

Available online at www.sciencedirect.com

ScienceDirect

journal homepage: www.intl.elsevierhealth.com/journals/dema

Adhesion durability of dual-cure resin cements and acid–base resistant zone formation on human dentin

San San May Phyo Aung^a, Tomohiro Takagaki^{a,c,*}, Aye Ko Ko^a,
Somayah Halabi^a, Takaaki Sato^a, Masaomi Ikeda^b, Toru Nikaido^{a,c},
Michael F. Burrow^d, Junji Tagami^a

^a Department of Cariology and Operative Dentistry, Graduate School of Medical and Dental Sciences, Tokyo Medical and Dental University, 1-5-45, Yushima, Bunkyo-ku, Tokyo 113-8549, Japan

^b Department of Oral Prosthetic Engineering, Graduate School of Medical and Dental Sciences, Tokyo Medical and Dental University, 1-5-45, Yushima, Bunkyo-ku, Tokyo 113-8549, Japan

^c Department of Operative Dentistry, Division of Oral Functional Science and Rehabilitation, School of Dentistry, Asahi University, 1851 Hozumi, Mizuho, Gifu 501-0296, Japan

^d Faculty of Dentistry, University of Hong Kong, Pokfulam, Hong Kong, China

ARTICLE INFO

Article history:

Received 26 December 2018

Accepted 13 February 2019

Keywords:

Bonding durability

Acid-base resistant zone

Adhesive resin cements

ABSTRACT

Objectives. Bond durability and resistance to acid-base challenge of dentin with dual-cure resin cement have been rarely investigated. The purpose of this study was to evaluate dentin bond strength and observe the adhesive-dentin interface after acid-base challenge using three different dual-cure resin cements.

Methods. Thirty dentin surfaces from caries-free human premolars were ground and bonded with one of three dual-cure resin cements: PANAVIA V5 (V5), ESTECCEM II (E II) and Rely X Ultimate (RXU) and thermocycled for 0, 5,000 and 10,000 cycles. A microtensile bond strength (μ TBS) test was undertaken and the interface of the bonded specimens after acid-base challenge was examined by SEM.

Results. The μ TBS of V5 exhibited a stable bond strength despite thermal cycling while the bond of EII and RXU after 10,000 cycles dropped significantly among all thermal cycle periods ($p < 0.05$). An acid-base resistant zone (ABRZ) was observed in all groups, however, the morphology of the bonded interface differed among the tested cements.

Conclusion. Bonding durability to dentin and ABRZ morphologies differed among the cements evaluated. PANAVIA V5 cement system with tooth primer provided the most reliable bond strength and was best able to resist the acid–base challenge.

© 2019 The Academy of Dental Materials. Published by Elsevier Inc. All rights reserved.

* Corresponding author.

E-mail address: takagaki.ope@tmd.ac.jp (T. Takagaki).

<https://doi.org/10.1016/j.dental.2019.02.020>

10109-5641/© 2019 The Academy of Dental Materials. Published by Elsevier Inc. All rights reserved.

1. Introduction

The retention and consequently the longevity of minimally prepared indirect restorations are strongly reliant on the adhesive qualities of the cement [1]. The micromechanical bond of dual-cure resin cements to dentin is based on the infiltration and polymerization of a synthetic resin into the collagen fibril network of the hybrid layer [2,3]. Some advantages of resin cements are low solubility, good film thickness, superior mechanical properties, optimal bonding to dental structures and reduced microleakage [4,5]. Different types of adhesive systems can be used prior to cementation with a resin cement to dentin/enamel surfaces as well as metal, ceramic or composite restorative material surfaces [6]. Recently, computer-aided design/computer-aided manufacturing (CAD/CAM) technology in dentistry has developed rapidly to the extent that design and manufacturing of dental restorations can be carried out either in a commercial dental laboratory or directly in the dental office. However, adhesion between resin CAD/CAM blocks and dual-cure resin cements may be compromised because of the high degree of polymerization of the resin-based CAD/CAM blocks [7,8].

Tsuchiya et al. firstly reported the presence of a zone beneath the hybrid layer in self-etching adhesives that was characterized by its resistance against acid and base challenges. This zone is different from the conventional hybrid layer and caries-inhibition zone observed in fluoride-releasing materials. Therefore, it was named the “acid–base resistant zone” (ABRZ), which is believed to play an important role in prevention of recurrent caries, sealing of restoration margins and promotion of restoration durability [9]. Inoue et al. investigated the ultrastructure of the ABRZ along the interface of human dentin specimens with three different bonding systems. In their study, an ABRZ was observed beneath the hybrid layer for both one-step and two-step self-etching primer systems but not with an etch and rinse bonding system that used phosphoric acid etching. There was an erosive pattern observed around the base of the ABRZ with the one-step self-etching primer system [10].

Umino et al. reported that resin cements also formed a hybrid layer and prevented artificial recurrent caries initiation [11]. However, there is less information about dual-cure resin cements on hybridization and formation of an ABRZ. ABRZ formation, in terms of morphological and mechanical characteristics, is greatly influenced by the comparison of the adhesive material. There have been many studies on the ABRZ with direct dental restorations but less information with regard to indirect restorations. Therefore, there are many interesting points about how dual-cure resin cements may achieve reliable and good bonding durability as well as understanding the morphology of ABRZ of dentin.

Based on these considerations, the purpose of this study was to investigate the effect of three dual-cure resin cement systems on the dentin bonding performance and the formation of an ABRZ. The null hypotheses to be tested were that different dual-cure resin cement systems would influence (a) the microtensile bond strength and (b) the formation of an ABRZ of dentin of human premolars.

2. Materials and methods

The compositions of the materials used in this study are listed in Table 1. Three dual-cure resin cements: PANAVIA V5 (V5, Kuraray Noritake Dental, Tokyo, Japan), ESTECHEM II (E II, Tokuyama Dental, Japan) and Rely X Ultimate (RXU, 3M ESPE, St. Paul, MN, USA) were evaluated in this study. Three adhesive systems were used in conjunction with the dual-cure resin cements tested. The adhesives are: Tooth Primer (TP) and Clearfil Ceramic Primer Plus (CP), Scotchbond Universal adhesive (SCU), both of which contain 10-methacryloxydecyl dihydrogen phosphate (10-MDP) and Bondmer Lightless (BL) consisting of a three-dimensional self-reinforcing monomer (3D-SR). The CAD/CAM block used for the bond test was Estelite block (Tokuyama Dental, Japan). The dentin substrate consisted of thirty non-carious human premolars collected after the individuals' informed consent was obtained according to a protocol approved by the Institutional Review Board of Tokyo Medical and Dental University. Verbal consent was obtained for tooth use in laboratory research under a protocol approved by the Ethics Committee of the Tokyo Medical and Dental University (No.723).

2.1. Microtensile bond strength (μ TBS) testing

The outline of the μ TBS test is schematically presented in Fig. 1. The coronal portion of each tooth was removed to expose a flat, mid-coronal dentin surface using a low-speed diamond saw (Buehler Isomet 1000, Buehler, Lake Bluff, IL, USA) under water cooling. The dentin surface was ground with wet 600-grit SiC paper to produce a standardized smear layer, and the teeth were randomly divided into three groups according to the resin cements used. Applying primer or adhesive and cementation procedure were performed according to the manufacturers' instructions. For V5 group, TP was applied to the dentin for 10 s and CP to the Estelite block for 10 s followed by air-blowing to remove excess material. For E II group, BL was applied to both the dentin and Estelite block for 10 s and afterwards was air-thinned. Similarly, SBU was also applied to both substrates (dentin and Estelite block) for 10 s respectively and air-thinned for 5 s followed by irradiation with a visible light curing unit (G-Light Prima-II Plus, 1200 mW/cm², GC Corp.; Japan). Resin cement was placed onto the dentin surface and the 2 mm-thick Estelite block. The CAD/CAM blocks were alumina-sandblasted with 50 μ m particles at 0.15 MPa and then treated with primer or adhesive and light-cured with a visible light curing unit (G-Light Prima-II Plus, 1200 mW/cm², GC Corp; Japan) for 40 s. After 24 h storage in water at 37 °C, the bonded specimens were sectioned perpendicularly at the resin/dentin interface into serial slabs using a low-speed diamond saw (Buehler Isomet 1000, Buehler, Lake Bluff, IL, USA). Each slab was further sectioned into 1.0 mm \times 1.0 mm resin-dentin beams. Approximately 6 beams were obtained from each of the 10 teeth per material group, yielding a total of 60 beams for bond strength evaluation. The specimens in each material group were further divided into three subgroups. One subgroup was tested after 24 h (n=10), and the other subgroups were thermocycled (Tokyo Giken, Tokyo, Japan) for either 5000 or 10,000 times between 5 °C and 55 °C with a dwell

Table 1 – Materials used in this study.

Material	Composition	Manufacturer	Batch Number
Clearfil Ceramic Primer Plus (CP)	ethanol, ^a γ-MPTS, ^b 10-MDP	Kuraray Noritake Dental	B90031
Tooth Primer (TP)	10-MDP, ^c HEMA, hydrophilic aliphatic dimethacrylate, accelerators, water	Kuraray Noritake Dental	2U0035
PANAVIAV5 (V5)	Paste A: ^d Bis-GMA, ^e TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, initiators, accelerators, silanated barium glass filler, silanated fluoroaluminosilicate glass filler, colloidal silica Paste B: Bis-GMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, silanated aluminium oxide filler, accelerators, dl-Camphorquinone, pigments	Kuraray Noritake Dental	BF0042
BONDMER Lightless (BL)	phosphoric acid monomer, Bis-GMA, TEGDMA, HEMA, MTU-6 (thiouracil monomer), silane coupling agent, peroxide, borate catalyst, acetone, isopropanol and purified water.	Tokuyama Dental	001057
ESTECHEM II (E II)	Bis-GMA, TEGDMA, ^f BisMPEPP, peroxide, camphorquinone and silica-zirconia Filler (Filler Load 74 wt%)	Tokuyama Dental	007007
Schotchbond Universal Adhesive (SBU)	10-MDP, dimethacrylate resins, HEMA, methacrylatemodified polyalkenoic acid copolymer, filler, ethanol, water, initiators, and silane	3M ESPE	41258S
Rely X Ultimate (RXU)	Base: methacrylate monomers, radiopaque silanated fillers, initiator components, stabilizers and rheological additives Catalyst: methacrylate monomers, radiopaque alkaline fillers, initiator components, stabilizers, pigments, rheological additives, fluorescence dye, dark polymerize activator for Scotchbond Universal Adhesive	3M ESPE	20170407
ESTELITE Block (A3-LT/14)	^f UDMA, TEGDMA, silica powder, silica- zirconia filler, pigments, others	Tokuyama Dental	009026

^a γ-MPTS; 3-trimethoxysilylpropyl methacrylate.

^b 10-MDP; 10-methacryloyloxydecyl dihydrogen phosphate.

^c HEMA; 2-hydroxyethyl methacrylate.

^d Bis-GMA; bisphenol A di (2-hydroxy propoxy) dimethacrylate TEGDMA; triethylene glycol methacrylate.

^e TEGDMA; triethylene glycol dimethacrylate.

^f BisMPEPP bisphenol A polyethoxy dimethacrylat.

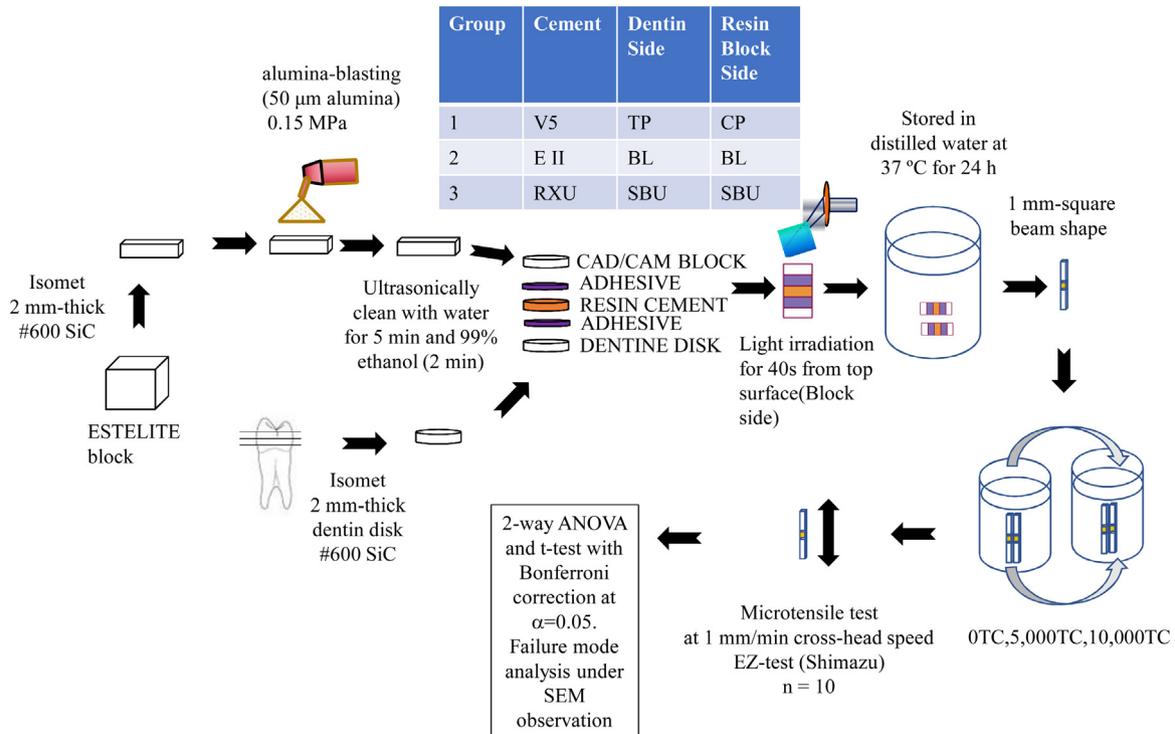


Fig. 1 – Schematic drawing for the sample preparation of μ TBS.

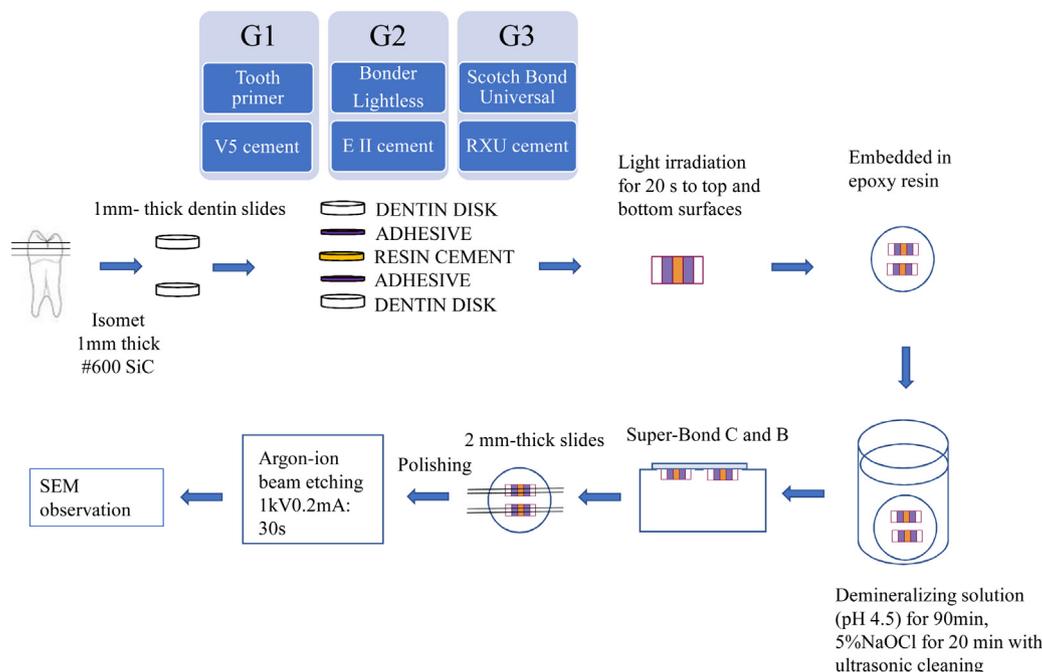


Fig. 2 – Schematic illustration of the methodology of ABRZ specimen preparation for SEM.

time of 30 s before testing. The specimens were then fixed to a universal testing device (EZ-test, Shimadzu, Kyoto, Japan) and stressed in tension at a crosshead speed of 1 mm/min.

After debonding, failure modes were inspected using a scanning electron microscope SEM (JSM-IT 100, JEOL, Tokyo, Japan) at 100 \times magnification. Failure modes were classified into four categories: A — adhesive failure at the resin-dentin interface; B — cohesive failure completely within the resin cement; C — partial adhesive failure, where remnants of resin remained on the dentin surface; and, D — cohesive failure in the dentin.

2.2. Tooth specimen preparation for ABRZ

The sample preparation method for SEM examination of the dentin-ABRZ specimens is illustrated in Fig. 2. The procedure was previously reported by Inoue et al. [10]. Thirty caries-free human premolars were used for this part of the study. Premolars were sectioned in the mid-coronal region, perpendicular to the long axis, with a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) to obtain two 1-mm-thick dentin disks. The dentin surfaces were ground with 600-grit silicon carbide paper under running water. Dentin disks were bonded with one of the three dual-cure resin cements: V5, E II and RXU then light-cured using a visible light curing unit for 20 s from the top and bottom surfaces of the specimens to make dentin disk sandwiches. After storage in distilled water for 24 h, each prepared specimen was sectioned perpendicular to the dentin-adhesive interface with the diamond saw and embedded in epoxy resin (Epoxi Cure Resin, Buehler, Lake Bluff, IL, USA). Each specimen was first stored in 100 ml of a buffered demineralizing solution (2.2 mmol/L CaCl₂, 2.2 mmol/L NaH₂PO₄, and 50 mmol/L acetic acid adjusted to a pH 4.5) for 90 min to simulate recurrent

caries. The specimens were then immersed in 5% sodium hypochlorite for 20 min to remove any demineralized dentin collagen fibrils, followed by rinsing under running water for 30 s. To protect the outer edge of the adhesive from wear which might occur during specimen polishing, 4-META/MMA-TBB resin (Super-Bond C&B, Sun Medical, Moriyama, Japan) was applied onto the treated dentin surface without acid-etching. After curing the 4-META/MMA-TBB resin, the specimens were sectioned perpendicular to the dentin-adhesive interface and reduced to a thickness of approximately 2 mm. Polishing was performed with diamond pastes (Struers A/S, Ballerup, Denmark) down to 0.25 μ m, and then the polished surfaces were etched with an argon ion beam (EIS-IE, Elionix, Tokyo, Japan) for 30 s. The specimens were gold sputter-coated, and morphological changes to the dentin-adhesive interface due to the acid-base challenge were observed using a scanning electron microscope SEM (JSM-IT 100, JEOL, Tokyo, Japan) at a magnification of 2000 \times .

2.3. Statistical analysis

As the data of the μ TBS test were normally distributed, statistical analysis was performed using 2-way ANOVA and t-test with Bonferroni correction at $\alpha = 0.05$. All the statistical analyses were performed using statistical software (SPSS 22, IBM; Armonk, NY, USA).

3. Results

3.1. μ TBS measurement

The mean μ TBS values are shown in Table 2. Two-way ANOVA and t-test with Bonferroni correction revealed that the μ TBS values were influenced by “resin cement” and “thermal

Table 2 – Mean values and standard deviations of microtensile bond strength values of all groups.

	V5	E II	RXU
0 TC	55.1 ± 6.1(10/70/20/0)	61.9 ± 6.3 ^{A, B} (10/80/10/0)	49.9 ± 5.5 ^{D, E} (20/70/10/0)
5000 TC	55.5 ± 5.1(0/90/10/0)	53.5 ± 5.2 ^{A, C} (0/80/20/0)	37.6 ± 6.0 ^{D, F} (30/30/40/0)
10,000 TC	52.6 ± 4.8(10/60/20/10)	44.7 ± 5.7 ^{B, C} (30/20/50/0)	28.8 ± 4.7 ^{E, F} (50/30/20/0)

n = 10.

Data are shown as mean ± standard deviation.

Same large superscript letters indicate statistically significant difference between bond strength ($p < 0.05$).

Percentage of failure mode (Adhesive failure at the resin-dentin interface/cohesive failure completely within the resin cement/partial adhesive failure, where remnants of resin remained on the dentin surface/cohesive failure in the demineralized dentin and intact dentin).

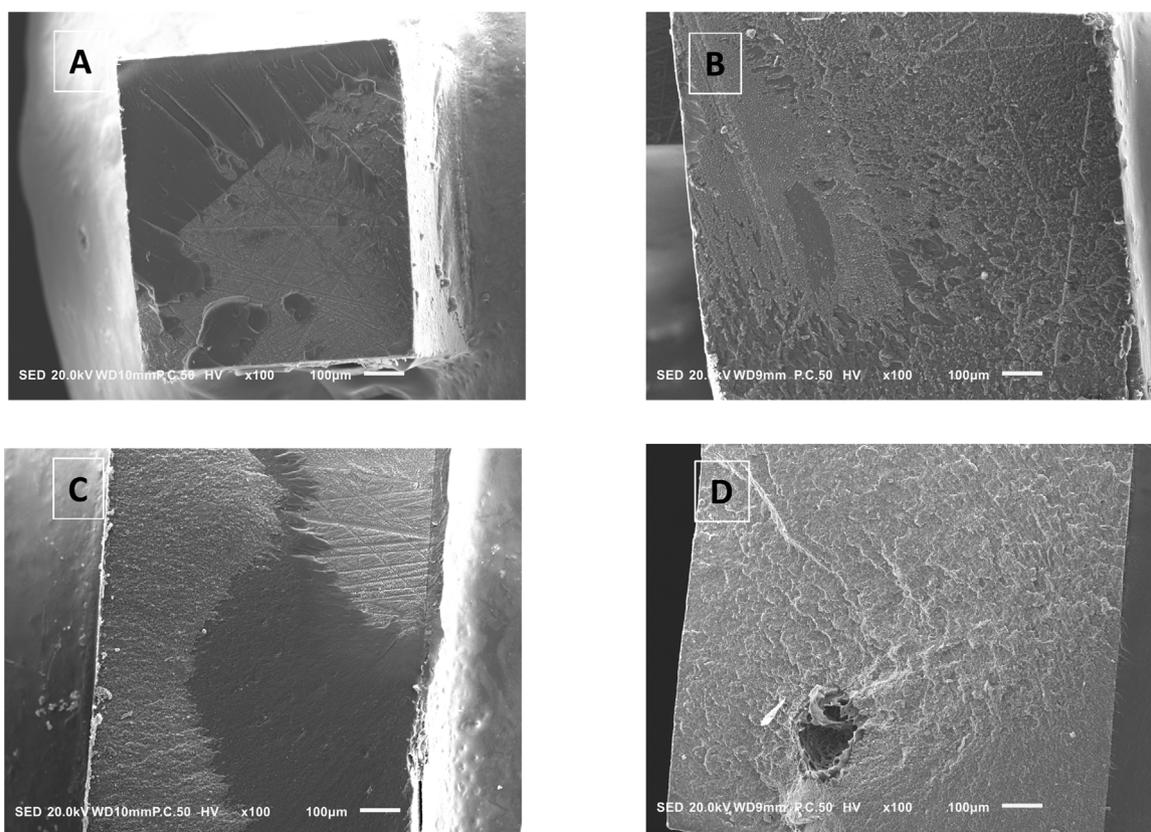


Fig. 3 – SEM images of failure modes of de-bonded surface of V5 group after 10,000 thermal cycles (magnification ×100).

(A) Adhesive failure at the resin-dentin interface **(B)** cohesive failure completely within the resin cement **(C)** partial adhesive failure, where remnants of resin remained on the dentin surface **(D)** cohesive failure in the demineralized dentin and intact dentin.

“cycling”. The mean 24-h μ TBS value for V5 was 55.1 MPa with no statistical difference found after thermocycling. There were statistical differences among the 24 h bond strength, 5000 TC and 10,000 TC groups for EII and RXU ($p < 0.05$). The 24-h bond strength of EII group was slightly higher than any other group. After 5000 and 10,000 thermal cycles, the bond strength of EII decreased gradually. The 24-h bond strength of RXU was the lowest compared the other materials and reduced significantly to 28.8 MPa after 10,000 thermal cycles.

3.2. Fracture mode analysis

No premature failure was observed in any group. The results of the fracture mode analysis are listed in Table 2 and SEM images are shown in Fig. 3. The fracture patterns of the V5 groups were predominately cohesive within the resin cement

under 0 TC, 5000 TC and 10,000 TC while adhesive failure at the resin-dentin interface was common for EII and RXU groups especially after 10,000 TC.

3.3. SEM observations of the adhesive-dentin interface after acid–base challenge

Fig. 4 shows the typical SEM images of the dentin-adhesive interface for each group after acid–base challenge. The outer lesion (OL), created by dissolution of dentin due to the acid–base challenge was observed in each group. The depth of the OL was approximately 20 μ m. The hybrid layer (HL) was hardly detected. ABRZ was observed in all groups after acid–base challenge. For V5 (Fig. 4a), a butt-joint ABRZ was observed. For EII (Fig. 4b), slight erosion was detected under-

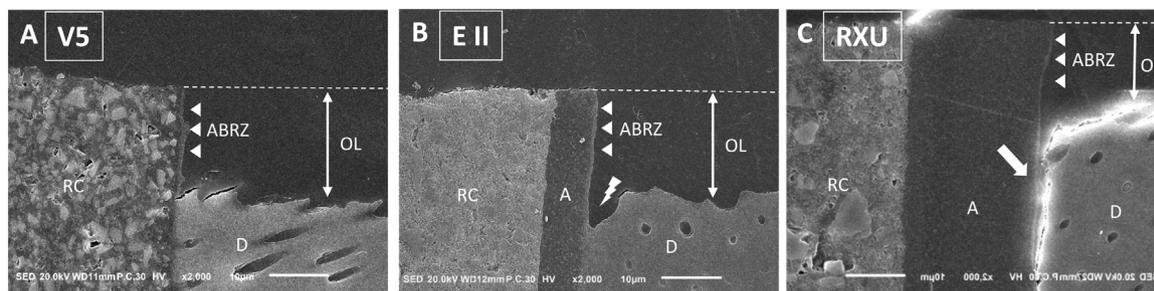


Fig. 4 – Representative SEM images of the adhesive-dentin interface after acid-base challenge (magnification $\times 2000$). ABRZ was categorized into three types; A: butt-joint with no slope formation, B: a slight erosive ABRZ at the joint (lightning) and C: ABRZ with a funnel-shaped erosion at the base of OL (arrow). RC: resin cement, ABRZ (triangle): acid-base resistant zone, A: adhesive layer, D: dentine, OL: outer lesion. Hybrid layer was hardly detected.

neath the ABRZ, and for RXU (Fig. 4c), a funnel-shaped erosion with thin ABRZ at the base of the OL was observed.

4. Discussion

The three resin cements used in this study are filled resin composites without acidic functional monomers. A former version of Panavia resin cement (Panavia F2.0, Kuraray-Noritake Dental) contained the functional monomer, 10-MDP, which has the advantage to enhance bonding performance to tooth substrates and dental materials. However, the addition of the acidic functional monomers hampered the polymerization behavior of the resin cement [12], resulting in possible reduced bonding durability (Table 2).

Cements with a high degree of conversion are also expected to provide good mechanical properties. The degree of conversion of autopolymerized cements is influenced by the concentration of the monomer and catalyst as well as the ambient temperature [13]. Generally, dual-cure resin cements have a higher degree of conversion when the dual-cure mode is used compared to only autopolymerization [14–16]. However, attenuation of light intensity of the curing light through the tooth-colored restorative materials is a critical issue in clinical procedures for dual-cure resin cements. A previous study reported that the intensity of transmitted light was decreased by 92% through a 2 mm thick resin composite disk [17]. When the dual-cure resin cement directly contacts the adhesive-treated dentin surface, the slow polymerization of the resin cement due to limited light transmission allows water to diffuse from the dentin across the adhesive into the resin cement and could form water droplets along the adhesive–cement interface [17–19]. These phenomena may cause reduced μ TBS values and lead to occurrence of adhesive failure between dentin and the resin cement. V5 and E II use “touch and cure” systems, which plays an important role to improve dentin bonding performance. The polymerization reaction starts with the mixing of base and catalyst paste, thus activating the chemical initiator. Photoinitiation allows the advancement of the polymerization reaction at the time a restoration is correctly placed and excess cement is removed.

The three resin cements of the current study had self-etch systems for dentin bonding, however, the bonding strategy was different for each resin cement. For V5, the one-bottle

self-etching primer, TP was used prior to application of the V5 resin cement. TP contains the functional monomer 10-methacryloxydecyl dihydrogen phosphate (10-MDP). It was reported that TP was able to supersede tooth etching in the adhesive cementation process and increase dentin bond strength of V5 [20]. E II has a two-bottle auto-curing one-step self-etch adhesive BL, which contains the three-dimensional self-reinforcing monomer (3D-SR) in the Bondmer Lightless liquid A and a borate catalyst in bottle Bondmer Lightless liquid B. 3D-SR can promote monomer penetration [21]. RXU uses the light-curing one-step self-etch adhesive of SBU, which also contains 10-MDP, however, it contains higher amounts of solvent and diluent components compared to TP [20]. The SBU adhesive is light cured prior to application of the RXU resin cement. Light irradiation to the adhesive can result in chemical co-polymerization with the cement due to a specialized accelerator present in the resin cement. It may cause incomplete polymerization and impact negatively on dentin adhesion.

To age the bonded specimens artificially, thermal cycling between 5 and 55 °C was selected to simulate the effect of varying temperatures present in the oral cavity [22,23]. Interestingly, adhesive failure between the CAD/CAM resin block and adhesives was never observed during the thermal cycling in the failure mode analysis of the debonded specimens. The thermal cycling effect may promote chemisorption of the silane molecules into silica surface, enhancing polymerization of vinyl groups of the silane species on the silica surface of CAD/CAM resin block [24].

The current results of the μ TBS test show there was no statistical difference in μ TBS of V5 including the thermocycled groups ($p > 0.05$). However, significant reductions of the μ TBS were observed after the thermocycling in both E II and RXU ($p < 0.05$). Müller et al. also reported that V5 was less affected by thermal cycling in dentin bond strength than the tested resin cements; RelyX Unicem 2 Automix (RUN), Multilink Speed CEM (MLS), Panavia SA Plus (PSA), RelyX Ultimate (RUL), and Multilink Automix (MLA). They suggested the stable bonding of V5 may be due to its low water sorption [25]. The V5 cement also has the potential to release fluoride, which may contribute to dentin bond durability. The μ TBS of E II decreased gradually after thermocycling probably due to a phosphoric acid monomer containing in BL. This functional monomer

should demineralize and infiltrate dentin effectively; however, the monomer or its Ca-salts may be prone to hydrolysis in the long-term or during acid challenge in an aqueous environment, resulting in a faster demineralization in the region below the hybrid layer [26]. The RXU group provided much lower μ TBS than the E II group especially after 10,000 TC. Application of SBU tended to create a thin layer, because of the hydrophilic characteristics with large amounts of water and ethanol solvent. After gently air-drying, oxygen in the air may diffuse into the thin adhesive layer, thereby compromising the polymerization of the adhesive and reducing the dentin bond strength [27].

Sample preparation for SEM observation of dentin ABRZ was established by Inoue et al. [10] and successfully applied to other studies [28–31]. It was assumed that penetration of monomers into tooth tissue beyond the hybrid layer and the chemical interaction between the functional monomer and hydroxyapatite (HAp) may contribute to the formation of ABRZ [29,31]. In order to visualize the dentin ABRZ, dentin specimens were demineralized for 1.5 h in order to create ‘outer lesions’, and 5% NaOCl solution was then applied for 20 min with ultrasonic vibration to completely remove the dentin proteins remaining after demineralization [29]. Previous studies reported that the ABRZ formation was observed at the adhesive-dentin interface beneath the hybrid layer only in self-etch adhesive systems, but not in acid etch adhesive systems [10]. Therefore, ABRZ formation was expected in the current study, because of the self-etch adhesive systems used for bonding of the resin cements to dentin. ABRZs were observed in all the tested resin cement systems, but with different morphologies. V5 provided a butt-joint ABRZ without erosion. TP is an acidic primer with a pH around 2, which is able to simultaneously etch and sufficiently penetrate into the underlying dentin [20]. A new accelerator contained in TP plays an important role to enhance interfacial polymerization of the V5 resin cement to dentin (touch and cure). In the case of E II, the BL adhesive is quite viscous, that may negatively affect the penetration of adhesive monomers into the dentinal substrates [32,33]. It was reported that an incompletely resin-impregnated hybrid layer was produced in EII [34]. RXU showed ABRZ with funnel-shaped erosion at the base of the OL. It was reported that one-step self-etch adhesives created dentin ABRZ with funnel-shaped erosion [35]. The erosive lesion may be caused by the presence of polyalkenoic acid (PAA) copolymer in SBU. The copolymer can compete in chemically bonding to calcium of hydroxyapatite with 10-MDP, resulting in interference with the stable formation of 10-MDP-HAp salts [36]. It was reported that Vitrebond copolymer could have prevented the approach of the monomers during polymerization due to its high molecular weight [37]. Nurrohman et al. suggested that the PAA copolymer might have difficulty penetrating into the narrow interfibrillar spaces of the demineralized dentin because of high molecular weight of the PAA copolymer [38].

The current study indicated that the morphological features of ABRZ are compatible with the result of μ TBS testing. In the V5 groups, the tensile bond strengths remained stable up to 10,000 TC, with a butt-jointed ABRZ without erosion being observed. Predominantly cohesive failure within resin cement was observed in the V5 groups. On the other hand, the

gradual reduction of the μ TBS after thermocycling while slight erosive ABRZ was detected in E II group. RXU showed the lowest μ TBS and the ABRZ exhibited funnel-shaped erosion. The failure mode of E II and RXU groups were mainly adhesive failure at the interface of resin and dentin, which seemed to be the weakest part of the bonded specimens. Guan et al., also pointed out that SBU formed an ABRZ with an erosive lesion and significant reduction in bond strength after 10,000 TC [35].

Within the limitations of this study, it was concluded that both dentin bonding durability of the resin cement and the ABRZ features at the interface are material dependent. V5 cement system with the tooth primer is more resistant to thermal stresses and acid–base challenge than the other resin cement systems, E II and RUX. The universal adhesives combined with resin cement systems remain questionable from the aspect of ensuring bond durability. The findings of this study led to acceptance of the null hypotheses that different adhesive resin cement systems would influence (a) the microtensile bond strength and (b) the formation of an ABRZ of dentin of human premolars. Furthermore, specific ABRZ patterns with cements after long-term water storage or thermal cycling should be considered in future studies to better illustrate bonding stability and the mechanism of ABRZ formation.

5. Conclusion

Three dual-cure resin cements: V5, E II and RXU provided different bonding performance after thermal cycling and different ABRZ morphology. V5 resin cement provided the most resistance against acid–base challenge.

Acknowledgment

This study was supported by JSPS KAKENHI (Grant No. 17K11701). The authors state that they did not have any conflict of interest.

REFERENCES

- [1] Vahey BR, Sordi MB, Stanley K, Magini RS, Novaes de Oliveira AP, Fredel MC, et al. Mechanical integrity of cement- and screw-retained zirconium-lithium silicate glass-ceramic crowns to Morse taper implants. *J Prosthet Dent* 2018, pii: S0022-3913(18)30103-3.
- [2] Cerqueira LAC, Costa AR, Spohr AM, Miyashita E, Miranzi BAS, Calabrez Filho S, et al. Effect of dentin preparation mode on the bond strength between human dentin and different resin cements. *Braz Dent J* 2018;29(3):268–74.
- [3] Zhou X, Wang S, Peng X, Hu Y, Ren B, Li M, et al. Effects of water and microbial-based aging on the performance of three dental restorative materials. *J Mech Behav Biomed Mater* 2018;80:42–50.
- [4] Loguercio AD, Hass V, Gutierrez MF, Luque-Martinez IV, Szezs A, Stanislawczuk R, et al. Five-year Effects of Chlorhexidine on the In Vitro Durability of Resin/Dentin Interfaces. *J Adhes Dent* 2016;18(1):35–42.
- [5] Nikaido T, Tagami J, Yatani H, Ohkubo C, Nihei T, Koizumi H, et al. Concept and clinical application of the resin-coating technique for indirect restorations. *Dent Mater J* 2018;37(2):192–6.

- [6] Isolan CP, Sarkis-Onofre R, Lima GS, Moraes RR. Bonding to sound and caries-affected dentin: a systematic review and meta-analysis. *J Adhes Dent* 2018;20(1):7–18.
- [7] Ali A, Takagaki T, Nikaido T, Abdou A, Tagami J. Influence of ambient air and different surface treatments on the bonding performance of a CAD/CAM composite block. *J Adhes Dent* 2018;20(4):317–24.
- [8] Shinagawa J, Inoue G, Nikaido T, Ikeda M, Burrow MF, Tagami J. Early bond strengths of 4-META/MMA-TBB resin cements to CAD/CAM resin composite. *Dent Mater J* 2018, <http://dx.doi.org/10.4012/dmj.2017-438>.
- [9] Van Landuyt KL, Snauwaert J, De Munck J, Peumans M, Yoshida Y, Poitevin A, et al. Systematic review of the chemical composition of contemporary dental adhesives. *Biomaterials* 2007;28(26):3757–85.
- [10] Inoue G, Nikaido T, Foxton RM, Tagami J. The acid-base resistant zone in three dentine bonding systems. *Dent Mater J* 2009;28(6):717–21.
- [11] Umino A, Nikaido T, Tsuchiya S, Foxton RM, Tagami J. Confocal laser scanning microscopic observations of secondary caries inhibition around different types of luting cements. *Am J Dent* 2005;18(4):245–50.
- [12] Tagami A, Takahashi R, Nikaido T, Tagami J. The effect of curing conditions on the dentin bond strength of two dual-cure resin cements. *J Prosthodont Res* 2017;61(4):412–8.
- [13] Meereis CTW, Münchow EA, de Oliveira da Rosa WL, da Silva AF, Piva E. Polymerization shrinkage stress of resin-based dental materials: A systematic review and meta-analyses of composition strategies. *J Mech Behav Biomed Mater* 2018;8:268–81.
- [14] Braga RR, Condon JR, Ferracane JL. Vitro wear simulation measurements of composite versus resin-modified glass ionomer luting cements for all-ceramic restorations. *J Esthet Restor Dent* 2002;14:368–76.
- [15] Gultekin P, Pak Tunc E, Ongul D, Turp V, Bultan O, Karataş B. Curing efficiency of dual-cure resin cement under zirconia with two different light curing units. *J Istanbul Univ Fac Dent* 2015;49(2):8–16.
- [16] Rohr N, Müller JA, Fischer J. Influence of ambient temperature and light-curing moment on polymerization shrinkage and strength of resin composite cements. *Oper Dent* 2018, <http://dx.doi.org/10.2341/17-085-L>.
- [17] Seki N, Nakajima M, Kishikawa R, Hosaka K, Foxton RM, Tagami J. The influence of light intensities irradiated directly and indirectly through resin composite to self-etch adhesives on dentin bonding. *Dent Mater J* 2011;30:315–22.
- [18] Souza EM, De Munck J, Pongprueksa P, Van Ende A, Van Meerbeek B. Correlative analysis of cement-dentin interfaces using an interfacial fracture toughness and micro-tensile bond strength approach. *Dent Mater* 2016;32(12):1575–85.
- [19] Nawareg MM, Zidan AZ, Zhou J, Chiba A, Tagami J, Pashley DH. Adhesive sealing of dentin surfaces in vitro: A review. *Am J Dent* 2015;28(6):321–32.
- [20] Giannini M, Makishi P, Ayres AP, Vermelho PM, Fronza BM, Nikaido T, et al. Self-etch adhesive systems: a literature review. *Braz Dent J* 2015;26(1):3–10.
- [21] Yoshida Y, Yoshihara K, Nagaoka N, Hanabusa M, Matsumoto T, Momoi Y. X-ray diffraction analysis of three-dimensional self-reinforcing monomer and its chemical interaction with tooth and hydroxyapatite. *Dent Mater J* 2012;31(4):697–702.
- [22] de Oliveira Lino LF, Machado CM, de Paula VG, Vidotti HA, Coelho PG, Benalcázar Jalkh EB, et al. Effect of aging and testing method on bond strength of CAD/CAM fiber-reinforced composite to dentin. *Dent Mater* 2018. S0109-5641(18)30310-5.
- [23] Saraiva LO, Aguiar TR, Costa L, Cavalcanti AN, Giannini M, Mathias P. Influence of intraoral temperature and relative humidity on the dentin bond strength: an in situ study. *J Esthet Restor Dent* 2015;27(2):92–9.
- [24] Arksornnukit M, Takahashi H, Nishiyama N, Pavasant P. Effects of heat and pH in silanation process on flexural properties and hydrolytic durabilities of composite resin after hot water storage. *Dent Mater J* 2004;23(2):175–9.
- [25] Müller JA, Rohr N, Fischer J. Evaluation of ISO 4049: water sorption and water solubility of resin cements. *Eur J Oral Sci* 2017;125(2):141–50.
- [26] Yoshida Y, Nagakane K, Fukuda R, Nakayama Y, Okazaki M, Shintani H, et al. Comparative study on adhesive performance of functional monomers. *J of Dent Res* 2004;83:454–8.
- [27] Sofan E, Sofan A, Palaia G, Tenore G, Romeo U, Migliau G. Classification review of dental adhesive systems: from the IV generation to the universal type. *Ann Stomatol (Roma)* 2017;8(1):1–17.
- [28] Shinohara MS, Yamauti M, Inoue G, Nikaido T, Tagami J, Giannini M, et al. Evaluation of antibacterial and fluoride-releasing adhesive system on dentin? microtensile bond strength and acid-base challenge. *Dent Mater J* 2006;25:545–52.
- [29] Waidyasekera K, Nikaido T, Weerasinghe DDS, Ichinose S, Tagami J. Reinforcement of dentin in self-etch adhesive technology: a new concept. *J Dent* 2009;37:604–9.
- [30] Nikaido T, Weerasinghe DDS, Waidyasekera K, Inoue G, Foxton RM, Tagami J. Assessment of the nanostructure of acid-base resistant zone by the application of all-in-one adhesive systems: Super dentin formation. *Biomed Mater Eng* 2009;19:163–71.
- [31] Iida Y, Nikaido T, Kitayama S, Takagaki T, Inoue G, Ikeda M, et al. Evaluation of dentin bonding performance and acid-base resistance of the interface of two-step self-etching adhesive systems. *Dent Mater J* 2009;28:493–500.
- [32] Kim JH, Han GJ, Kim CK, Oh KH, Chung SN, Chun BH, et al. Promotion of adhesive penetration and resin bond strength to dentin using non-thermal atmospheric pressure plasma. *Eur J Oral Sci* 2016;124(1):89–95.
- [33] Abdul Aziz AH. Comparison of Viscosity among Four Orthodontic Adhesive Primers in Two Different Temperatures. *J Dent Biomater* 2015;2(4):149–54.
- [34] Miyazaki M, Onose H, Moore BK. Analysis of the dentin-resin interface by use of laser Raman spectroscopy. *Dent Mater* 2002;18(8):576–80.
- [35] Guan R, Takagaki T, Matsui N, Sato T, Burrow MF, Palamara J, et al. Dentin bonding performance using Weibull statistics and evaluation of acid-base resistant zone formation of recently introduced adhesives. *Dent Mater J* 2016;35(4):684–93.
- [36] Yoshida Y, Yoshihara K, Nagaoka N, Hayakawa S, Torii Y, Ogawa T, et al. Self-assembled nano-layering at the adhesive interface. *J of Dent Res* 2012;91(4):376–81.
- [37] Muñoz MA, Luque I, Hass V, Reis A, Loguercio AD, Bombarda NH. Immediate bonding properties of universal adhesives to dentin. *J Dent* 2013;41(5):404–11.
- [38] Nurrohman H, Nikaido T, Takagaki T, Sadr A, Ichinose S, Tagami J. Apatite crystal protection against acid-attack beneath resin-dentin interface with four adhesives: TEM and crystallography evidence. *Dent Mater* 2012;28:e89–98.