INTRODUCTION

Due to their superior mechanical properties, biocompatibility, and esthetics, yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) ceramics have been adopted for the framework or monolithic material in implant-supported prostheses. A number of studies reported that implant-supported Y-TZP ceramic prostheses result in satisfactory long-term survival and clinical outcomes comparable to those for implant-supported metal-ceramic prostheses. Implant-supported prostheses were luted with adhesive resin luting agent (RLA), glass ionomer cement (GIC), or zinc phosphate cement (ZPC). For MPZ and MLD specimens, fracture loads were significantly higher for RLA group than for GIC and ZPC groups. For PLZ specimens, fracture loads did not significantly differ in relation to luting agent. Fracture loads were significantly higher for MPZ specimens than for other test specimens, regardless of luting agent. Use of an adhesive resin luting agent is recommended for placement of premolar implant-supported monolithic Y-PSZ and lithium disilicate ceramic prostheses.

MATERIALS AND METHODS

The materials assessed in the current study are described in Table 1. To simulate clinical absence of a mandibular first premolar, 99 dental implants (ILA 20, Biomet 3i, Palm Beach Gardens, FL, USA) were placed perpendicularly in autopolymerizing acrylic resin (Technovit 4000, Heraeus Kulzer, Wehrheim, Germany), which has an elastic modulus similar to that of human bone. Titanium abutments (GingiHue Post WPP452G, Biomet 3i) were aligned to a thickness of 2.0 mm at the occlusal area by using diamond rotary cutting instruments (Bur No. 106RD, Shofu, Kyoto, Japan).
| Material (Trade name) | Lot No. | Components | Manufacturer |
|-----------------------|---------|------------|--------------|
| Implant body          |         |            |              |
| Implant Lab Analog ILA20 | 1207953 | Cu 60%, Zn 40% | Biomet 3i, Palm Beach Gardens, FL, USA |
| Abutment              |         |            |              |
| GingiHue Post WPP452G  | 1196424 | Ti 99% (grade 4) | Biomet 3i |
| Abutment screw        |         |            |              |
| Hexed UniScrew UNIHT  | 1203897 | Ti 99% (grade 4) | Biomet 3i |
| Y-PSZ material        |         |            |              |
| Katana Zirconia STML A2 | —      | ZrO$_2$ 88–93%, Y$_2$O$_3$ 7–10%, others | Kuraray Noritake Dental, Tokyo, Japan |
| Y-TZP framework material |     |            |              |
| Katana Zirconia HT    | —      | ZrO$_2$ 94.4%, Y$_2$O$_3$ 5.4%, others | Kuraray Noritake Dental |
| Feldspathic porcelain |         |            |              |
| Cerabien ZR (SBA2, A2B, E2) | SBA2: 026626, A2B: DRDIA E2: 058284 | SiO$_2$, Al$_2$O$_3$, Na$_2$O, K$_2$O, others | Kuraray Noritake Dental |
| Lithium disilicate material |     |            |              |
| e.max Press LT A2     | U54251  | SiO$_2$ 57–80%, LiO$_2$ 11–19%, K$_2$O 0–13%, P$_2$O$_5$ 0–11%, ZrO$_2$ 0–8%, ZnO 0–8%, others 0–10% | Ivoclar Vivadent, Schaan, Liechtenstein |
| Luting agent          |         |            |              |
| Panavista V5          | 8N0061  | A Paste: Bis-GMA, TEGDMA, hydrophilic aromatic dimethacrylate, silanated barium glass filler, fluoroaluminosilicate glass filler, colloidal silica, accelerator, initiator | Kuraray Noritake Dental |
| Ketac Cem Easymix     | 1708071 | Glass powder, polycarboxylic acid, others | 3M ESPE, St. Paul, MN, USA |
| Elite Cement 100      | 031752  | ZnO, MgO, Zn(PO$_4$)$_2$, H$_3$PO$_4$, others | GC, Tokyo, Japan |
| Bonding agent         |         |            |              |
| Clearfil Photo Bond    | 3P0031  | Catalyst: MDP, HEMA, Bis-GMA | Kuraray Noritake Dental |
| Clearfil Porcelain Bond Activator | BA0034 | 3-TMSPMA | Kuraray Noritake Dental |

Y-PSZ: yttria-partially stabilized zirconia, Y-TZP: yttria-stabilized tetragonal zirconia polycrystal, Bis-GMA: bisphenol A-diglycidyl methacrylate, TEGDMA: triethyleneglycol dimethacrylate, CQ: dl-camphorquinone, MDP: 10-methacryloyloxydecal dihydrogen phosphate, HEMA: 2-hydroxyethyl methacrylate, and 3-TMSPMA: 3-methacryloyloxypropyl trimethoxysilane.
and water coolant (Presto Aqua, Nakanishi, Kanuma, Japan). The abutments were tightened with titanium screws (Titanium Hexed UniScrew UNIHT, Biomet 3i) at 20 N·cm torque with a torque control system (Torque Driver HTD-C, Biomet 3i).

The implant-abutment complexes were divided into three test groups (n=33) for implant-supported prostheses, namely, (1) monolithic yttria-partially stabilized zirconia (Y-PSZ) single restorations (MPZ specimens), (2) porcelain layered on yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) single restorations (PLZ specimens), and (3) monolithic lithium disilicate ceramic single restorations (MLD specimens). The three test groups were then further divided by luting agent type into three subgroups: an adhesive resin luting agent (RLA; Panavia V5, Kuraray Noritake Dental, Tokyo, Japan), a glass ionomer cement (GIC; Ketac Cem Easymix, 3M ESPE, St. Paul, MN, USA), and a zinc phosphate cement (ZPC; Elite Cement 100, GC, Tokyo, Japan).

**MPZ specimens**

The wax pattern was developed on the titanium abutment by using dental inlay wax (Dental Inlay Casting Wax, GC, Fig. 1). The abutment and wax pattern were doubly scanned with a measurement apparatus (KATANA Dental Scanner D2000, Kuraray Noritake Dental) and modeled with computer-aided design (CAD) software. The monolithic single crown restorations were designed for CAD software. The space of luting agents was set at 40 μm, except for the finish line. Data for the crown design were transferred to the milling machine (DWX-51D, Kuraray Noritake Dental) for fabrication of the monolithic Y-PSZ (KATANA Zirconia STML A2, Kuraray Noritake Dental) crowns. The crowns were sintered to full density in a furnace (KATANA F-1, Kuraray Noritake Dental) at 1,550°C for 120 min and then finished and polished.

**PLZ specimens**

Eleven Y-TZP (KATANA Zirconia HT, Kuraray Noritake Dental) frameworks (thickness: 0.5 mm, Fig. 2) were fabricated with the same procedure used for MPZ specimens. The frameworks were layered with feldspathic porcelain (Cerabien ZR, Kuraray Noritake Dental) by using a conventional technique (Table 2, Fig. 2).

**MLD specimens**

A wax pattern was made with the same method used for the MPZ specimens (Fig. 1). Wax patterns were invested with a commercial phosphate-bonded investment material (IPS Press VEST Speed, Ivoclar Vivadent, Schaan, Liechtenstein). The investment was heated in a furnace (Auto Furnace QM-1, GC) at 700°C for 60 min. IPS e.max Press ingots (e.max Press LT A2, Ivoclar Vivadent) were heat-pressed into the mold cavity with a Cerafusion Press (MORITA, Tokyo, Japan), in accordance with the manufacturer’s instructions (Table 2).

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**Table 2** Firing procedures for feldspathic porcelain and lithium disilicate ceramics, as recommended by the manufacturers

| Material       | Pre-heating temperature (°C) | Pre-heating time (min) | Heating rate (°C/min) | Firing temperature (°C) | Holding time (min) | Cooling time (min) |
|----------------|-----------------------------|------------------------|-----------------------|-------------------------|-------------------|-------------------|
| Cerabien ZR SBA2 | 600                         | 5                      | 45                    | 930                     | 1                 | 4                 |
| Cerabien ZR A2B  | 600                         | 8                      | 45                    | 935                     | 1                 | 4                 |
| Cerabien ZR E2   | 600                         | 5                      | 50                    | 930                     | 0.5               | 4                 |
| e.max Press LT A2 | 700                         | 0                      | 60                    | 930                     | 25                | 0                 |
Table 3 Descriptive statistics for fracture load values (kN)

| Specimen | Luting agent | Median | IQR | Category* |
|----------|--------------|--------|-----|-----------|
|          | RLA          |        |     |           |
| MPZ      | 3.89         | 0.77   | (4.32: 3.55) | a, A |
| PLZ      | 2.32         | 0.70   | (2.72: 2.02) | c, B |
| MLD      | 2.49         | 0.39   | (2.58: 2.36) | d, B |
|          | GIC          | 3.42   | 0.43 | (3.75: 3.32) | b, C |
|          |              | 2.25   | 0.46 | (2.48: 2.02) | c, D |
|          |              | 2.06   | 0.49 | (2.33: 1.83) | e, D |
|          | ZPC          | 3.16   | 0.50 | (3.59: 3.09) | b, E |
|          |              | 2.32   | 0.61 | (2.72: 2.11) | c, F |
|          |              | 2.07   | 0.57 | (2.39: 1.82) | e, F |

MPZ specimen: monolithic yttria-partially stabilized zirconia (Y-PSZ) single restorations, PLZ specimen: porcelain layered on yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) single restorations, MLD specimen: monolithic lithium disilicate ceramic single restorations, RLA group: adhesive resin luting agent, GIC group: glass ionomer cement, and ZPC group: zinc phosphate cement.

* Identical lowercase letters indicate that the values for the luting agents are not significantly different (Mann-Whitney U-test with Bonferroni correction, p<0.05). Identical uppercase letters indicate that the values for the implant-supported prostheses are not significantly different (Mann-Whitney U-test with Bonferroni correction, p<0.05).

1 IQR: interquartile range.

Statistical analysis

Statistical software (IBM SPSS Statistics, version 24.0, IBM, Armonk, NY, USA) was used to analyze equality of variance with the Levene test. When equality of variance was not detected, a nonparametric test was used. The Kruskal-Wallis test was utilized when using median values to evaluate differences in the three luting agents. On the basis of the results, the Mann-Whitney U-test with Bonferroni correction for multiple comparisons was additionally performed to assess differences among luting agents. Differences among the three implant-supported prostheses were analyzed by using the Mann-Whitney U-test with Bonferroni correction. Significance in all tests was defined as p<0.05.

Observation of fracture pattern

The failure patterns of the test specimens were classified as (a) fracture of the layering porcelain, (b) total fracture of the zirconia framework, together with the layering porcelains, and (c) complete fracture. After fracture load testing, the inner surfaces of specimens were observed with an optical microscope (Stemi DV4, Carl Zeiss, Oberkochen, Germany). For the representative specimens, the ratios of remaining luting agent were calculated as follows, ratio of remaining luting agent (%)=remaining luting agent area (mm²)/fracture area (mm²)×100, using surface analysis software (LM eye, Lasertec, Tokyo, Japan). Additionally, the elemental and molecular composition of the fractured surface was analyzed by using energy-dispersive X-ray spectrometry (EDX, Rany EDX-900, Shimadzu, Kyoto, Japan).

RESULTS

The descriptive statistics of fracture load values are presented in Table 3. For MPZ and MLD specimens,
median fracture load values were significantly higher for the RLA group than for the GIC and ZPC groups. No significant difference was detected between the GIC and ZPC groups for MPZ and MLD specimens ($p=0.237$ and $p=0.974$, respectively). For PLZ specimens, the median fracture load values of the luting agents did not significantly differ ($p=0.279$). In the comparison of implant-supported prostheses, fracture loads for the MPZ specimens were significantly higher than those for the other test specimens, regardless of the luting agent used.

In the PLZ group, one RLA group specimen and two GIC group specimens exhibited complete fracture; all other specimens exhibited fracture of the layering porcelain (Fig. 3a). The fracture pattern was complete fracture in the MPZ and MLD specimens, as shown in Fig. 3b.

Figures 4 and 5 show optical microscopic images of the red outline shows the remnants of luting agents.

Fig. 4 Representative fracture surface of an MPZ specimen and ratio of remaining luting agent; (a) RLA, (b) GIC, and (c) ZPC groups.

The red outline shows the remnants of luting agents.

Fig. 5 Representative fracture surface of an MLD specimen and ratio of remaining luting agent; (a) RLA, (b) GIC, and (c) ZPC groups.

The red outline shows the remnants of luting agents.

Fig. 6 Results of EDX spectra and element analysis of test luting agents; (a) RLA, (b) GIC, and (c) ZPC groups.
the inner surfaces and ratios of remaining luting agent for MPZ and MLD specimens, respectively. RLA group specimens showed more remnants of luting agent on the inner surface (Figs. 4a and 5a; ratio of remaining luting agent, 77.0 and 38.7%, respectively), as compared with GIC (Figs. 4b and 5b; ratio of remaining luting agent, 9.4 and 4.9%, respectively) and ZPC group specimens (Figs. 4c and 5c; ratio of remaining luting agent, 4.5 and 4.6%, respectively).

Figure 6 shows EDX spectra and element composition of the investigated luting agents. Characteristic indicators of the luting agents were detected: Si, Al, and Ba (75.5, 10.4, and 9.9 atomic percentage, at%, respectively) for the RLA group (Fig. 6a); Si, Al, and Ca (36.0, 31.9, and 22.4 at%, respectively) for the GIC group (Fig. 6b); and Zn and P (49.3 and 36.8 at%, respectively) for the ZPC group (Fig. 6c). Representative EDX spectra and element composition of the inner surfaces of MPZ and MLD group specimens are shown in Figs. 7 and 8, respectively. The inner surfaces of MPZ specimens were composed of Zr, Y, and characteristic elements of each luting agent (Figs. 7a, b, and c). For the MLD group, the peaks of K, Zn, P, and the characteristic elements of each luting agent were detected (Figs. 8a, b, and c).
DISCUSSION

The current study investigated the effect of luting agent type on fracture loads of premolar implant-supported ceramic prostheses, namely, bi-layered Y-TZP, monolithic Y-PSZ, and lithium disilicate ceramic prostheses. The results are consistent with rejection of the null hypothesis, i.e., that there would be no significant difference in fracture load in relation to luting agent type or ceramic material. The RLA group exhibited the highest fracture loads of the tested groups, for MPZ and MLD specimens. For premolar implant-supported ceramic prostheses, the fracture loads of MPZ specimens were significantly higher than those of PLZ and MLD specimens, for each luting agent tested.

The present results indicate that use of an adhesive resin luting agent (RLA group) increased the fracture loads of implant-supported monolithic Y-PSZ (MPZ specimens) and lithium disilicate ceramic (MLD specimens) prostheses in the premolar area, and the findings of optical microscopic observation and EDX analysis study support these results. For the MPZ and MLD specimens (Figs. 4 and 5, respectively), the ratios of remaining luting agent of the RLA group were greater compared with those of the GIC and ZPC groups. In addition, the lower content Zr and Y in the RLA group for the MPZ specimens (Fig. 7) implies much remnants of luting agents on the inner surface of the specimens tested. These findings are likely attributable to the difference in bond strength between Y-PSZ/lithium disilicate ceramics and the luting agents tested. Adhesive luting agents yield higher bond strengths to ceramic materials than do conventional dental cements such as glass ionomer cement and zinc phosphate cement.\(^{20,21}\) The present results are consistent with those of previous studies\(^{10,22-24}\) that reported that Y-PSZ and lithium disilicate ceramic prostheses should be seated by using adhesive luting agents. However, other studies found no significant difference in the durability of lithium disilicate ceramic prostheses\(^{25,26}\). This disparity might be attributable to differences in the assessed materials and testing methods, including the size and form of the specimens tested.

For PLZ specimens, the fracture load values of the three different luting agents did not significantly differ, which suggests that conventional dental cements, as well as resin luting agents, can be used for placement of premolar implant-supported bi-layered Y-TZP ceramic prostheses. These findings are consistent with those of previous studies\(^{25,27}\) and are attributable to the superior mechanical properties of Y-TZP frameworks, which can withstand masticatory forces in the premolar area. In other words, because the flexural strength and fracture toughness of Y-PSZ and lithium disilicate ceramics are inferior to those of Y-TZP ceramics, adhesive resin bonding is recommended for placement of posterior implant-supported monolithic Y-PSZ and lithium disilicate ceramic prostheses.

In all luting agent groups, fracture load values were significantly higher for MPZ specimens than for MLD specimens. The mechanical properties of restorative materials are associated with the fracture resistance of restorations\(^{29}\). The present findings are supported by those of other studies, which reported that flexural strength and Weibull characteristic strength were higher for Y-PSZ ceramics than for lithium disilicate ceramics\(^{6,29}\). The current study is the first to evaluate fracture resistance of monolithic ceramic prostheses, especially Y-PSZ ceramics, luted on titanium implant abutments. Although this study used monolithic Y-PSZ ceramics, it did not evaluate Y-TZP alone, as it is not clinically indicated for monolithic prostheses.

In this in vitro study, the median fracture load values of all test groups were greater than 2.0 kN. The maximum masticatory force in the molar region was reported to be as high as 0.9 kN\(^{20}\). Thus, the present results clearly show that premolar implant-supported ceramic prostheses, including the bi-layered Y-TZP, monolithic Y-PSZ, and lithium disilicate ceramic prostheses, have stable fracture resistance in clinical use. A limitation of this study was that the premolar implant-supported ceramic prostheses were not subjected to artificial aging. Future studies should use cyclic thermomechanical loading to investigate the durability of implant-supported ceramic prostheses.

Within the limitations of this in vitro study, the present findings are consistent with the following conclusions. Use of an adhesive resin luting agent is recommended for placement of premolar implant-supported monolithic Y-PSZ and lithium disilicate ceramic prostheses. Fracture load values are higher for premolar implant-supported monolithic Y-PSZ ceramic prostheses than for lithium disilicate ceramic prostheses. In addition, all the investigated implant-supported ceramic prostheses are suitable for clinical use.

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CONFLICTS OF INTEREST

The authors certify that they have no conflicts of interest.

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