INTRODUCTION

Applications of CAD/CAM systems to dentistry are growing rapidly, especially because these systems can facilitate the fabrication of restorations using machinable materials of various types\(^9\). The classes of materials commonly used in the production of CAD/CAM restorations are glass-ceramics, interpenetrative network materials\(^2\) and resin composites. Glass-ceramics have superior mechanical and esthetic properties, however, the ceramic materials are still susceptible to brittle fracture when subjected to tensile stresses in the oral cavity\(^3\). Moreover, the abrasiveness of these materials against enamel antagonist is still a clinical concern.

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Cementation technique is important to ensure clinical success and longevity of a restoration. In order to establish a strong and durable bond, which is necessary for the biomechanical aspect of the tooth restoration system, appropriate treatment of the respective bonding surfaces is crucial. Achieving a strong durable bond to both the tooth structure and the indirect restoration should be feasible. However, recent machinable resin composite blocks (RCB) have a drawback in that the adhesion to resin cement is often weak since the resin matrix of the RCB is highly cross-linked, resulting in the difficulty for monomer penetration of a resin cement into the RCB polymer network\(^8\). Hence, where maximum adhesive strength is needed due to limited retention form of a preparation, it means dislodgement and/or fracture of the restoration and recurrent caries may occur more easily. Selection of a resin cement for CAD/CAM resin composite restorations is therefore a key to achieve success of such restorations. Hence, improvement of the bonding to the RCB is necessary to ensure clinical longevity of restorations.

Four-methacryloxyethyl trimellitate anhydride (4-META) in methyl methacrylate (MMA) initiated by tri-n-butyl borane (TBB) (4-META/MMA-TBB) is a unique MMA-based adhesive resin cement, which has been widely used clinically for a long time\(^10-12\). It has been shown to be a reliable resin cement for bonding to tooth structure\(^13,14\). A conventional 4-META/MMA-TBB resin cement (Super Bond C&B, SB, Sun Medical, Moriyama, Japan) is composed of a liquid, including a functional monomer (4-META) in MMA with a chemical self-cure initiator (TBB) and a polymethyl methacrylate (PMMA) powder. The molecular weight of MMA (mw=100) is the smallest among the dental polymeric resin methacrylates. 4-META has been reported to promote monomer penetration into tooth structures\(^15\) and also chemically react with hydroxyapatite\(^16-18\). The TBB catalyst can promote interfacial polymerization at the enamel and dentin substrates in the presence of intrinsic water\(^19\). Therefore, the 4-META/MMA-TBB resin is able to provide good adhesion to tooth structures and dental restorative materials, such as metal alloys, ceramics and resin composites.

Recently, a rapid cure type of Super Bond C&B (SBQ; Sun Medical) was developed and launched onto the market. SBQ contains a newly developed liquid (Quick Monomer), containing MMA, 4-META and a multifunctional methacrylate.

The purpose of this study was to evaluate the early tensile bond strengths of three different resin cements; Super Bond C&B (conventional and quick type) and ResiCem to a CAD/CAM composite block. A CAD/CAM composite block (Shofu Block HC) was ground and silanized according to the manufacturers’ instructions before cementation. A conventional tensile bond strength test (Ø: 4 mm) was performed 10 min, 1 h and 24 h after bonding. Super Bond C&B (quick type) showed the highest bond strength at 24 h.

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The 4-META/MMA-TBB resin is a suitable resin cement to bond to a CAD/CAM composite block. A CAD/CAM composite block (Shofu Block HC) was ground and silanized according to the manufacturers’ instructions before cementation. A conventional tensile bond strength test (Ø: 4 mm) was performed 10 min, 1 h and 24 h after bonding. Super Bond C&B (quick type) showed the highest bond strength at 24 h.

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Early bond strengths of 4-META/MMA-TBB resin cements to CAD/CAM resin composite

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Resin composite is considered preferable for patients who may exhibit grinding or bruxing behavior when a restoration is in contact with natural enamel\(^4,5\). In addition, resin composite materials may offer significant advantages related to their machinability and especially intra-oral reparable which is much simpler than ceramic repair\(^6,7\). The aforementioned factors make resin composite materials a promising option in CAD/CAM dentistry.

Cementation technique is important to ensure clinical success and longevity of a restoration. In order to establish a strong and durable bond, which is necessary for the biomechanical aspect of the tooth restoration system, appropriate treatment of the respective bonding surfaces is crucial. Achieving a strong durable bond to both the tooth structure and the indirect restoration should be feasible. However, recent machinable resin composite blocks (RCB) have a drawback in that the adhesion to resin cement is often weak since the resin matrix of the RCB is highly cross-linked, resulting in the difficulty for monomer penetration of a resin cement into the RCB polymer network\(^8\). Hence, where maximum adhesive strength is needed due to limited retention form of a preparation, it means dislodgement and/or fracture of the restoration and recurrent caries may occur more easily. Selection of a resin cement for CAD/CAM resin composite restorations is therefore a key to achieve success of such restorations. Hence, improvement of the bonding to the RCB is necessary to ensure clinical longevity of restorations.

Four-methacryloxyethyl trimellitate anhydride (4-META) in methyl methacrylate (MMA) initiated by tri-n-butyl borane (TBB) (4-META/MMA-TBB) is a unique MMA-based adhesive resin cement, which has been widely used clinically for a long time\(^10-12\). It has been shown to be a reliable resin cement for bonding to tooth structure\(^13,14\). A conventional 4-META/MMA-TBB resin cement (Super Bond C&B, SB, Sun Medical, Moriyama, Japan) is composed of a liquid, including a functional monomer (4-META) in MMA with a chemical self-cure initiator (TBB) and a polymethyl methacrylate (PMMA) powder. The molecular weight of MMA (mw=100) is the smallest among the dental polymeric resin methacrylates. 4-META has been reported to promote monomer penetration into tooth structures\(^15\) and also chemically react with hydroxyapatite\(^16-18\). The TBB catalyst can promote interfacial polymerization at the enamel and dentin substrates in the presence of intrinsic water\(^19\). Therefore, the 4-META/MMA-TBB resin is able to provide good adhesion to tooth structures and dental restorative materials, such as metal alloys, ceramics and resin composites.

Recently, a rapid cure type of Super Bond C&B (SBQ; Sun Medical) was developed and launched onto the market. SBQ contains a newly developed liquid (Quick Monomer), containing MMA, 4-META and a multifunctional methacrylate.
Early bond strength of a resin cement to a restoration is important to ensure the restoration can become functional almost immediately following placement, especially when the retention form is minimal. However, few studies have focused on early bond strength of resin cements to the machinable resin composite materials. Therefore, the purpose of this study was to evaluate early bond strengths of three resin cements to a RCB material. The null hypothesis of this study was that early bond strengths to a RCB were not influenced by the different type of resin cement.

MATERIALS AND METHODS

Materials used in this study

The materials and their compositions are listed in Table 1. Two self-cure resin cements; Super Bond C&B (SB) and Super Bond quick-type (SBQ) (Sun Medical) and a dual-cure resin cement; Resicem (RC; Shofu, Kyoto, Japan) were used. SB is a conventional 4-META/MMA-TBB resin cement, which is composed of a MMA-based liquid, PMMA powder and TBB catalyst. The liquid contains 4-META in MMA. SBQ is also comprised of a MMA-based liquid, PMMA powder and TBB catalyst, however, the liquid (Quick Monomer) contains an additional multifunctional methacrylate that accelerates polymerization of the cement. RC contains di-methacrylates, such as UDMA and TEGDMA and filler particles.

A RCB, Shofu Block HC (HC; Shofu) was used as the adherend, which contains a high density of filler particles. Pretreatment of the surface of the RCB for each of the cements requires the use of a silane-coupling agent. For SB and SBQ, the coupling agent was PZ Primer (PZ, Sun Medical), while Porcelain Primer (PP, Shofu) was used for RC.

Tensile bond strength (TBS) test

The surface of the RCB (10×12×16 mm) was ground with 600-grit silicon carbide paper under a water stream, then abraded by 50 µm alumina at 0.2 MPa air pressure, and sonicated in distilled water for 2 min. The adhesive area was then demarcated using a double-sided adhesive tape and aluminum foil (approximately 100 µm thickness total) with a 4-mm diameter hole.

For SB and SBQ, the surface was silanized with PZ according to the manufacturer’s instructions. Following this, a stainless-steel rod (6 mm diameter) was bonded to the surface of the RCB with either SB or SBQ by bulk-mix technique according to the manufacturer’s instructions. For RC, the surface of the RCB was silanized with PP, then the resin cement placed to bond the composite rod (6 mm diameter). The bonded assembly was liked cured to facilitate the dual-curing process.

The bonded specimens (n=10) were left at room temperature (23°C) for 10 min, 1 h and 24 h, after which the tensile bond strengths were tested using a universal testing machine (EZ-Test, Shimadzu, Kyoto, Japan) at a crosshead speed of 2 mm/min.

After debonding of the specimens, the failure modes were classified into the following four categories at a magnification of ×125 under a confocal laser scanning microscope:

1. Adhesion failure
2. Cohesive failure
3. Mixed failure
4. Resin/cement failure

Table 1  Materials used in this study

| Code | Material                  | Composition                                      | Manufacturer          |
|------|---------------------------|--------------------------------------------------|-----------------------|
| SB   | Resin cement              | Monomer: MMA, 4-META                             | Sun Medical, Moriyama, |
|      | Super Bond C&B            | Catalyst: TBB                                    | Japan                 |
| SBQ  | Super Bond C&B            | Quick Monomer: MMA, 4-META, Multi-functional methyl acrylate |                        |
|      | Quick type                | Catalyst: TBB                                    |                       |
|      |                            | Polymer: PMMA                                    |                       |
| PZ   | Silane-coupling agent     | A Liquid: MMA, Functional Monomer, others         | Shofu, Kyoto, Japan   |
|      | PZ Primer                 | B Liquid: MMA, Silane                            |                       |
| RC   | Resin cement              | Paste A: UDMA, TEGDMA, Fluoro-alumino-silicate glass, others |                      |
|      | ResiCem                   | Paste B: UDMA, TEGDMA, Fluoro-alumino-silicate glass, 4-AET, HEMA, others | Shofu                  |
| PP   | Silane-coupling agent     | Ethanol, Silane coupling agent, others            |                       |
|      | Porcelain Primer          |                                                   |                       |
| HC   | CAD/CAM resin block       | UDMA, TEGDMA, Silica powder, Silic acid micro particle, Zirconium Silicate | Shofu                  |

4-AET: 4-acryloxyethyltrimellitic acid; 4-META: 4-methacryloxyethyl trimellitate anhydride; HEMA: 2-hydroxyethyl methacrylate; MMA: methyl methacrylate; PMMA: polymethyl methacrylate; TBB: tri-n-butyl borane; TEGDMA: triethyleneglycol dimethacrylate; UDMA: urethane dimethacrylate.
microscope (1LM15W, Lasertec, Yokohama, Japan); A: adhesive failure at the interface between a resin cement and RCB, B: mixed failure including cohesive failure in the resin cement and adhesive failure at the interface between resin cement and RCB, C: mixed failure including cohesive failure in a RCB and adhesive failure at the interface between resin cement and a RCB, and D: cohesive failure in the RCB.

### Three-point bend test

A three-point bend test of the three cured resin cements was performed based on the method described in ISO 4049 Standard for resin-based restorative materials to investigate a relationship between TBS and mechanical property of resin cements. Each resin cement was mixed according to the manufacturers’ instructions, placed in a silicone mold (2×2×25 mm) and pressed flat with a plastic strip and glass plate. For SB and SBQ, the specimens were left for 1 h at room temperature and then stored in an incubator at 37°C for 24 h in distilled water. For RC, the cement specimens were made in the same mold, then light cured using a quartz-halogen light curing unit (Optilux 501, 600 mW/cm², Demetron, Danbury, CT, USA) through the glass plate for 20 s×7 times for each side and left for 24 h at 37°C water storage.

Following this, a three-point bend test was performed using a universal testing machine (Autograph AG-50 kN, Shimadzu) at a crosshead speed of 1 mm/min. Specimens were loaded to failure in case of RC, however for SB and SBQ the specimens only deformed in the center. The deflection was measured to 5 mm after which the test was stopped. Dimensions of the specimens were measured with a digital caliper (CD-15PSX, Mitsutoyo, Kawasaki, Japan). The flexural strength (σ) in MPa was calculated using the following formula:

\[
\sigma = \frac{3Fl}{2bh^2}
\]

where \( F \) is the maximum load (N), \( l \) is support span (20 mm), \( b \) is the width (mm) and \( h \) is height (mm) of the specimen. The flexural modulus (\( E \)) was calculated in GPa using the following formula:

\[
E = \frac{(Fl)^3}{4bh^3d}
\]

where \( d \) is the deflection due to the load \( F \) applied at the middle of the beam (mm). For the SB and SBQ, the loads at the yield point were used to calculate flexural strengths because the specimens were too elastic to be fractured.

### RESULTS

#### Tensile bond strength test

The mean TBS values are shown in Table 2. Two-way ANOVA indicated that the TBS values were influenced by the two factors (time and resin cement) and there was interaction between the two factors. For the 10 min groups, the TBSs of the three resin cements to the RCB were 3–4 MPa, with no significant difference identified among the three cements (\( p > 0.05 \)). For the 1 h and 24 h time periods, the bond strengths of SB and SBQ to RCB were significantly higher than those of RC. RC did not show any change in TBS value over time. The highest bond strength was obtained for SBQ after 24 h which was significantly stronger than the other resin cements tested (\( p < 0.05 \)).

Figure 1 shows the results of the failure mode analysis. Adhesive failure (A) was predominantly observed for SB and SBQ at 10 min. However, mixed failure (M) and cohesive failure in RCB gradually increased as time progressed. After 24 h, cohesive failure in RCB was the major form of failure for the SBQ bonded group.

#### Three-point bending test

Table 3 shows the results of the three-point bending test. RC showed significantly higher flexural strength than the other two resin cements, SB and SBQ (\( p < 0.05 \)).

### DISCUSSION

The CAD/CAM composite block is a mixture of inorganic fillers and liquid monomers, which is molded into a block and then polymerized\(^1\). Generally, the industrial polymerization of a CAD/CAM resin composite is completed under high pressure or heat treatment.
Fig. 1 The results of failure mode analysis. Adhesive failure was dominant for SB and SBQ after 10 min. However, mixed failure and cohesive failure in CAD/CAM block increased over time. After 24 h of SBQ, cohesive failure in CAD/CAM block was the most frequent. A: adhesive failure at the interface between a resin cement and CAD/CAM composite block, B: mixed failure including cohesive failure in cement and adhesive failure at the interface between resin cement and CAD/CAM composite block, C: mixed failure including cohesive failure in CAD/CAM composite block and adhesive failure at the interface between resin cement and CAD/CAM composite block, and D: cohesive failure in CAD/CAM composite block.

Table 3 Flexural strengths (MPa) and flexural modulus (GPa) of the resin cements (SB, SBQ and RC)

|       | Flexural strength (MPa) | Flexural modulus (GPa) |
|-------|-------------------------|------------------------|
| SB    | 47.0±14.0 \(^a\)       | 1.4±0.5 \(^a\)         |
| SBQ   | 50.9±10.6 \(^a\)       | 1.5±0.4 \(^a\)         |
| RC    | 97.1±13.8 \(^b\)       | 6.1±0.4 \(^b\)         |

\(n=5\), mean±SD
Same letter in same column means no significant difference (\(p>0.05\), Games-Howell test).

Burrow et al. investigated early bond strengths to dentin using several resin cements\(^{21}\). They reported that a dual-cure resin cement showed higher \(\mu\)TBSs to dentin at 10 min than the 4-META/MMA-TBB resin cement tested when the resin cement was light cured. Initial polymerization behavior of a dual-cure resin cement can be effectively accelerated for light irradiation, resulting in enhancement of immediate bonding after cementation\(^{24}\). However, the current study demonstrated that the dual-cure resin cement, RC, produced a low TBS from the initial storage time at 10 min up to 24 h (Table 2). RC is a much more viscous cement compared with SB and SBQ. This is due to the dimethacrylate-based resin matrix and filler particles. Therefore, the monomer penetration of RC into the polymer network of the RCB may not be as effective as the more fluid cements, SB and SBQ. On the other hand, SB and SBQ use a MMA-based resin matrix, which may more easily penetrate into the resin matrix of the RCB. The 4-META/MMA-TBB resin cements, SB and SBQ provided initial low bond strengths, being similar to RC, however, by 1 h and 24 h the TBSs to the RCB were significantly stronger than RC. Since SB and SBQ are self-cure resin cements, the polymerization speed may not have sufficiently progressed to provide good bonding to the RCB at the earliest time period. On the other hand, the 4-META/MMA-TBB resin cements may penetrate into the resin matrix and polymerize \textit{in situ} after 1 h and 24 h, which was able to result in the increased TBS observed.

The results of the three-point bend test showed that RC provided higher flexural strength than SB and SBQ, which was believed to be due to the different compositions for each resin cement. RC is a filled resin, while SB and SBQ are both unfilled resin cements. In addition, RC is a dimethacrylate-based resin cement while SB and SBQ are MMA-based resin cements. There seemed to be no relationship between the results for the three-point bend test and the TBS values among RC, SB and SBQ. If anything, there was an inverse relationship. SBQ contains an additional multi-functional methacrylate in the MMA-base monomer, which may enhance the flexural strength of SBQ. In addition, SB and SBQ were too elastic to be fractured. The high degree of plasticity of SB and SBQ may possibly be due to relatively high proportion of the TBB catalyst in the cured cement that is a very unique characteristic of SB and SBQ and enhanced the TBS of SB and SBQ.

From the limited information of the current study,
SB and SBQ seem preferable for bonding to the CAD/CAM RCBs. Therefore, the null hypothesis of this study was rejected. However, the flexural strengths of SB and SBQ are lower than that of the dual cure resin cement, RC. Therefore, if an exposed cement line exists when SB and SBQ are used, this could be regarded as a weak point clinically. Inlay/onlay restorations should perhaps not be luted with SB and SBQ due to the large marginal area that is also more likely to be exposed to wear during function. Further study should be carried out to evaluate the clinical performance of the CAD/CAD resin composite restorations that have been cemented with SB and SBQ.

CONCLUSIONS

Three resin cements (SB, SBQ and RC) demonstrated low bond strengths at 10 min to a RCB. However, SB and SBQ demonstrated higher bond strengths than RC after 1 h and 24 h, in spite of the lower flexural strength of SB and SBQ compared with RC. SBQ provided the highest bond strength at 24 h. The 4-META/MMA-TBB resin cements, SB and SBQ, were regarded as being suitable for luting to a CAD/CAM RCB.

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