INTRODUCTION

Currently, requirements for metal-free restorations have increased because of patient demands for esthetics in dentistry. In the past, porcelain-fused-to-metal crowns were the most common form of esthetic prosthesis. Computer-aided design/computer-aided manufacturing (CAD/CAM) technologies were introduced in 1971 (1), and with the advent of this technology, various fabrication techniques have been developed to enhance the consistency and predictability of restorations in terms of production cost, time requirements, and quality (2,3). In Japan, indirect resin composite premolar crowns using CAD/CAM technology were approved for coverage by the Japanese social insurance system in 2014 (4). For luting indirect resin composite crowns, adhesive resin cement is used to prevent secondary caries and debonding due to its high bond strength to abutment and low solubility in saliva. However, in clinics, a high initial debonding rate of the indirect resin composite crown has been reported even using adhesive resin cements (5). Two interfaces, an abutment-cement interface and a cement-crown interface, exist when the indirect resin crown is luted. An in vitro study revealed that the cement-resin block for CAD/CAM offers sufficient bond strength (6). However, diverging from the in vitro situation, adhesion-inhibiting factors are present in clinical settings. For example, the long-term durability of bonding between the resin composite block and luting cement is markedly decreased by saliva contamination at the cement-crown interface (7). Other adhesion inhibitors are present at the abutment-cement interface, such as temporary cement remaining on the abutment.

At present, 1-day treatment remains uncommon, so provisional restoration with temporary cement is needed during the fabrication of indirect restorations, to avoid infection, tooth sensitivity, and tooth movement (8). The presence of this residual temporary cement on prepared abutment teeth might exert negative effects on the performance of self-adhesive cements by interfering with the penetration of adhesive monomers into the tooth substrate (8,9). Such residual temporary cement must be sufficiently removed from the abutment surface prior to definitive cementation. Previous studies have examined the effects of removing remnant cement. Using an air scaler as a mechanical removal method reportedly results in the same adhesion performance as that achieved without any cleaning (10), and using a rotational brush with running water was identified as the best method to achieve consistent bond strength between luting cement and primary dentin subjected to temporary cementation (10). On the other hand, cleaning using a brush did not achieve sufficient cleaning for some types of resin cement (11). The data to date have been contradictory, and no standardized removal methods have been defined for clinical practice. More
effective methods to remove temporary cement are thus anticipated.

The present study focused on the use of 10-methacryloyloxydecyl dihydrogen phosphate (MDP) as a new method for removing temporary cement. Basically, a functional monomer can ionically interact through a carboxylic/phosphoric/phosphonic group with the Ca in hydroxyapatite (HAp) and can copolymerize with other resin monomers at the methacrylate groups\textsuperscript{12-15}. Interestingly, since MDP also has a structure with both hydrophilic and hydrophobic groups\textsuperscript{14}, temporary cement may be effectively removed by the surfactant action of MDP. Surfactant action involves the action of the hydrophobic groups in the surfactant surrounding and separating a material by attaching to the surface of a solid, penetrating and attaching between interfaces of different polarities. Not only is MDP expected to show the ability to remove temporary cement, but also any MDP remaining at the bonding interface after removal of temporary cement is not problematic. No previous studies have examined MDP as a temporary cement remover.

The present study aimed to elucidate how remnant temporary cement affects the effectiveness of dentin bonding and to identify a decontamination method that can restore the original bond strength as determined by micro-tensile bond strength (\(\mu\)TBS) and observation of interface morphology under transmission electron microscopy (TEM). Before the \(\mu\)TBS test and TEM observation, surface morphology was examined under microscopy (TEM). Before the \(\mu\)TBS test and TEM of interface morphology under transmission electron microscopy and energy dispersive X-ray spectroscopy (EDS), and contact angle was measured. The null hypotheses were that: 1) no difference in the state of residual temporary cement on the dentin surface would be seen after application of the various cleaning methods; and 2) the ability of new decontaminant containing MDP to remove temporary cement would be no different from those of other cleaning methods.

MATERIALS AND METHODS

Specimen preparation

Following approval by the ethics committee at Osaka University (protocol number H26-E6), a total of 105 extracted human third molars were collected and stored in distilled water at 4°C (details for numbers of teeth in each group are provided in Fig. 1). Flat dentin surfaces were ground using a model trimmer perpendicular to the long axis of the tooth under water lubrication, then wet-polished using 400- and 600-grit SiC paper for 30 s to create a standardized smear layer. A control group (Co) was created without application of temporary materials. For the remaining samples, a 10×10×5-mm resin plate made using quick cure resin (Uni-fast III; GC, Tokyo, Japan) was applied to dentin with temporary cement (HY-BOND temporary cement hard; Shofu, Kyoto, Japan) under a 1-kg load and was left for 5 min (Table 1). All specimens were then stored in 37°C distilled water for 1 week.

The resin plate was removed and temporary cement was cleaned out using an excavator until no cement was visible with the naked eye for all specimens. The Sc group of specimens was cleaned only with an air-scaler for 20 s, and the Br group was cleaned only with a rotating brush under running water for 20 s. The NC group was cleaned with the air-scaler for 20 s, then treated with phosphoric acid (K-etchant gel; Kuraray Noritake Dental, Tokyo, Japan) for 20 s and water rinsed with a 3-way syringe for 20 s, and finally treated with sodium hypochlorite (AD-gel; Kuraray Noritake Dental) for 60 s and water rinse for 20 s. As the new method using MDP, the MC group of specimens were cleaned with the air-scaler for 20 s, then the prototype cleaner with MDP (Kuraray Noritake Dental) was applied, left for 10 s, and rinsed with water for 20 s. The MC+AG group were treated with the same way as the MC group before rinsing off the cleaner, but agitated with a micro-brush for 10 s, and followed by water rinse for 20 s. As a control for the MC+AG group, the AG group of specimens was rubbed with distilled water instead of cleaner and then rinsed with a 3-way syringe for 20 s.

SEM surface observation and EDS surface analysis

Three teeth from each of the 7 groups of Co, Sc, Br, NC, MC, MC+AG, and AG were fixed to the sample table with carbon tape, and the surface underwent osmium deposition using an osmium plasma coater (OPC60A; Filgen, Nagoya, Japan). FE-SEM (JSM-6335F; JEOL, Tokyo, Japan) and EDS (JED-2300; JEOL) was performed at an acceleration voltage of 20.0 kV.

Contact angle measurement

Nine teeth each from the 5 groups of Co, Sc, Br, NC, and MC+AG were processed as described in the Specimen preparation section. In addition, the MDP group were prepared by applying 5% MDP ethanol solution to a dentin sample without contamination by temporary cement. One microliter of purified water was dropped onto the cleaned surface after each cleaning method, and the contact angle was measured (\(n=9\)) with a fully automatic small contact angle meter (P200A; Meïwa Forsys, Tokyo, Japan). Data were analyzed using one-way analysis of variance and the Tukey test, with a significance level of 0.05. All statistical analyses were performed using EZR software (www.jichi.ac.jp/saitama-sct/SaitamaHP.files/statmed.html).

\(\mu\)TBS test

A dentin smooth surface was processed for 30 additional teeth using the method described above in the Specimen preparation section, and randomly divided into 5 groups of Co, Sc, Br, NC, and MC+AG. A CAD/CAM indirect resin block (KATANA Avencia A3 LT; Kuraray Noritake Dental) was cut into a 15×15×2-mm block using a diamond cutter (MC201N; Marto, Tokyo, Japan). The surface of the resin block was subjected to alumina blasting at 0.2 MPa for 20 s using an air brush (AD ABRADER; Morita, Tokyo, Japan). For alumina blasting, 50-\(\mu\)m alumina particles (AEX-6; Paasche Airbrush Company, Chicago,
Table 1 Materials used

| Materials                      | Product name                          | Manufacturing company                  | LOT       | Composition                                                                 |
|-------------------------------|---------------------------------------|----------------------------------------|-----------|-----------------------------------------------------------------------------|
| Temporary cement              | HY-BOND TEMPORARY CEMENT (HARD)       | Shohu                                  | 071657    | Powder Zinc oxide, Silica, Magnesium oxide, Tannin-fluoride compound, Colorant etc. |
|                               |                                       |                                        |           | Liquid Acrylic acid-tricarboxylic acid copolymer, Sodium salt, Purified water |
| Quick cure resin for temporary crowns | Uni-fast III                          | GC                                     | 1706261   | Powder Methacrylate polymer Liquid Methyl methacrylate                      |
| Dentin treatment agent        | K-etchant gel                         | Kuraray Noritake Dental                | 5R0079    | Purified water, Phosphoric acid, Colorant, Thickener                        |
|                               | ADgel                                 | Kuraray Noritake Dental                | 5J0187    | Base (sodium hypochlorite), Thickener (alumina micro filler)                |
| Decontamination agent         | Prototype cleaner with MDP            | Kuraray Noritake Dental                | —         | Water, MDP, Sodium hydrogen phosphate, Thickener (silica), Colorant, Moisturizer (macrogol) |
| CAD/CAM resin block           | KATANA AVENCIA Block                  | Kuraray Noritake Dental                | 00526     | Light weight silicic anhydride / Aluminum oxide mixed filler, Methacrylic acid monomer cured product (UDMA, other methacrylic monomer), Colorant etc. |
| Alumina blaster               | AD ABRADER                            | DELDENT                                | —         | Alumina oxide (50 µm)                                                      |
| CAD/CAM resin block treatment agent | Clearfil ceramic primer plus          | Kuraray Noritake Dental                | CL0022    | 3-trimethoxysilylpropyl methacrylate, Monomer (MDP), Ethanol                |
|                               |                                       |                                        |           | A paste Monomers (MDP, Bis-GMA, TEGDMA, HEMA, other methacrylic monomers), Fillers (surface-treated barium glass, surface-treated silica-based microfills), Photopolymerization catalysts, Chemical polymerization catalysts, Colorants etc. |
| Resin cement                  | SA cement plus automix                | Kuraray Noritake Dental                | 6L0099    | B paste Methacrylic acid monomer, Filler (Surface treatment barium glass, Surface treatment sodium fluoride), Polymerization accelerator, Colorant etc. |
| 5% MDP ethanol solution       | (Experimental)                        | —                                      | —         | Ethanol, MDP                                                                |

IL, USA) were used. The adhesive surface of the resin block was treated with silane (Clearfil Ceramic Primer Plus; Kuraray Noritake Dental) for 10 s. Self-adhesive resin cement (SA Cement Plus Automix; Kuraray Noritake Dental) was applied to the dentin surface and the resin block was luted. Excess cement was wiped off with a cotton ball and pressed under a 1-kg weight for 5 min. The cement was light-cured from the block side for 10 s at a maximum light intensity of 2,200 mW/cm² (Mini LED III; Satelec, Mérignac, France). Specimens were stored in distilled water at 37°C for 24 h. Each bonded specimen was sectioned parallel to the long axis of the tooth into beams with a bonded surface area of 1.0 mm² using the MC201N diamond cutter. The prepared samples were further divided into 3 subgroups in each group and immersed in water for 24 h, 1 month, or 6 months. To measure µTBS, beams were glued to a jig with Model Repair II Blue (DENTSPLY-Sankin, Tochigi, Japan) and tested using a universal testing machine (EZ-test short; Shimadzu, Kyoto, Japan) at a crosshead speed of 1.0 mm/min until fracture. Mean cross-sectional area of each specimen was measured using a CD15 micrometer (Mitutoyo, Kanagawa, Japan) to calculate the cross-sectional area, and tensile strength at the time of measurement was converted to mega-Pascals. Pre-testing failures (ptfs) during specimen preparation for each group of specimens was included in the test results as 0 MPa. Data were analyzed using...
Kruskal-Wallis methods and the Mann-Whitney U test with a significance level of 0.05. Statistical analysis was performed using EZR software (www.jichi.ac.jp/saitama-sct/SaitamaHP.files/statmed.html).

Failure mode analysis
The fracture surface after µTBS testing was observed with a 30× optical microscope (SZ61; OLYMPUS, Tokyo, Japan) to confirm fracture mode, which was classified into four types: resin block-resin cement interface failure; resin cement-dentin interface failure; resin block-resin cement-dentin mixed failure; and cohesive failure in dentin. Combined failure between the resin block, resin cement, and dentin was defined as including ≥30% of each interface failure mode. Typical samples were also fixed on an aluminum sample stand with carbon tape. Gold was then applied to the deposition surface (JFC-1600 AUTO FINE COATER; JEOL) and observed under SEM (JSM-6510LV; JEOL).

TEM observation
Four samples from the Co, Sc, Br, NC, and MC+AG groups were randomly extracted from each 1×1-mm beam prepared for the µTBS test. Sample pieces after dehydration in rising ethanol series were embedded in epoxy resin (Quetol812; Nissin EM, Tokyo, Japan) and subjected to heat polymerization at 60°C for 72 h. After curing, an ultra-thin section of approximately 70 nm was prepared at the site including the adhesive interface using a diamond knife (Nanotome Chic; Sakai Electron Microscope Application Laboratory, Saitama, Japan) and an ultramicrotome (Ultrome V; LKB, Bromma, Sweden). Sections were observed under TEM (H-800; Hitachi, Tokyo, Japan) at an applied voltage of 200 kV.

RESULTS

SEM observation and EDS analysis
Surface observation under SEM revealed a smear layer on the surface in the Co, Sc, Br, and MC+AG groups (Figs. 2b, e1, g). In the Sc group, a lump of temporary cement with a diameter of about 10 µm was found remaining over the entire surface (Fig. 2b). In the NC group, the smear layer was completely removed and dentinal tubules were open (Fig. 2d).

Surface analysis by EDS detected Zn in the Sc group at the same site as the remaining mass of the temporary cement confirmed on SEM (Table 2, Figs. 3b1, 2, 4b). In the Br group, no residual temporary cement was evident under SEM, but Zn was clearly present on the surface (Table 2, Figs. 3c2, 4c). Zn was not detected in the NC and MC+AG groups. In the NC group, Cl was also confirmed.

Contact angle measurement
Contact angles were significantly larger in the Sc, Br, MC+AG, and MDP groups than in the Co group (Fig. 5; Sc/Co, p<0.001; Br/Co, p=0.001; MC+AG/Co, p=0.003; MDP/Co, p=0.03). No significant difference was apparent between the NC and Co groups (p=0.39).

µTBS and failure mode
The Kruskal-Wallis test revealed that µTBS was influenced by cleaning method and storage period (Table 3, Fig. 6, p<0.001). Multiple comparison testing by the Mann-Whitney U test showed significantly higher bond strength in the Co group than in the Sc group or Br group (p<0.001 each). Furthermore, the Sc group showed significantly lower bond strength than the Br group (p=0.01). On the other hand, the NC group (p=0.99) or MC+AG group (p=0.38) did not show...
Fig. 2 Photomicrographs from SEM of dentin surfaces after cleaning. Photomicrographs from SEM of dentin surfaces from Co (a), Sc (b), Br (c), NC (d), MC (e1,2), MC+AG (f), and AG (g). Approximately 10 µm of temporary material (arrows) remains in Sc (b), MC (e1), and AG (g). In Br (c), NC (d), and MC+AG (f), no temporary cement remains. In NC, the smear layer has been removed and dentinal tubule openings are observed (d).

Fig. 3 Mapping of zinc by EDS. Sites of zinc detection are shown in green. Zinc is detectable in Sc (b1,2), Br (c1,2), MC (e1,2), and AG (g1,2). Regarding Br, zinc is observed in a fine granular form as a whole (c1). In MC, zinc is detected in the remaining part of temporary material observed from SEM (e2).

Table 2 Percentage of each chemical element from analyses of the different experimental groups

|       | Co   | Sc   | Br   | NC   | MC   | MC+AG | AG   |
|-------|------|------|------|------|------|-------|------|
| C     | 23.6 | 25.3 | 24.5 | 32.6 | 29.0 | 26.8  | 22.9 |
| O     | 45.5 | 42.8 | 43.6 | 38.7 | 43.1 | 43.2  | 42.8 |
| P     | 11.3 | 10.4 | 10.7 | 9.9  | 10.2 | 11.4  | 10.7 |
| Ca    | 18.9 | 16.9 | 17.5 | 17.9 | 16.5 | 18.1  | 17.3 |
| Mg    | 0.7  | 0.6  | 0.6  | 0.4  | 0.5  | 0.6   | 0.4  |
| Cl    | 0    | 0    | 0    | 0.4  | 0    | 0     | 0    |
| Zn    | 0    | 4.0  | 3.2  | 0    | 0.7  | 0     | 5.9  |

unit: %, C: carbon; O: oxygen; P: phosphorus; Ca: calcium; Mg: magnesium; Cl: chlorine; Zn: zinc.
Fig. 4 Surface analysis using energy dispersive X-ray spectroscope EDS analysis. Sc (b), Br (c), MC (e) and AG (g) reveal distinct peaks, representing the element zinc (arrow). Chloride is recognized only in NC (d, arrow head). Carbon, oxygen, zinc, phosphorus, calcium and magnesium are observed in all groups.

Table 3 Microtensile bond strength (µTBS) and failure mode analysis

| Groups  | Co     | Sc     | Br     | NC     | MC+AG  |
|---------|--------|--------|--------|--------|--------|
| 24 h    | 12.6 (5.4) [0/25/5/0] | 7.0 (7.1) [0/22/8/0] | 12.0 (8.3) [0/23/7/0] | 12.8 (9.7) [0/24/6/0] | 13.9 (4.8) [0/21/9/0] |
| 1 M     | 12.4 (6.7) [0/23/7/0] | 4.5 (4.4) [0/24/6/0] | 5.7 (4.4) [0/23/7/0] | 10.8 (9.1) [0/26/4/0] | 13.2 (4.5) [0/24/6/0] |
| 6 M     | 8.6 (7.1) [0/26/4/0] | 2.5 (3.0) [0/25/5/0] | 4.5 (5.8) [0/26/4/0] | 9.5 (7.2) [1/22/7/0] | 11.1 (3.8) [0/21/9/0] |
| pfts    | 0/90   | 24/90  | 6/90   | 0/90   | 0/90   |

Upper: µTBS (MPa), n=30, Lower: Failure mode, [Cohesive failure in dentin/Interfacial failure between cement and dentin/Mixed failure between block, cement and dentin/Interfacial failure between cement and block], pfts=pre testing failures

Table 3

| Groups  | Co     | Sc     | Br     | NC     | MC+AG  |
|---------|--------|--------|--------|--------|--------|
| 24 h    | 12.6 (5.4) [0/25/5/0] | 7.0 (7.1) [0/22/8/0] | 12.0 (8.3) [0/23/7/0] | 12.8 (9.7) [0/24/6/0] | 13.9 (4.8) [0/21/9/0] |
| 1 M     | 12.4 (6.7) [0/23/7/0] | 4.5 (4.4) [0/24/6/0] | 5.7 (4.4) [0/23/7/0] | 10.8 (9.1) [0/26/4/0] | 13.2 (4.5) [0/24/6/0] |
| 6 M     | 8.6 (7.1) [0/26/4/0] | 2.5 (3.0) [0/25/5/0] | 4.5 (5.8) [0/26/4/0] | 9.5 (7.2) [1/22/7/0] | 11.1 (3.8) [0/21/9/0] |
| pfts    | 0/90   | 24/90  | 6/90   | 0/90   | 0/90   |

Upper: µTBS (MPa), n=30, Lower: Failure mode, [Cohesive failure in dentin/Interfacial failure between cement and dentin/Mixed failure between block, cement and dentin/Interfacial failure between cement and block], pfts=pre testing failures

Fig. 5 Contact angle data.

Fig. 6 Box plot of data from µTBS. Features of each box plot represent, from the top: maximum, 75th percentile, median, 25th percentile, and minimum.

In all groups, adhesive failure between cement and dentin was the most common type of failure (Table 3). Mixed failure between block, cement, and dentin was the second most common, and one instance of cohesive failure in dentin was observed. No adhesive failure between cement and block was observed. In all groups, significantly different bond strength from the Co group. Bond strength decreased over time (0M vs 1M, p=0.01; 0M vs 6M, p<0.001; 1M/6M, p=0.008). Bond strength in the MC+AG group did not differ significantly from that of the Co group, but tended to be higher than that in the NC group.
no significant differences in fracture mode due to differences in cleaning methods or storage periods were identified.

**TEM observation**

TEM revealed the presence of a smear layer and peritubular dentin in the Co and MC+AG groups, with resin cement showing no infiltration into the dentinal tubules, but instead forming a smear-resin complex (Figs. 7a, c). In the MC+AG group, thickness of the smear layer seemed thinner than that in the Co group (Fig. 7c). On the other hand, no smear layer was observed in the NC group. Moreover, peritubular dentin was decalcified, with infiltration of resin cement and formation of a resin tag (Fig. 7b). Because of the low bond strength, the interface between dentin and cement was peeled off during TEM sample preparation, so observation was not possible in the Sc and Br groups.

**DISCUSSION**

The present study evaluated how temporary cement remnants affect the effectiveness of dentin bonding and identified a decontamination method that can restore the original bond strength. Three types of temporary adhesive are in use: eugenol-containing temporary cement; eugenol-free temporary cement; and polycarboxylate cement. The temporary cement used in this study was classified as a polycarboxylate cement, and contained HY-agent, “a tannic acid-strontium fluoride-zinc fluoride complex” that is known to promote mineralization. HY compound presents with properties of dentinal tubule closure and dental substrate hardening, permitting relatively long-term provisional cementation or restoration. This component also has a secondary caries-preventing effect and is widely used in dental treatments9. However, this temporary cement presents with a high level of hardness and chemical bonding to dentin/enamel, which may limit mechanical elimination using an excavator9. SEM revealed that cleaning with an air-scaler was not sufficient to remove the remnant temporary cement and zinc was detected exactly at area of remnant temporary cement on EDS. Among the components of temporary cement characteristically detected by EDS, only zinc contained in zinc oxide was not included in the composition of dentin. Mapping images of zinc showed that zinc was found in fine particles throughout the Br group. In contrast, no zinc was detected at the dentin surface in the NC group. Null hypothesis 1, predicting no difference in the state of residual temporary cement on the dentin surface after removing temporary cement by various cleaning methods, was thus rejected.

In the NC group, treatment dissolved the smear layer and inorganic substrates of dentin such as HAp due to the effects of phosphoric acid. After removing the collagenous layer by NaOCl, dental tubules were exposed and opened16-19. Unlike the Sc and Br groups in which a smear layer was confirmed, temporary cement was considered to be completely removed together with the smear layer by treatment with phosphoric acid and NaOCl. Moreover, since no smear layer was seen on the surface and exposure of dentin tubules was observed in the area of intertubular dentin in the NC group16-18,20, proportions of carbon, oxygen, phosphorus, calcium, and magnesium also differed from those of the other groups. On the other hand, Cl was also detected in the NC group, as the only component of sodium hypochlorite that remained on the adhered surface even after washing with water.

In the MC group that only received application of the MDP-containing cleaner, a region of temporary cement (diameter, 10 µm) was observed on part of the dentin surface, no change from the SEM image was seen after cleaning with the air-scaler. On the other hand, in the MC+AG group that had been rubbed with a cleaner containing MDP, no temporary cement was detected even under EDS. According to those results, agitating
with a micro-brush after application of MDP-containing cleaner appears effective for removing temporary cement. MDP-containing cleaner includes purified water. Using MDP in a moist state, a strong surfactant effect can be expected from a structure showing both a hydrophilic group terminal and a hydrophobic group terminal. MDP binds to the surface of two substances with different polarities at the end of the hydrophobic group, so the temporary cement is easily separated from the adhered surface. The ratio of carbon was high in the MC and MC+AG groups. MDP was suggested to bind to the dentin surface. According to the "adhesion-decalcification" concept, MDP can ionically interact through a phosphonic group with the Ca in HAp.

In the present study, wettability was found to be reduced due to remnant temporary cement. Contact angle was larger in the Sc and Br groups than in the Co group, but contact angle in the NC group did not differ from that in the Co group. These results do not contradict previous results that temporary cement was completely removed in the NC group. Interestingly, the MC+AG group showed a large contact angle, as in the Sc and Br groups. These results suggest that residual MDP on the dentin surface has an effect on the increase in contact angle. To confirm the effect of MDP on dentin surface, an MDP group was prepared by applying MDP to dentin free of temporary cement contamination and the contact angle was measured. As the result, the MDP group also showed a large contact angle, similar to that in the MC+AG group. Since MDP binds to dentin at the terminal of the hydrophilic group, the surface presumably becomes hydrophobic and the contact angle becomes large.

In the μTBS test used in the present study, the surface tested was so small that the variability introduced by these factors was greatly reduced. The ptf was observed in the Sc and Br groups in which temporary cement remained under SEM observation and EDS analysis. The fracture mode of the sample showed interfacial failure between dentin and resin cement significantly more frequently, followed by some mixed fractures in the dentin-resin cement-resin block. This is consistent with reports that in vitro study revealed that the cement-resin block for CAD/CAM provides sufficient bond strength. The initial bond strength of the Sc group, in which the lump-shaped temporary cement was observed, was significantly lower than that of the Co group. This clearly indicates that residual temporary cement causes adhesion inhibition. In the Br group, no remnant temporary cement was observed under SEM, but was confirmed on EDS. Although initial bond strength in the Br group did not differ significantly from that in the Co group, bond strength decreased significantly after long-term storage (1 or 6 months). The Br group would thus clearly experience a problem in long-term durability.

In the NC group, the open dentinal tubules observed under SEM might positively influence μTBS. TEM images also show the demineralization of peritubular dentin and the infiltration of resin cement into the dentinal tubules, which may provide a mechanical interlocking. However, μTBS showed no significant difference between the NC and Co groups. One possibility is that the presence of reactive residual free-radicals in Cl-containing sodium hypochlorite-treated dentin may compete with the propagating vinyl free-radicals generated during light activation of the adhesive, resulting in premature chain termination and incomplete polymerization. Furthermore, treatment with phosphoric acid followed by sodium hypochlorite gel cleaning shows the problem of marked irritation to the dental pulp and use on vital teeth is difficult even if complete cleaning is achieved. For that reason, sufficient and mild treatment to remove temporary cement is considered necessary.

The present study confirmed bond strength to a dentin surface contaminated with temporary cement was restored by removing temporary cement using MDP-containing cleaner. Bond strength in the MC+AG group did not differ significantly from that in the Co group, and the MC+AG group showed significantly higher bond strength than the NC group. Therefore, when MDP-containing cleaner was used in agitation mode to test null hypothesis 2, the notion that the ability to remove temporary cement by this new decontaminant containing MDP was not different from other cleaning methods was rejected. A sufficient adhesive interface was formed because MDP has a polymerizable group, which can react both with other adhesive monomers and the restorative material by copolymerization, and an acid adhesive capable of both etching dental hard tissues and interacting with tooth substance. MDP was considered to have bonded to dentin at the phosphate group and to have formed a sufficient adhesive interface by copolymerization with the monomer contained in the resin cement at the opposite methacryl group. The self-adhesive resin cement used in this experiment also contained MDP. If a cement that does not contain MDP had been used, the MC+AG group might show a significant difference from the Co group.

The MC+AG group did not show the resin tag found in the NC group, but sufficient bonding was obtained. The MDP-containing cleaner used in this study had a high pH of 4–5, so the smear layer could not be removed (Fig. 6c). The thinner smear layer compared to controls was thought to mainly result from agitation. Traditionally, removal of the smear layer on prepared dentin was thought to be an irreversible change to obtain favorable bonding to resin, but the results of this study did not simply fit this theory. Given the abundant presence of HAp in this resin-smear complex and the effective chemical binding potential of the MDP functional monomer with this HAp, these residual smear particles appear very likely to have reinforced the resin-smear complex. Furthermore, the MDP in cleaner can be considered to improve the chemical bonding to dentin and the interactions between dentin and resin cement. MDP-containing cleaner can thus effectively remove the temporary cement by agitation. Additionally, the cleaner can be expected to improve the
chemical bonding ability by binding more MDP to the dentin. On the other hand, compared to MDP, a bipolar solvent may efficiently remove temporary cement at lower cost. However, remaining bipolar solvent would represent another contaminant on the dentin surface. From this perspective, MDP may represent an effective method of temporary cement removal. Further research regarding the mechanisms underlying the surfactant effect of MDP and bipolar solvent on contaminated tooth surfaces is needed.

In 2019, KATANA CLEANER (Kuraray Noritake Dental) was launched as an MDP-containing cleaner similar to the cleaner used in this study. The difference between those two cleaners was the presence or absence of sodium hydrogen phosphate and thickeners (silica). The prototype cleaner contains thickeners, but surface analysis failed to detect any silica. Moreover, KATANA CLEANER shows a relatively high pH of 4–5, the same as that of the prototype cleaner. The ability of those two cleaners should be basically the same, holding promise for improved removal of temporary cement.

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