |
| CE 1.26 (0.02)bB | 1.03 (0.02)cB | 89.1 |
| HC 1.35 (0.03)cC | 1.14 (0.02)cD | 87.9 |
| ES 1.09 (0.01)bD | 0.89 (0.02)cD | 90.6 |

*: The LED light was exposed without a CAD/CAM resin block after the exit window for the LED light was positioned at a distance of 0 mm.
**: The LED light was exposed through the CAD/CAM resin block after the exit window for the LED light was positioned at a distance of 0 or 2 mm from the upper surface of the 1.5-mm-thick CAD/CAM resin block.
***: The reduction was determined by using a following equation: Reduction=100×(1–the ratio of the light energy). There, the ratio of the light energy was calculated by dividing the amount of light energy transmitted through the four types of 1.5-mm-thick CAD/CAM resin blocks determined in Groups 1 and 2 by the amount of light energy, 9.44 J/cm² determined in the absence of any resin block as a control, assuming that the amount of light energy determined as a control represents the amount of light energy of 100%.

The different superscript characters (a–c) at each horizontal line indicate a significant difference in the amount of light energy in each experimental group and the different subscript characters (A–D) at each vertical column indicate a significant difference in the amount of light energy among the four types of CAD/CAM resin blocks (p<0.05).

The number of specimens was 6.

( ): SDs.

The light at 468 nm (Table 3). The four types of resin blocks significantly reduced the amount of light energy transmitted from 9.44 to 0.90 J/cm² for KA, 1.26 J/cm² for CE, 1.35 J/cm² for HC and 1.09 J/cm² for ES, respectively (p<0.05), even the exit window head for the LED light unit was directly contacted with the resin block surface (a distance of 0 mm). There were significant differences in the optical transparencies observed among four types of CAD/CAM resin blocks (p<0.05).

An increase in the distance of the exit window head from the CAD/CAM resin block surfaces to 2.0 mm led to further decreases in the amount of light energy (p<0.05). The optical transmittances of the four types of resin blocks ranged from 0.76 to 1.14 J/cm². There were significant differences in the optical transparencies observed among four types of CAD/CAM resin blocks (p<0.05).

Shear bond strength of the four types of resin cement adhesive systems to corresponding CAD/CAM resin blocks

Table 4 shows the mean shear bond strength values (MPa), standard deviations (SD) of the four types of resin cement adhesive systems to corresponding CAD/CAM resin blocks.

Interposition of the four types of 1.5-mm-thick CAD/CAM resin blocks (at a distance of 0 mm) and thus a reduction in the amount of light energy reaching the corresponding resin cement adhesive system by approximately 88% decreased the initial bond strengths by from 17 to 46%, respectively (p<0.05). There were significant differences in the fracture type of four types of resin cement adhesive systems observed between the experimental group 1 and the control, respectively (p<0.05, Table 5).

Further decreases in the amount of light energy reaching the resin cement adhesive system from approximately 88 to 100% allowed further reductions in the bond strengths of the HC and ES bonding systems from 11.6 to 1.1 MPa and from 15.8 to 11.7 MPa (p<0.05). The predominant fracture type of the HC and ES bonding systems changed from a Category 2 to Category 0 or a Category 1, respectively (p<0.05). The HC (γ=−76.104x+77.270, R²=0.899) and ES (γ=−37.224x+48.916, R²=0.323) bonding systems showed a linear correlation between the amount of light energy and the bond strength, respectively (Table 6). However, we did not observe a significant difference in the bond strengths between experimental groups 1 (0 mm) and 2 (2 mm) in terms of the distance of the exit window head from the resin block surface. The ES bonding system showed significantly different effects of the amount of light energy on the bonding performance and on the fracture type from the HC bonding system (p<0.05).

In contrast, the KA and CE bonding systems a constant value in the bond strengths at approximately 3 and 7 MPa, respectively, even the amount of light energy was decreased to zero. The CE bonding system showed a greater bonding performance than the KA bonding system and exhibited different fracture types...
Table 4  Shear bond strengths of the four types of bonding systems to the respective 1.5-mm-thick resin-ceramic hybrid material for CAD/CAM (CAD/CAM resins) with or without light irradiation (Unit: MPa) and their ratio of the mean bond strength (Unit: %)

| Bonding system | Control* | Experimental group** | Non-irradiation group |
|----------------|----------|----------------------|-----------------------|
|                | Bond strength | Irradiation group | Non-irradiation group |
|                | | Group 1: 0 mm | Group 2: 2 mm | |
|                | Bond strength | Ratio*** | Bond strength | Ratio*** | Bond strength | Ratio*** |
| KA             | 5.9 (2.5)a  | 3.3 (0.9)b   | 55.9 | 3.2 (0.7)b   | 54.2 | 3.2 (0.6)b   | 54.2 |
| CE             | 13.0 (4.2)b | 7.0 (1.4)b   | 53.8 | 7.2 (0.7)b   | 55.4 | 7.1 (1.7)b   | 54.6 |
| HC             | 17.8 (3.6)c | 11.6 (1.9)c  | 65.2 | 10.9 (2.0)c  | 61.2 | 1.1 (0.5)c   | 6.2  |
| ES             | 19.0 (4.3)c | 15.8 (2.3)d  | 83.2 | 15.4 (2.6)d  | 81.1 | 11.7 (3.4)c  | 61.6 |

*: The LED light was exposed after the exit window for the LED light was positioned at a distance of 0 mm from the upper surface of the dual-curable resin cement.

**: The LED light was exposed to the four types of dual-curable resin cements through the corresponding 1.5-mm-thick CAD/CAM resin blocks after the exit window for the LED was positioned at a distance of 0 or 2 mm from the upper surface of the resin block.

***: The ratio of the bond strength of each resin cement adhesive system to the corresponding CAD/CAM resin block was then determined by using a following equation: Ratio=100×(Bond strength of the Group 1, 2 or 3/Bond strength of the respective control), assuming that the bond strength determined as a control represents a bond strength of 100%.

The different superscript characters (a–c) at each horizontal line indicate a significant difference in the mean bond strength in each experimental group and the different subscript characters (A–D) at each vertical column indicate a significant difference in the mean bond strength among four types of bonding systems (p<0.05).

The number of specimens was 15.

( ): SDs.

Table 5  Fracture type of bonded specimens with or without light irradiation (Category: [0/1/2/3/4])

| Bonding system | Control* | Experimental group** | Non-irradiation group |
|----------------|----------|----------------------|-----------------------|
|                | | Irradiation group | Non-irradiation group |
|                | | Group 1: 0 mm | Group 2: 2 mm | Group 3 |
| KA             | [10/5/0/0/0]a | [13/2/0/0/0]a | [15/0/0/0/0]a | [15/0/0/0/0]a |
| CE             | [3/4/8/0/0]b | [7/5/3/0/0]b | [7/6/2/0/0]b | [9/3/3/0/0]b |
| HC             | [0/0/3/11/1]c | [0/0/14/1/0]c | [0/1/13/1/0]c | [15/0/0/0/0]c |
| ES             | [0/0/7/8/0]c | [0/1/10/4/0]d | [0/4/7/4/0]d | [0/12/2/1/0]d |

*: The LED light was exposed after the exit window for the LED light was positioned at a distance of 0 mm from the upper surface of the dual-curable resin cement.

**: The LED light was exposed to the four types of dual-curable resin cements through the corresponding 1.5-mm-thick CAD/CAM resin blocks after the exit window for the LED was positioned at a distance of 0 or 2 mm from the upper surface of the resin block.

Fracture type: [0/1/2/3/4]; A Category 0 is an interfacial failure where no resin cement remains on the resin block surface, a Category 1 is a mixed failure where less than 1/2 of the resin cement remains on the resin block surface, a Category 2 is a mixed failure where less than 1/2 of the resin block is fractured at the bonding interface, a Category 3 is a mixed failure where more than 1/2 of the resin block is fractured at the bonding interface, and a Category 4 is a cohesive failure where resin block is completely fractured at the bonding interface.

The number of specimens was 15.

The number of each column in [0/1/2/3/4] shows the number of specimens in each category.

The different superscript character (a–d) at each horizontal line indicate a significant difference in the fracture type in each experimental group and the different subscript characters (A–D) at each vertical column indicate a significant difference in the fracture type among four types of bonding systems (p<0.05).
The dead spaces are developed when the exit window head for the LED light unit is contacted with the buccal and lingual and occlusal surfaces of the CAD/CAM resin crown milled for the maxillary and mandibular first molars. The areas marked with a pink show the dead spaces developed between the exit window head for LED light unit and the resin crown.

### Table 6  Regression analysis results of the four types of bonding systems

| Bonding system | Intercept (SE), $p$-value $^*$ | $B$** (SE), $p$-value $^*$ | $R^{***}$ |
|----------------|---------------------------------|----------------------------|-----------|
| KA             | 3.711 (2.397), $p$=0.129        | $-0.502 (2.544)^*$, $p$=0.844 | 0.001     |
| CE             | 7.429 (3.086), $p$=0.020        | $-0.343 (3.351)^*$, $p$=0.919 | <0.001    |
| HC             | 77.270 (3.555), $p$<0.001       | $-76.104 (3.889)^*$, $p$<0.001 | 0.899     |
| ES             | 48.916 (7.661), $p$<0.001       | $-37.224 (8.225)^*$, $p$<0.001 | 0.323     |

$^*$: The statistical significance of regression coefficients (Intercepts and Slopes).

$^{**}$: Slope; Degradation rate of the bond strengths in each resin cement adhesive system corresponding to the reduction of the amount of light energy transmitted.

$^{***}$: Coefficient of determination.

The different superscript characters (A–C) at vertical column indicate a significant difference in the slope of regression line among four types of bonding systems ($p$<0.05).

SE: Standard error.

from the KA bonding system ($p$<0.05). The KA and CE bonding systems showed different effects of the amount of light energy of the bond strengths from the HC and ES bonding systems.

The ES bonding system, in each experimental group, showed greater bond strengths than other three types of bonding systems and had significantly different fracture types from other three types of bonding systems ($p$<0.05).

**DISCUSSION**

In this study, the position of the exit window head for the LED light unit was set at the 2-mm higher portion than the top surface of the CAD/CAM resin block to simulate the clinical situation for CAD/CAM resin restoration. This is because the dead space of approximately 2 mm will be developed at the path way of the LED light to the fissure portion of the CAD/CAM resin crown at the direct contact of the exit window head with the cusps in the occlusal surface (distance of 0 mm), if the occlusal surface of the CAD/CAM resin crown was properly reproduced, referring the configuration of the maxillary and mandibular first molars of Japanese without the groove and fossa portions (Fig. 6). This consideration is possible since the depths developed from the standard tricuspal plane to the central fossa for the maxillary and mandibular first molars were 2.55±0.23 and 2.30±0.15 mm, respectively. Furthermore, the dual-curable resin cement was chemical-cured without any light exposure and the dual-curable resin cement existed underneath the mesial and distal proximal surfaces of the resin crown in contrast to its buccal and lingual surfaces.

The amount of light energy transmitted through the four types of the 1.5-mm-thick CAD/CAM resin blocks strongly affected the bond strengths and fracture types of the corresponding resin cement adhesive systems. The observed decreases in the bond strengths and alterations in the fracture types were due to decreases in the degree of polymerization conversion of the adhesive and/or dual-curable resin cement, since decreases in the amount of light energy reduced the light-curable ability of monomer components employed in the adhesive and dual-curable resin cement. This consideration is possible since the light-irradiating photo-initiator system has a greater potential to polymerize monomer components utilized in the dual-curable resin cement than the redox-initiator system. Therefore, the first null hypothesis tested had to be rejected.

The KA and CE bonding systems showed different effects of the amount of light energy reaching the corresponding resin cement adhesive systems from the...
HC and ES bonding systems. The observed limitation in their bonding performance was due to the bonding site of the dual-curable resin cement being limited by the γ-MPTS species chemisorbed on the hydroxy -OH group existed at the abraded silica surface, which had been fleshly exposed on the CAD/CAM resin block during an alumina-blasting process.28,29,30. This was possible since the polymer matrix exposed at the blasted KA and CE block surfaces was not a chemisorption site of γ-MPTS species. Previously, our group reported that the efficacy of the experimental silane primer to enhance the bonding of the dual-curable resin cement to the blasted CAD/CAM resin block was strongly affected by the particle size of the silica filler utilized in the resin block.31 The observed result that GMP promoted a greater bond strength of the dual-curable resin cement than CPP may be therefore due to the particle size of the silica filler utilized in the CE block (particle size of the silica filler: 0.1–0.8 µm) being greater than that in the KA block (particle size of the silica filler: 0.04 µm) by three- to twenty-times.32,26,27. This was because an increase in the particle size of the silica filler utilized in the CAD/CAM resin block increased the area of the abraded silica filler surface but also the number of -OH group existed at the abraded silica filler surface.

In contrast to the KA and CE bonding systems, HC and ES bonding systems showed a strong linear correlation between the amount of light energy and the bond strength, respectively. However, the HC bonding system showed a greater slope of the regression line and thus a faster degradation rate in the bond strength than ES bonding system, even the bonding mechanism of both dual-curable resin cements was a micro-mechanical inter-locking of the corresponding resin primers, HCP and TBL to the blasted resin block. This is probably due to a difference in the initiator system to polymerize the monomer components utilized in both adhesives, since HCP is a light-curable resin primer (containing photo-curable initiators)29, but TBL is a chemical-curable resin primer (containing chemical-curable initiators). Therefore, the observed rapid degradation in the bond strength of HC bonding system with decreasing the amount of light energy directly corresponded to decreases in the degree of polymerization conversion of the monomer components utilized in HCP. In contrast, the chemical-curable TBL remained 62% of the initial bond strength of ES bonding system, 11.7 MPa, even the amount of light energy reached zero. Therefore, the observed decreases in the bond strength of ES bonding system with decreasing the amount of light energy may correspond to decreases in the degree of polymerization conversion of the dual-curable resin cement.16,29-31. Therefore, the second null hypothesis had to be rejected.

Our results clearly show that the bonding performance of the resin cement adhesive system consisting of an adhesive and a dual-curable resin cement to the mesial and distal proximal surfaces of the CAD/CAM resin crown may affect the survival rate of the CAD/CAM resin restoration. This is because we are unable to supply the light energy to the adhesive and dual-curable resin cement, which has existed under the proximal surfaces of the CAD/CAM resin crown. To enhance the bonding performance of a dual-curable resin cement to the mesial and distal proximal surfaces of the CAD/CAM resin crown, we should increase the chemical-curable ability of monomer components employed in the resin primer.

CONCLUSION

Adhesives play a key role in the bonding performance of resin cement adhesive systems to the CAD/CAM resin restorations, since the bond strength is strongly affected by the types of adhesive and initiator system utilized. The chemical-curable resin primer provides greater bond strengths than the light-curable resin primer and also silane primers, even with decreasing the amount of light energy reaching the resin cement adhesive system existed under CAD/CAM resin restoration until zero.

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CONFLICT OF INTEREST

The authors report no conflicts of interest.

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