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Physico-Mechanical Properties of a Newly Developed Base Material Containing Mineral Trioxide Aggregate

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Abstract: The aim of this study was to evaluate the physico-mechanical properties of a new cavity base material containing mineral trioxide aggregate, LA-T1, prototyped by Neo Dental Chemical Products for indirect restoration. Three base materials, LA-T1, Cavios (CAV, Neo Dental Chemical Products), and Bulk Base Hard (BBH, Sun Medical), were examined. The depth of cure, microshear bond strength with a resin-based luting cement, and the compressive strength of these materials were investigated. The depth of cure of LA-T1 was similar to that of CAV, while the depth of cure of BBH was above the measurement limit. The distance from the base material to the light source, 0 mm and 4 mm, did not significantly affect the depth of cure of all materials. The microshear bond strength of LA-T1 bonded to a resin-based luting cement was slightly higher than that of CAV and similar to that of BBH, both of which were bonded to the same luting cement under the same conditions. The compressive strength of LA-T1 was similar to that of CAV but less than that of BBH. The results of this study indicate that LA-T1 has properties that are similar to those of CAV and thus can be clinically applied.

Keywords: dental base material; depth of cure; compressive strength; microshear bond strength; mineral trioxide aggregate

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1. Introduction

Currently, the principal strategy for the treatment of dental caries involves restoration of the natural tooth structure using synthetic materials after cavity preparation. Because dentin is exposed on the surface of the cavity after the removal of caries-affected dentin, pulp damage due to extraneous stimuli might occur. Nowadays, with improvements in dentin adhesives [1,2], polymerization rates [3], and light-curing devices [4–6], direct restorations using resin-based composites do not require pulp-capping with calcium hydroxide preparations or base application and lining with glass-ionomer cements [7–9].

Indirect restoration is recommended for large cavities and defects with lost cusps in molars [10,11]. In recent years, the development of digital dentistry including computer-assisted design and manufacturing (CAD/CAM) has made it possible to perform esthetic restorations using teeth-colored materials, and the demand for such restorations has been increasing. Dentin tubules are exposed for a certain period of time after cavity preparation for indirect restoration. In addition, the reinforcement of lost defects using base materials is necessary to standardize prepared cavities.

Cavios® (Neo Dental Chemical Products, Tokyo, Japan) is a composite base material composed of α-tricalcium phosphate (α-TCP), an inorganic biomaterial, and urethane dimethacrylate (UDMA), an organic monomer. Cement based on α-TCP can provide pulp protection due to the closure of dentin tubules by crystallization [12–14]. A clinical study reported that applying a Cavios lining/base into caries-removed surfaces, followed by either filling with a resin-based composite or cast metal inlay restoration, removes postoperative discomfort such as cold water pain, suggesting good protection of the dental pulp [15]. Additionally, Cavios can provide excellent pulp protection with no serious damage to the dental pulp tissue and good marginal sealing [16,17]. Cavios is a direct-application syringe-type paste that does not require mixing, photo-polymerizable, and furthermore, provides an X-ray contrast.

Generally, the larger the distance between the curing light tip and material surface, the smaller the depth of cure [18]. Therefore, light-cured restorative materials such as resin-based composites are generally polymerized with light-curing devices placed as close as possible to the material. However, because the lining/base material is generally applied into the deep cavity surface, the curing light tip cannot reach the surface of the base material. Hence, the influence of the distance between the curing light tip and the surface of the base material on the depth of cure should be considered.

Mineral trioxide aggregate (MTA), a hydrophilic inorganic material mainly composed of Portland cement, hardens in the presence of humidity [19] with good biocompatibility [20], sealing ability [21], and mineral induction ability [22,23]. Therefore, it has been used in a variety of procedures such as pulp capping [24,25], perforation repair [26,27], retrograde filling of root canals [28], and root canal filling [29]. However, MTA is known to have poor compressive strength after hardening [30]. Compressive strength might be necessary to withstand occlusal/masticatory forces applied to occlusal cavity surfaces for posterior restorations [31]. In addition, integration between the adherent and indirect tooth-colored restorative material might be needed [32], and thus the adhesion of luting materials to the base material should also be investigated.

Recently, a modified version of Cavios, named LA-T1, was developed by Neo Dental Chemical Products. LA-T1 is composed of MTA instead of α-TCP. The purpose of this study was to compare the physico-mechanical properties, specifically the depth of cure, compressive strength, and microshear bond strength (μSBS) with a resin-based luting cement of LA-T1, Cavios and a commercial bulk-fill resin-based composite base material (Bulk Base Hard, Sun Medical, Moriyama, Shiga, Japan). We hypothesized that base material LA-T1 containing MTA is similar to product Cavios containing α-TCP in (1) depth of cure, (2) compressive strength, and (3) μSBS with resin-based composite luting cements.

2. Materials and Methods

2.1. Materials Used in This Study

Table 1 lists the specifications of the three base materials used in this study: LA-T1 (Neo Dental Chemical Products, Tokyo, Japan), Cavios (CAV; Neo Dental Chemical Products), and Bulk Base Hard® Medium Flow Blue (BBH; Sun Medical). A two-step self-etch adhesive (OptiBond™ eXTRa Universal, Kerr, Orange, CA, USA) and a resin-based composite dual-cured luting cement (Nexus™ Universal, Kerr, Orange, CA, USA) were also used to prepare the μSBS specimens. A light-emitting diode (LED) light-curing device (Pencure, J Morita, Tokyo, Japan) was used for light-curing, and the light intensity was controlled using a hand-held dental radiometer (Bluephase® Meter II; Ivoclar Vivadent, Schaan, Liechtenstein) to ensure that the light output was at least 1100 mW/cm^2.

Table 1. Specifications of the materials used in this study.

Material	Manufacturer	Composition	Batch No.
[Base material]			
LA-T1	Neo Dental Chemical Products (Tokyo, Japan)	UDMA, mineral trioxide aggregate, barium sulfate, CQ, other	LA-T1
Cavios®	Neo Dental Chemical Products (Tokyo, Japan)	UDMA, α-tricalcium phosphate, barium sulfate, CQ, other	B0A1
Bulk Base Hard® (Medium Flow, Blue)	Sun Medical (Moriyama, Shiga, Japan)	methacrylic acid esters (Bis-MPEPP, other), acrylic acid esters (urethane acrylate), barium silica glass, strontium silica glass, aromatic amines, other	TT1
[Dental adhesive]			
OptiBond™ eXTRa Universal	Kerr (Orange, CA, USA)	Primer: GPDM, HEMA, acetone, ethanol, purified water, other Adhesive: GPDM, HEMA, Bis-GMA, glycerol dimethacrylate, ethanol, barium glass, sodium hexafluorosilicate, CQ, other	Primer: 7941589 Adhesive: 7945265
[Resin-based luting cement]			
Nexus™ Universal	Kerr (Orange, CA, USA)	Bis-GMA, TEGDMA, UDMA, HEMA, glycerol dimethacrylate, aluminoborosilicate glass, ytterbium fluoride, other	7509656

Bis-GMA: bisphenol A-glycidyl methacrylate, Bis-MPEPP: 2,2-bis(4-methacryloxy polyethoxyphenyl) propane, CQ: camphorquinone, GPDM: glycerol dimethacrylate dihydrogen phosphate, HEMA: hydroxyethyl methacrylate, TEGDMA: triethylene glycol dimethacrylate, UDMA: urethane dimethacrylate.

2.2. Depth of Cure

A schematic illustration of the specimen preparation is shown in Figure 1. The depth of cure of the investigated materials was analyzed in accordance with the International Organization for Standardization (ISO) 4049 [33]. A reusable cylindrical stainless steel split mold with an orifice of 8 mm in height and 4 mm in internal diameter was placed on a micro glass slide plate (Matsunami Glass, Kishiwada, Osaka, Japan) covered by a transparent polyester strip (Frasaco GmbH, Tettnang, Germany). The mold was then filled in bulk with one of the tested materials. The topside of the mold was covered with a second polyester strip and pressed with a micro glass slide to obtain a flat surface. After the removal of the glass plate, light was irradiated adjacent to the topside of the mold (0 mm) or 4 mm from the topside of the mold with the polyester strip interleaved using an LED light-curing device. As soon as curing was over, the material was removed from the mold, and the unpolymerized part was removed with a plastic spatula. Then, the remaining cured part was measured three times with a digital caliper (CD67-S15PS, Mitutoyo, Kawasaki, Kanagawa, Japan). The mean of the measured value was further divided by two, and these values were recorded as the "depth of cure", as specified by ISO 4049 [33] (n = 10).

2.3. Compressive Strength

A schematic illustration of the specimen preparation is shown in Figure 2. A Teflon™ split mold with a height of 6 mm and internal diameter of 3 mm was placed on a micro glass slide plate (Matsunami Glass) covered by a transparent polyester strip. The mold was then filled in bulk with one of the tested materials. The topside of the mold was covered with a second transparent polyester strip and pressed with a micro glass slide to obtain a flat surface. After removal of the glass plate, light was irradiated onto both the topside and

bottom of the mold for 40 s on each side using an LED light-curing device with the curing light tip positioned directly onto the polyester strip at zero distance (n = 10).

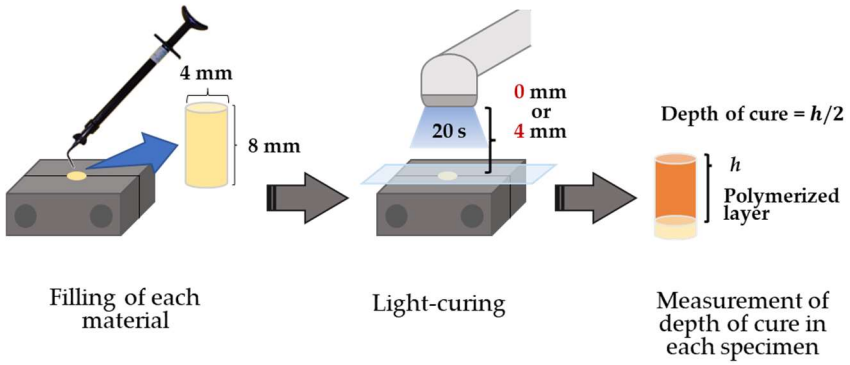

Figure 1. Schematic illustration of the specimen preparation and measurement of the depth of cure.

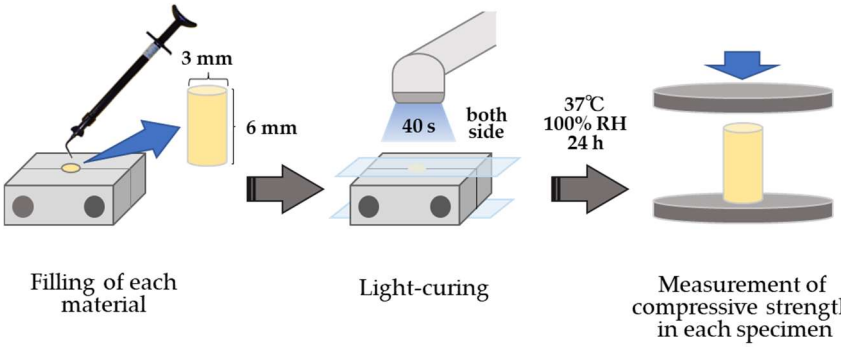

Figure 2. Schematic illustration of the specimen preparation and measurement of the compressive strength. RH: relative humidity.

As soon as the curing was complete, the material was removed from the mold and incubated at 37 °C and 100% relative humidity for 24 h. All samples were individually and vertically mounted on a computer-controlled universal testing machine (SV-301N, Imada Seisakusho, Toyohashi, Aichi, Japan) and tested with a load cell of 500 N at a cross-head speed of 1 mm/min. The maximum failure load was recorded in N and converted into MPa. The compressive strength was calculated from the recorded peak load divided by the sample surface according to the following equation:

$$CS = 4P/\pi d^2 \quad (1)$$

where CS is the compressive strength (MPa); P is the load (N) at the fracture point; and d is the diameter (mm) of the cylindrical specimen.

2.4. μSBS with a Resin-Based Composite Luting Cement

A schematic illustration of the specimen preparation is shown in Figure 3. An acrylic ring mold with a height of 3 mm, internal diameter of 9 mm, and external diameter of 11 mm was placed on a micro glass slide plate (Matsunami Glass) covered by a polyester strip. The ring was then filled in bulk with one of the tested materials. The topside of the mold was covered with a second polyester strip and pressed with a micro glass plate to obtain a flat surface. Then, light was irradiated onto both the topside and bottom with the polyester strip interleaved for 10 s on each side. One of the surfaces was then abraded with #600 waterproof SiC paper (Fuji Star, Sankyo Rikagaku, Okegawa, Saitama, Japan) under a stream of water to prepare a uniform and flat surface. The surfaces of the tested composites were treated for bonding using OptiBond eXTRa Universal. Then, cylindrical transparent Tygon® tubes (Saint-Gobain, Hara Village, Suwa, Nagano, Japan) with an internal diameter

of 1.0 mm, external diameter of 3.0 mm, and height of 1.0 mm were positioned on each treated surface. Nexus Universal were filled into each Tygon tube, and then a transparent polyester strip was placed over the Tygon tube and gently pressed into place. The resin cement was photoactivated for 10 s using an LED light-curing device. The specimens were then incubated at 37 °C and 100% relative humidity for 24 h.

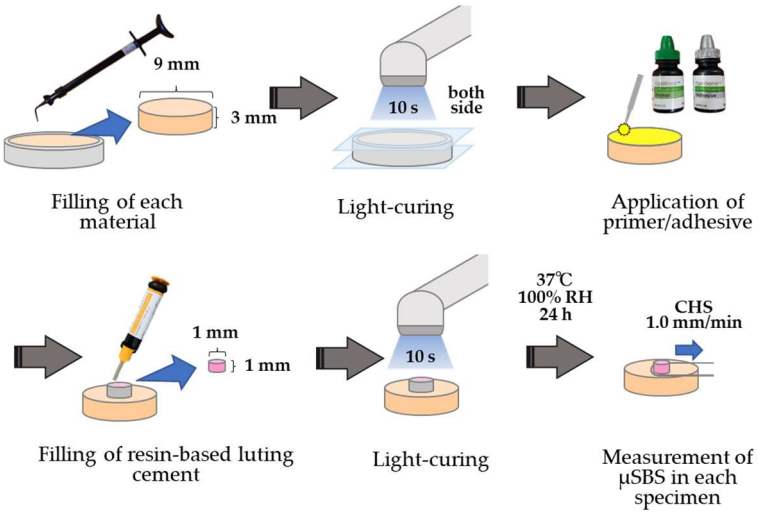

Figure 3. Schematic illustration of the specimen preparation and measurement of the microshear bond strength. RH: relative humidity, CHS: cross-head speed.

Twenty-four hours later, the Tygon tubes were carefully removed, and specimens were then attached to one of the two free-sliding parts of a purpose-built holding device (Micro Tensile Test Jaw, Bisco, Schaumburg, IL, USA) using cyanoacrylate glue (Model Repair II Blue, Dentsply Sirona, Tokyo, Japan). A steel wire (0.25 mm diameter) was fixed to the other free-sliding part and looped around each resin cement cylinder as close as possible to its base, positioned to surround the bottom of each resin cement cylinder. The wire was aligned with the loading axis of the upper movable compartment of the testing machine to ensure the proper distribution of shear load. The μSBS of the specimens were then tested using a Micro Tensile Tester (Bisco) at a cross-head speed of 1 mm/min until failure (n = 9). The μSBS values were calculated using the following equation:

$$\tau = F/\pi r^2 \quad (2)$$

where τ is the microshear bond strength (MPa); r is the radius of the cylinder in mm (0.4 mm); and F is the load (N) at the failure point.

2.5. Statistical Analyses

After the Shapiro–Wilk normality test, the depth of cure data were evaluated statistically with two-way analysis of variance (ANOVA). Data of both compressive strength and μSBS were evaluated statistically with post-hoc Tukey multiple comparisons at a significance level of 0.05.

3. Results

3.1. Depth of Cure

The mean depth of cure and standard deviation (SD) of each group and the results of multiple comparisons are shown in Figure 4. Two-way ANOVA revealed that a significant effect was found for the factor "base material" (p = 0.000) only, and no significant effect was found for the factor "distance from light-curing tip" (p = 0.063). Because a significant interaction was found for the two factors (p = 0.015), multiple comparisons were conducted using the post-hoc Tukey honest significant difference (HSD) test for all six groups at a

significance level of 0.05. For 0 mm, the depth of cure of LA-T1 (1.34 ± 0.02 mm) was significantly lower than that of CAV (1.47 ± 0.03 mm) (p = 0.000). For BBH, the depth of cure was considered as 4.0 mm for convenience because the specimens were fully hardened to a depth of 8 mm. For 4 mm, the depth of cure of LA-T1 (1.34 ± 0.03 mm) was significantly lower than that of CAV (1.43 ± 0.04 mm) (p = 0.000). For BBH, the depth of cure value was also considered as 4.0 mm for convenience because the specimens were fully hardened to a depth of 8 mm. Although the depth of cure of both LA-T1 (p = 1.000) and BBH (p = 1.000) was not significantly different between irradiation distances 0 and 4 mm, the depth of cure of CAV cured at an irradiation distance of 4 mm was significantly lower than that of CAV cured at an irradiation distance of 0 mm (p = 0.007).

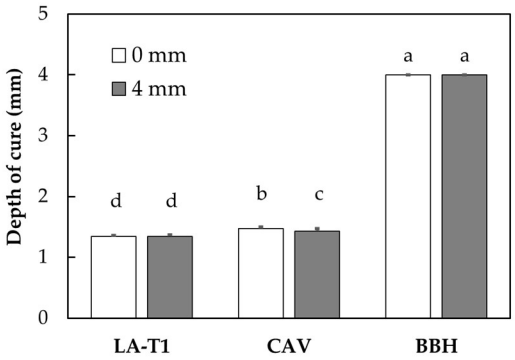

Figure 4. Depths of cure for light irradiated adjacent to the topside of the mold (0 mm) and 4 mm from the topside of the mold with the polyester strip interleaved using an LED light-curing device (mean ± SD, mm). Error bars represent standard deviations. The same letters indicate the absence of statistically significant difference while different letters indicate significant differences (p > 0.05).

3.2. Compressive Strength

The mean compressive strength and standard deviation (SD) of each group and the results of multiple comparisons are shown in Figure 5. Although the compressive strength of LA-T1 (203.0 ± 16.6 MPa) was significantly lower than that of BBH (266.4 ± 16.9 MPa) (p = 0.000), it was similar to that of CAV (196.5 ± 13.9 MPa) (p = 0.633).

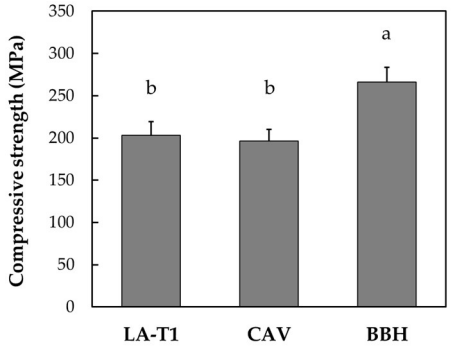

Figure 5. Compressive strengths of LA-T1, CAV, and BBH (mean ± SD, MPa). Error bars represent standard deviations. The same letters indicate the absence of a statistically significant difference while different letters indicate significant differences (p > 0.05).

3.3. μSBS

The mean μSBS and standard deviation (SD) of each group and the results of multiple comparisons are shown in Figure 6. LA-T1 (19.0 ± 3.9 MPa) did not show any significant difference from both CAV (16.8 ± 3.0 MPa, p = 0.354) and BBH (20.9 ± 3.2 MPa, p = 0.498). BBH had a significantly higher μSBS than CAV (p = 0.045).

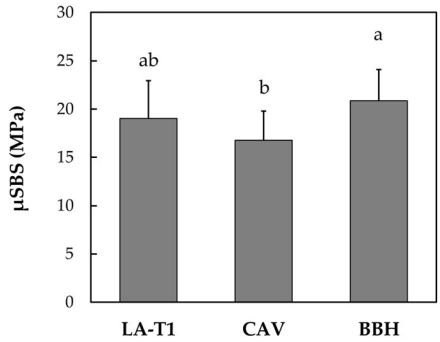

Figure 6. μSBS of LA-T1, CAV, and BBH (mean ± SD, MPa). Error bars represent the standard deviations. The same letters indicate the absence of a statistically significant difference while different letters indicate significant differences ($p > 0.05$).

4. Discussion

The purpose of this study was to compare the depth of cure, μSBS with resin-based dual-cured luting cement, and the compressive strength of LA-T1, a prototype MTA-based backing material, with those of existing products.

In this study, the depth of cure of LA-T1 was compared with those of CAV, an existing product, and BBH, a commercial bulk-fill resin-based composite base material. In general, the depth of cure of the resin-based composites used for crown restorations is approximately 2 mm [34,35]. All BBH specimens cured to a depth of 8 mm (depth of cure >4 mm). The depth of cure of the bulk-fill resin-based composite base materials is reported to be considerably larger than that of conventional resin-based composites [36–38]. Therefore, the very large depth of cure of BBH obtained in this study is consistent with previous reports [39,40]. In contrast, the depth of cure of LA-T1 and CAV was smaller than 2 mm. In particular, the depth of cure of LA-T1 was lower than that of CAV. Therefore, the first hypothesis was accepted. The depth of cure of the resin-based composites was reported to be greatly affected by the refractive index of light between the base monomer and filler. Compared with the base materials containing silica-based fillers, Cavios, containing α-TCP, and LA-T1, containing MTA, have a large refractive index between the base monomer and filler, which may cause light dispersion and reduce the depth of cure. UDMA-based monomers are known to have lower light transmittance than bisphenol A-ethoxylated dimethacrylate (Bis-EMA)-based monomers or a monomer mixture of Bis-EMA and UDMA [41]. These factors may contribute to the small depth of cure of LA-T1 and Cavios. Additionally, incomplete monomer conversion during polymerization leads to a large amount of residual monomer that gradually elutes from resin-based composites, which not only damages the dentin–pulp complex, but also reduces the strength of the restoration for occlusal forces [42,43].

We additionally examined the effects of two light irradiation distances, 0 mm and 4 mm, on the depth of cure. Generally, both the polymerization rate and depth of cure decreased as the distance between the curing light tip and surface of the resin-based composites increased [18]. While the depth of cure of CAV decreased as the light irradiation distance decreased to 4 mm than 0 mm, there were no significant differences in the depths of cure of both LA-T1 and BBH when the light irradiation distance changed. Therefore, the depth of cure of LA-T1 was unaffected by the light irradiation distance, which is considered as a good characteristic of a base material because the surface of the base material is always far from the curing light tip. While the in vivo study of Seino et al. [16] did not find any severe pulpal damage with CAV, the polymerization depth of both LA-T1 and CAV should be further improved.

The compressive strength of human dentin was in the range of 200–300 MPa [44]. The compressive strength of BBH was almost similar to that of human dentin, suggesting that BBH is useable as a substitute for dentin, which is lost during the removal of caries.

In contrast, the compressive strength of LA-T1 was almost similar to that of CAV, but significantly lower than that of BBH. In general, the compressive strengths of both the α-TCP based and MTA-based cements do not reach 100 MPa [45–48]. The compressive strengths of LA-T1 and CAV were both higher than those of these cements, suggesting that the inclusion of UDMA, a resin monomer, enhanced their compressive strengths. Nevertheless, the compressive strength of the resin-based composites in which the inorganic filler is replaced by bioactive glass [49] is equal to or higher than that of the glass-ionomer base material [50], suggesting that LA-T1 and Cavios are clinically acceptable base materials.

The μSBS of each base material bonded to a resin-based luting cement was 15 MPa or higher, and there was no significant difference among the three groups. Perkersoy et al. recently reported that the μSBS of bulk-fill resin-based composites bonded to human occlusal dentin via OptiBond XTR, a former version of OptiBond eXTRa, is 15–20 MPa [51]. Similarly, Akturk et al. reported that the μSBS of Estelite Σ Quick, a conventional resin-based composite bonded to human mid-coronal dentin via OptiBond XTR was 18.99 ± 3.53 MPa [52]. Therefore, the adhesion strength of the investigated base materials was almost equivalent to that of direct resin-based composite restorations.

The findings of this study suggest that the physico-mechanical properties of LA-T1, a base material containing MTA, are comparable to those of CAV, an existing product containing α-TCP, and thus LA-T1 is a potential candidate for clinical applications. However, both LA-T1 and CAV cannot adhere to the prepared cavity surface because they do not contain an adhesive component [53]. Therefore, for clinical applications, further improvements in these materials such as the development of specialized adhesive materials and higher light transmission are required. This article only reports on the physico-mechanical properties of the newly developed LA-T1, which was based on CAV. We are also currently conducting a histopathological study of LA-T1 against the exposed dental pulp of rats compared to CAV, suggesting that LA-T1 may perform better as a pulp-capping agent rather than as a base material. We expect that LA-T1 may perform better as a pulp-capping agent rather than as a base material, and we hope to publish our results in a subsequent report.

5. Conclusions

The conclusions of this work are as follows:

1. The depth of cure of the base material LA-T1 containing MTA was significantly but slightly lower than that of the existing product Cavios containing α-TCP, irrespective of the light irradiation distance, 0 mm (light irradiated adjacent to the topside of the mold) and 4 mm (from the topside of the mold).
2. The bulk-fill resin-based composite base material Bulk Base Hard had a significantly higher depth of cure than Cavios and LA-T1, irrespective of the light irradiation distance of 0 and 4 mm.
3. The compressive strength of LA-T1 was not significantly different from that of Cavios.
4. The microshear bond strength of LA-T1 bonded to the resin-based composite luting cement was similar to those of Cavios and Bulk Base Hard bonded to the same luting cement.

Author Contributions: Conceptualization: K.N., T.O., K.S., S.M., H.H. and A.K. (Atsushi Kameyama); Methodology: K.N. and A.K. (Atsushi Kameyama); Software: K.N.; Validation: A.K. (Akihiro Kuroiwa), S.M. and A.K. (Atsushi Kameyama); Formal analysis: A.K. (Akihiro Kuroiwa) and A.K. (Atsushi Kameyama); Investigation: K.N., N.H., T.O. and R.U.; Resources: A.K. (Atsushi Kameyama); Data curation: K.N. and A.K. (Atsushi Kameyama); Writing—original draft preparation: K.N. and A.K. (Atsushi Kameyama); Writing—review and editing: N.H., A.K. (Akihiro Kuroiwa), S.M. and H.H.; Visualization: K.N., T.O. and R.U.; Supervision: A.K. (Akihiro Kuroiwa), S.M. and H.H.; Project administration: S.M., H.H. and A.K. (Atsushi Kameyama); Funding acquisition: S.M., H.H. and A.K. (Atsushi Kameyama). All authors have read and agreed to the published version of the manuscript.

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