



Can a New HEMA-free Two-step Self-etch Adhesive Improve Dentin Bonding Durability and Marginal Adaptation?

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Purpose: A new two-step self-etch adhesive (2-SEA) free of hydrophilic monomers was evaluated. Its microtensile bond strength (μ TBS) to dentin under various aging conditions, interfacial gap formation, water sorption/solubility, and formation of an acid-base resistant zone (ABRZ) were evaluated and compared with a gold-standard 2-SEA.

Materials and Methods: The new 2-SEA G2-Bond Universal (G2; GC) was compared to Clearfil SE Bond 2 (CSE2, Kuraray Noritake). Their μ TBS to sound coronal dentin was tested after 1 week, 10,000 thermal cycles (TC), 20,000 TC, 6 months and 1 year. Failure mode was determined using scanning electron microscopy (SEM). Gap formation at the interface of 2-mm deep tapered cavities with an enamel border was observed using swept-source optical coherence tomography after 1 day, 1 week, 10,000 TC, and 20,000 TC. In addition, water sorption and solubility of the bonding agents was measured, and ABRZ formation was evaluated using SEM.

Results: There was no significant difference in μ TBS between G2 and CSE2 ($p > 0.05$), and the aging conditions had no significant effect on μ TBS ($p > 0.05$). In all groups, cohesive failures prevailed (55%-95%). Interfacial gap formation was initially similar for both adhesives ($p > 0.05$), but G2 exhibited a significantly lower gap formation than CSE2 after TC ($p < 0.05$). The water sorption of G2 was significantly lower compared to CSE2 ($p < 0.05$), and their solubility was statistically similar ($p > 0.05$). ABRZ of similar thickness was observed with both adhesives.

Conclusion: The new 2-SEA exhibited stable dentin bonding and increased hydrophobicity resulting from the absence of hydrophilic monomers.

Keywords: adhesion, hydrophilicity, HEMA, microtensile bond strength, aging, gap formation, optical coherence tomography, acid-base resistant zone.

J Adhes Dent 2021; 23: 505–512.
doi: 10.3290/j.jad.b2288233

Submitted for publication: 21.02.21; accepted for publication: 07.05.21

Self-etch adhesives (SEAs) have been gaining popularity over etch-and-rinse adhesives (ERAs), as they do not require a separate etching step. Consequently, their application is faster and less technique sensitive, because the simultaneous etching and priming of dentin enables dry

bonding. Furthermore, the demineralizing effect of the acidic monomers contained in SEAs is much milder compared to that of phosphoric acid, decreasing the incidence of post-operative sensitivity.⁴² SEAs are either two-step systems (2-SEAs) consisting of a self-etching primer and a

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bonding agent, or one-step systems (1-SEAs) containing both these components in a single bottle. Systematic reviews of clinical trials reported excellent retention of mildly acidic 2-SEAs ($\text{pH} \geq 1.5$),^{10,26} slightly superior even to three-step ERAs (3-ERAs). In addition, 2-SEAs exhibited the lowest intragroup variability, as opposed to 1-SEAs, whose performance was more product dependent.^{10,26}

Nevertheless, achieving durable adhesion to the intrinsically moist dentin remains an issue. Dentin priming is usually facilitated by hydrophilic monomers, but the frequently used 2-hydroxyethyl methacrylate (HEMA) was proven to increase water sorption into the adhesive layer^{11,33} and its susceptibility to hydrolytic degradation.^{4,6} Some HEMA-free adhesives were launched, but since HEMA helps maintain the miscibility of hydrophilic and hydrophobic components,⁴¹ phase separation was observed.³⁹ Recent studies examining the effects of hydrophobic coating on the bonding performance of 1-SEAs revealed that the application of bonding agents in a second step dramatically reduced the number of voids caused by phase separation in the adhesive layer of a HEMA-free 1-SEA (G-Premio Bond [GPB]; GC, Tokyo, Japan).^{1,37,43} The hydrophobic layer also significantly improved the immediate and aged bond strength of GPB.^{1,37,43}

Based on these findings, a new HEMA-free 2-SEA, G2-Bond Universal (G2; GC), was designed. The primer of G2 contains the same functional monomers as GPB, ie, 4-methacryloxyethyl trimellitic acid (4-MET), 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), and 10-methacryloyloxydecyl dihydrogen thiophosphate (MDTP), and it is also solvated by acetone. The bonding agent of G2 reportedly contains only hydrophobic monomers to ensure ideal co-polymerization with resin composites and long-term stability, as functional monomers could increase water sorption and hence the risk of hydrolytic degradation. It has previously been reported that the presence of 10-MDP in the bonding agent of a 2-SEA decreases the ultimate tensile strength of the resin upon storage in water,^{15,20} as well as the bond strength to enamel¹⁵ and dentin²⁰ after *in vitro* aging. On the other hand, the presence of 10-MDP in the bonding agent has been shown to improve the immediate bond strength to dentin and produced an acid-base resistant zone (ABRZ) beneath the hybrid layer.²⁰

The objective of this study was to evaluate G2 and compare its performance to the HEMA- and 10-MDP-containing 2-SEA Clearfil SE Bond 2 (CSE2; Kuraray Noritake; Tokyo, Japan). CSE2 is currently considered a gold standard, as its predecessor, Clearfil SE Bond (CSE), had the lowest annual failure rate among the available 2-SEAs²⁶ and an excellent 13-year success rate of 93% with selective enamel etching and 86% without it.²⁷ In this study, adhesion under various aging conditions was evaluated using the microtensile bond strength (μTBS) test, and swept-source optical coherence tomography (SS-OCT) was employed to analyze interfacial gap formation. In addition, the water sorption/solubility of the bonding agents and the formation of ABRZ were examined. The null hypothesis tested in this study was that there would be no difference between the adhesives' performance in any of the tests.

MATERIALS AND METHODS

The research protocol of this study was approved by the Human Research Ethics Committee of Tokyo Medical and Dental University (No. 2014-022). The composition and application procedures of the tested 2-SEAs (G2 and CSE2) are listed in Table 1. Table 1 also presents the composition of the hybrid resin composite (Clearfil AP-X, shade A2, Kuraray Noritake) used for the preparation of μTBS specimens and the flowable resin composite (Clearfil Majesty Flow, shade A2, Kuraray Noritake) used for the preparation of specimens to observe of gap formation and ABRZ.

μTBS Test

Specimen preparation

Twenty sound human molars were used for the μTBS test. The teeth were placed in a 0.5% chloramine-T solution for 1 week and then stored in distilled water at 4°C until use (within six months of extraction). Occlusal enamel was removed using a low-speed diamond saw (Isomet 1000, Buehler; Lake Bluff, IL, USA) under water cooling to expose mid-coronal dentin surfaces, which were ground with a 400-grit SiC paper (DCCS, Sankyo Fuji Star; Saitama, Japan) under running water to produce a standardized smear layer.¹⁴ Half of the teeth were bonded with G2, while the other half were bonded with CSE2 according to the instructions for use. Light curing was performed using a Valo LED LCU (Ultradent; South Jordan, UT, USA) in standard mode (1000 mW/cm²) at a distance of approximately 2 mm for 10 s. The bonded surfaces were built up with Clearfil AP-X in three increments to a thickness of approximately 5 mm. Each increment was light cured for 20 s using the Valo lamp in standard mode. After 1 week, the specimens were sectioned into 10 non-trimmed sticks (1x1 mm in cross section) using a low-speed diamond saw (Isomet 1000, Buehler) under water cooling. From each specimen, a pair of sticks was tested immediately, and the remaining four pairs were assigned to the following aging conditions: 10,000 thermal cycles (TC), 20,000 TC, 6-month water storage, and 1-year water storage. The sticks stored in water were incubated at 37°C, and the water was changed every month. Thermocycling was conducted between 5°C and 55°C with a dwell time of 30 s and a transfer time of 2 s.

μTBS testing and statistical analysis

After the respective aging procedures, the sticks were attached to an active testing jig using a cyanoacrylate adhesive (Model Repair II Blue, Dentsply-Sankin; Tokyo Japan) and stressed in tension using an EZ Test testing machine (Shimadzu; Kyoto, Japan) at a crosshead speed of 1 mm/min until failure. The μTBS was obtained by dividing the force at failure by the specimens' bonded area, which was determined by measuring the dimensions using a digital caliper Mitutoyo CD15 (Mitutoyo; Kawasaki, Japan). As pairs of sticks originating from a single tooth were assigned to different aging conditions, a mean μTBS was calculated for each pair. The means ($n = 10$) were statistically analyzed using a two-way repeated measures ANOVA (factors: adhe-



Table 1 Composition, batch number, and application procedure of adhesives and composites used in this study

Material (manufacturer)	Composition (batch number)	Application procedure
G2 Bond Universal (GC; Tokyo, Japan)	Primer: 4-MET, 10-MDP, MDTP, dimethacrylates, water, acetone, photoinitiator, filler (1806201) Bonding agent: dimethacrylates, filler, photoinitiator (180907)	1. Apply primer to the entire dentin surface and leave for 10 s. 2. Air dry strongly for 5 s. 3. Apply bonding agent, air blow gently for 3 s. 4. Light cure for 10 s.
Clearfil SE Bond 2 (Kuraray Noritake; Tokyo, Japan)	Primer: 10-MDP, HEMA, hydrophilic aliphatic dimethacrylate, water, CQ (C60080) Bonding agent: 10-MDP, bis-GMA, HEMA, hydrophobic aliphatic dimethacrylate, CQ, initiators, accelerators, silica (CK0122)	1. Apply primer to the entire dentin surface and leave for 20 s. 2. Air dry for 5 s with mild air pressure. 3. Apply bonding agent, air blow gently for 3 s. 4. Light cure for 10 s.
Clearfil AP-X, shade A2 (Kuraray Noritake)	Bis-GMA, TEG-DMA, silanated barium glass filler, silanated silica filler, silanated colloidal silica, CQ Inorganic filler: approx. 71 vol%, particle size range 0.02-17 μm (A90078)	1. Place an increment less than 2 mm in thickness. 2. Light cure with an LED light-curing unit for 20 s.
Clearfil Majesty Flow, shade A2 (Kuraray Noritake)	TEG-DMA, hydrophobic aromatic dimethacrylate, silanated barium glass filler, silanated colloidal silica, CQ Inorganic filler: approx. 62 vol%, particle size range 0.02-19 μm (AD0022)	1. Place an increment less than 2 mm in thickness. 2. Light cure with an LED light-curing unit for 20 s.

4-MET: 4-methacryloxyethyl trimellitic acid; 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; MDTP: 10-methacryloyloxydecyl dihydrogen thiophosphate; HEMA: 2-hydroxyethyl methacrylate; CQ: camphorquinone; bis-GMA: bisphenol-A-glycidyl methacrylate; TEG-DMA: triethyleneglycol dimethacrylate.

sive system and aging condition) to account for the multiple measurements on each tooth. Tukey's post-hoc test was used for pairwise comparisons. The significance level was set to 0.05 (SPSS Statistics 26.0, IBM; Armonk, NY, USA).

Fractographic Analysis

The fractured specimens were desiccated, coated with a fine layer of gold, and observed using a scanning electron microscope (JSM-IT100, JEOL; Tokyo, Japan). Three different failure modes were distinguished: 1. adhesive failure: more than 80% of the failure at the adhesive-dentin interface or within the adhesive layer; 2. cohesive failure within substrates: more than 80% of the failure in dentin or the resin composite; 3. mixed failure: neither type 1 nor 2 were present on at least 80% of the fracture surface.^{35,36}

Gap Formation in Tapered Cavities

Specimen preparation

Human teeth were used in a pilot study, but it was difficult to standardize the cavities due to their uneven enamel surfaces and limited dentin thickness. Therefore, ten sound bovine mandibular incisors were used. The central part of their labial surfaces was ground with a 600-grit SiC paper to flatten the surface and reduce enamel thickness to approximately 0.7 mm. Then, tapered cavities (depth: 2 mm; bottom diameter: 2 mm; top diameter: 3 mm) were prepared with a coarse diamond bur (207CR; Shofu; Kyoto, Japan) and finished with a fine diamond bur of the same shape (SF207CR; Shofu). The remaining enamel thickness and the dimensions of the cavities were checked using SS-OCT. Cav-

ities of the desired dimensions (± 0.2 mm) were rinsed with water, air dried, and bonded with G2 or CSE2 according to the manufacturers' instructions. The cavities were then filled with Clearfil Majesty Flow in one increment and light cured for 20 s using the Valo LCU in standard mode. The surfaces were flattened and polished using 1500-grit SiC paper.

SS-OCT observation and gap analysis

The specimens were observed with an SS-OCT device (IVS-2000, Santec; Tokyo, Japan) four times: after 1 day of water storage, 1 week of water storage, 10,000 TC, and 20,000 TC. Water storage and thermocycling were conducted as described for the μTBS test above. Prior to observation, the specimens were air dried and fixed on a metal stage. The area of interest (5 mm in width, 5 mm in depth) was scanned using a built-in laser (central wavelength 1330 nm, sweep rate 30 kHz) to obtain raw 2-D SS-OCT tomograms of 2000 x 1000 px resolution. Each specimen was scanned in six planes by rotating the laser beam by 0, 30, 60, 90, 120, and 150 degrees.^{2,3,16,18}

In the tomogram, gaps at the adhesive-tooth interface appear as bright spots or lines due to an increased reflection of light caused by the difference in refractive indices of the substrates and air in the gap. The gap formation was analyzed using an image-processing program (ImageJ 1.51s, National Institutes of Health; Bethesda, MD, USA). A median filter was applied to decrease the background noise, and gap formation was measured using an experimental threshold determination algorithm developed as a plugin for ImageJ.^{2,3,16,18} The region of interest was selected along

the interface, and the ratio of white spots to the total length of the interface was calculated by the plugin. A mean value was calculated for each specimen based on the data obtained in all six planes, and the means ($n = 5$) were statistically analyzed using a two-way repeated-measures ANOVA, and t-tests with a Bonferroni correction for multiple comparisons at $\alpha = 0.05$ (SPSS Statistics 26.0, IBM).

Water Sorption and Solubility Test

Water sorption was measured as described in ISO 4049:2019¹³ with slight modifications according to Kuno et al.¹⁷ Round silicone molds (diameter: 6 mm; thickness: 1 mm) placed on a glass slide covered with a transparent plastic strip were filled with the bonding agents of G2 and CSE2. They were covered with another transparent plastic strip and light cured for 20 s from both sides using the Valo LCU in standard mode to ensure sufficient polymerization. The polymerized disks ($n = 5$) were pushed out of the molds, and the periphery was ground with a 1000-grit SiC paper to remove excess material. The dimensions of the disks were measured using the Mitutoyo CD15 digital caliper for a precise calculation of volume (V).

A laboratory digital balance was used for weighing. The disks were initially stored in dry conditions at 37°C until their mass stabilized (m_1). The disks were then individually immersed in distilled water at 37°C for 10 days to obtain their mass in the wet state (m_2). Afterward, the disks were desiccated and weighed daily until a constant mass in the dry state (m_3) was obtained. Water sorption was calculated using the equation $(m_2 - m_3)/V$, and water solubility using the equation $(m_1 - m_3)/V$ (13). The difference between G2 and CSE2 was statistically analyzed using a t-test at $\alpha = 0.05$ (SPSS Statistics 26.0, IBM).

Acid-base Challenge

Ten additional sound human molars were used for the acid-base challenge.¹² Two dentin disks (1 mm in thickness) were obtained from each tooth and ground with a 400-grit SiC paper from one side to produce a standardized smear layer. Half of the disks were bonded with G2, while the other half were bonded with CSE2. The adhesives were applied according to the manufacturer's instructions with 10 s of air blowing of the bonding agents. This was done to reduce the thickness of the adhesive layer and allow its full observation at a magnification of 3500X, which is standard for the observation of the acid-base resistant zone (ABRZ).³⁸ After light curing the adhesives for 10 s, Clearfil Majesty Flow was placed between pairs of the bonded disks to form a "sandwich", which was light cured for 20 s from both sides using the Valo LCU in standard mode. After 24 h of water storage at 37°C, each specimen was sectioned in half using the Isomet 1000 (Buehler) low-speed diamond saw and embedded in an epoxy resin (EpoxiCure 2, Buehler).

The specimens were exposed to a demineralizing solution (2.2 mmol/l CaCl_2 , 2.2 mmol/l NaH_2PO_4 , 50 mmol/l acetic acid, pH adjusted to 4.5) for 90 min, followed by immersion in 6% NaOCl for 20 min. They were then ultrasonically cleaned in water, air dried, and the surface was

coated with a transparent resin (Super-Bond C&B, Sun Medical, Moriyama, Japan) to protect the specimens from wear after the acid-base challenge.¹² The specimens were sectioned perpendicular to the adhesive-dentin interface, their thickness was reduced to approximately 2 mm, and they were polished using diamond pastes (DP-Paste P, Struers; Copenhagen, Denmark) with a decreasing particle size (6 μm , 3 μm , 1 μm and 0.25 μm). Finally, the specimens were etched with an argon-ion beam (EIS-IE, Elionix, Tokyo, Japan) for 30 s at an accelerating voltage of 1 kV and an ion current density of 0.2 mA/cm². The specimens were sputter-coated with gold, and the ultrastructure of the adhesive-dentin interface after acid-base challenge was observed using the JSM-IT 100 SEM at 3500X magnification.

RESULTS

μTBS

The results of the μTBS test and the fractographic analysis are presented in Table 2. Two-way repeated-measures ANOVA revealed that the effects of adhesives ($p = 0.377$) and aging conditions ($p = 0.496$) were not significant, and nor was their interaction ($p = 0.395$). The pairwise comparisons did not reveal any significant differences between G2 and CSE2 under any of the aging conditions ($p > 0.05$). Adhesive failures were scarce (0% to 15%), whereas cohesive failures in substrates prevailed (55% to 95%).

Gap Formation

The rates of gap formation are summarized in Table 3, and representative SS-OCT images are presented in Fig 1. Gaps were observed in all specimens, mainly at the bottom of the cavities. Marginal enamel was gap-free in most specimens, but minor defects were sometimes observed, especially after thermocycling. There was no significant difference in gap formation between G2 and CSE2 after 1 day ($p = 0.129$) and 1 week ($p = 0.554$). However, thermocycling significantly increased the rate of gap formation with CSE2 ($p < 0.01$), resulting in a significantly lower gap formation with G2 after 10,000 TC ($p = 0.013$) and 20,000 TC ($p = 0.014$).

Water Sorption/Solubility

The water sorption of G2 ($48.3 \pm 2.8 \mu\text{g}/\text{mm}^3$) was significantly lower compared to CSE2 ($72.2 \pm 2.2 \mu\text{g}/\text{mm}^3$; $p < 0.001$). The solubility of G2 ($4.7 \pm 3.1 \mu\text{g}/\text{mm}^3$) was not significantly different from that of CSE2 ($5.9 \pm 3.7 \mu\text{g}/\text{mm}^3$; $p > 0.05$).

Acid-base Challenge

A total of 20 specimens were observed for each adhesive; representative images are presented in Fig 2. Dentin was demineralized to a depth of 15–20 μm , and ABRZ was present in all specimens. There was no clear difference in the thickness of ABRZ (~1–2 μm) between the adhesives, and no signs of funnel-shaped erosion adjacent to the ABRZ were observed. The hybrid layer was not distinguishable with either of the adhesives.

Table 2 Microtensile bond strength and failure mode distributions

		1 week	10,000 TC	20,000 TC	6 months	1 year
G2-Bond Universal	Mean μ TBS \pm SD (MPa)	65.2 \pm 11.7	58.1 \pm 8.3	59.3 \pm 5.2	61.5 \pm 12.9	66.7 \pm 5.5
	Failure mode (A/C/M)	3/11/6	3/15/2	0/16/4	0/19/1	1/13/6
Clearfil SE Bond 2	Mean μ TBS \pm SD (MPa)	61.9 \pm 10.7	67.1 \pm 15.7	62.8 \pm 8.7	64.1 \pm 12.5	66.7 \pm 11.6
	Failure mode (A/C/M)	1/19/0	3/14/3	3/14/3	3/13/4	2/14/4

There was no significant difference in μ TBS between the adhesives in any of the aging conditions ($p > 0.05$). The aging conditions had no significant effect on the μ TBS of each adhesive ($p > 0.05$). TC: thermal cycles; μ TBS: microtensile bond strength; SD: standard deviation; A: adhesive failure; C: cohesive failure; M: mixed failure.

Table 3 The ratio of gap formation to the total length of bonded interfaces of tapered cavities (means \pm SD in %)

	1 day	1 week	10,000 TC	20,000 TC
G2-Bond Universal	28.1 \pm 2.8 ^{Aa}	30.0 \pm 7.1 ^{Aa}	32.1 \pm 2.7 ^{Aa}	32.8 \pm 2.7 ^{Aa}
Clearfil SE Bond 2	31.7 \pm 3.9 ^{Aa}	32.6 \pm 6.5 ^{Aa}	40.7 \pm 5.5 ^{Bb}	41.3 \pm 4.7 ^{Bb}

Different superscript letters indicate significant differences between groups: uppercase letters in columns (between the adhesives) and lowercase letters in rows (between the aging conditions). TC: thermal cycles.

DISCUSSION

Excessive hydrophilicity of the adhesive layer has been recognized as a fundamental problem in the bond durability of 1-SEAs,³⁴ and several studies showed that a hydrophobic coating layer can significantly improve their adhesion to dentin.^{1,7,9,21,25,29,31,37,40,43} Since considerable improvement was obtained with GPB,^{1,37,43} a new 2-SEA, G2, was developed. Its primer is based on the chemistry of GPB, so it could retain the advantages of so-called universal 1-SEAs, such as applicability with or without phosphoric acid etching or bonding to various substrates, while the bonding agent was designed to be hydrophobic to improve bond durability.

In this study, G2 exhibited significantly lower gap formation after thermocycling and significantly lower water sorption than CSE2, so the null hypothesis had to be rejected. Both tested adhesives exhibited high μ TBSs, and no significant difference was found between them. These results are in agreement with previous studies which evaluated the μ TBS of GPB with an experimental bioactive bonding agent BZF-21^{37,43} and with the bonding agent of CSE2.¹ Moreover, none of the aging conditions had a significant effect on μ TBS, and cohesive failures in dentin or the resin composite prevailed in all groups, whereas adhesive failures were seldom observed. These findings are indicative of strong, durable adhesion. On the other hand, the low fre-

quency of adhesive failures diminishes the precision of the μ TBS test, because the strengths of non-adhesive failures do not equal bond strength.^{5,35} To reduce the frequency of cohesive failures and allow a more accurate characterization of the adhesive joint, a fracture mechanics approach might be more suitable than the μ TBS test.²⁸

The observation of interfacial gap formation using SS-OCT is another way of assessing the bonding performance of dental adhesives.³² A gap is thought to appear if polymerization shrinkage stress exceeds bond strength. Therefore, a higher rate of gap formation is indicative of weaker adhesion. It can be seen as an advantage of SS-OCT analysis that clinically representative setups can be used, such as restorations of standardized cavities, as opposed to bond strength testing methods which require a flat bonding surface. Moreover, SS-OCT is a non-destructive method that enables repeated assessments of the interface.

Since the μ TBS of both tested adhesives was high and CSE exhibited excellent sealing ability in tapered cavities with an upper diameter of 4 mm,³ cavities with a higher taper were used in this study to challenge the adhesion. The ratio of bonded to unbonded surfaces, also known as the configuration factor (C-factor),⁸ equaled 2.7. Shrinkage stress was further increased by filling the 2-mm-deep cavity in one increment with a flowable composite (Clearfil Majesty Flow), for which a maximum of 1.5 mm per increment

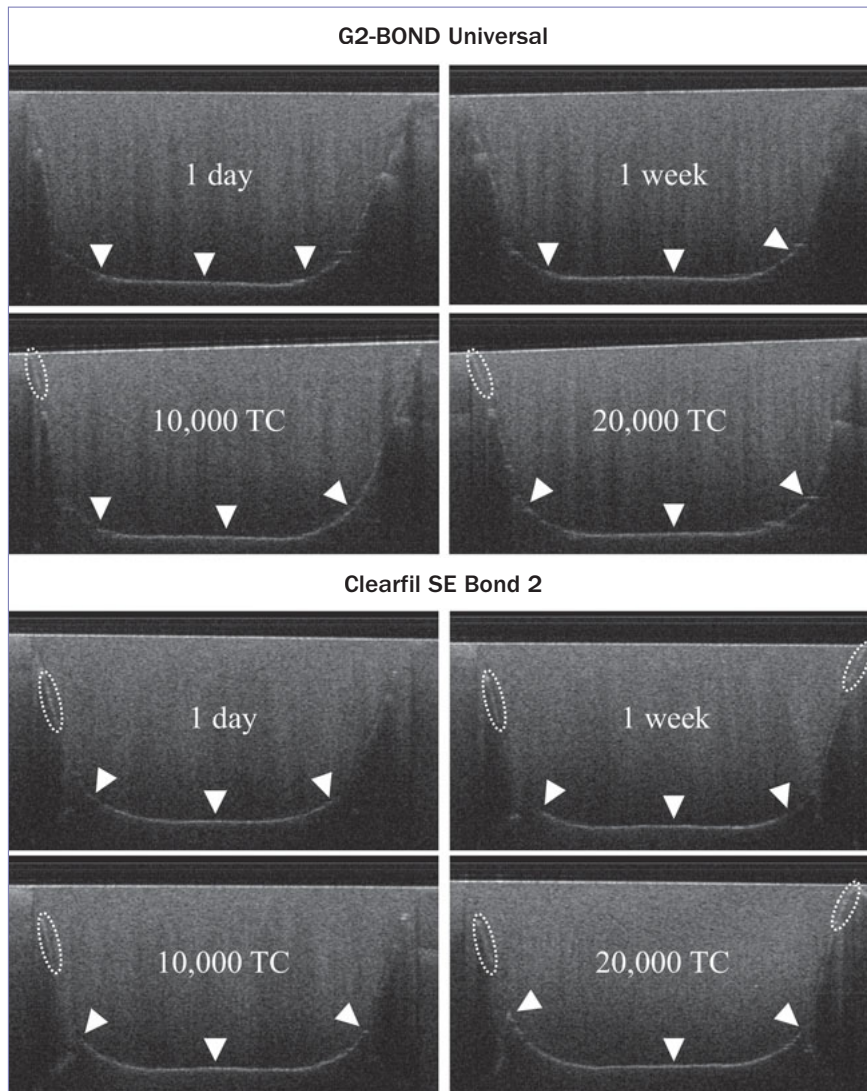


Fig 1 Representative SS-OCT images. Gap formation appears as white spots or lines at the interface and was observed in all specimens. Gaps were mainly present at the bottom of the cavities (white triangles), but minor defects at the enamel margin were sometimes observed as well (dotted ellipses). After thermocycling, the rate of gap formation with G2-Bond Universal was significantly lower than with Clearfil SE Bond 2.

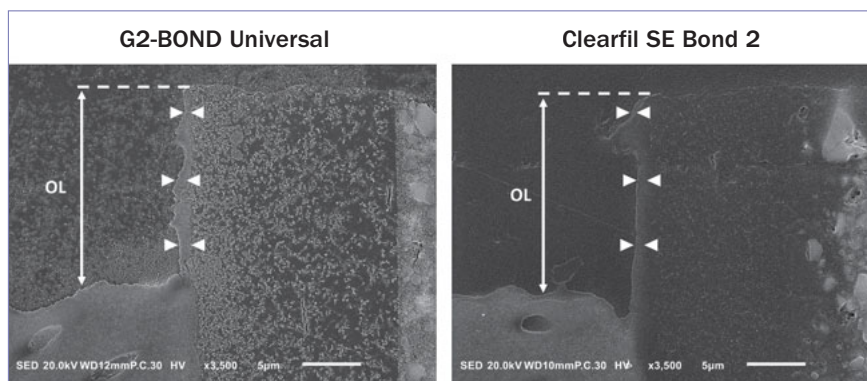


Fig 2 Representative micrographs of the adhesive interfaces after the acid-base challenge (magnification 3500X). The depth of dentin demineralization (outer lesion [OL], indicated by the double-sided arrow) was 15-20 μm . The white triangles point at the acid-base resistant zone (ABRZ), which was present in all specimens. There was no obvious difference in the thickness of ABRZ ($\sim 1\text{-}2\ \mu\text{m}$) between the adhesives, and there were no signs of funnel-shaped erosion adjacent to the ABRZ. The hybrid layer was not distinguishable with either of the adhesives.

is recommended by the manufacturer. An observed consequence of this setup was the relatively high rate of gap formation, especially at the bottom of the cavities (Fig 1). Initially, there was no significant difference between G2 and

CSE2, but gap formation with CSE2 increased significantly after thermocycling, which had no significant effect on G2. This seems to contradict the outcome of the μTBS test, but these results should not be compared directly, because

µTBS was tested on a flat surface ground with 400-grit SiC paper, while the cavities observed using SS-OCT had a high C-factor and were prepared with diamond burs. The different preparation methods led us to speculate that the bonding of G2 to bur-cut dentin/enamel was more effective compared to CSE2. This could be due to the relatively low pH of the primer of G2 (1.5), which may improve the ability of G2 to demineralize enamel and penetrate the thicker smear layer on bur-cut dentin.³⁰ However, to confirm this hypothesis, bond strength to bur-cut enamel and bur-cut dentin would have to be tested.

The stable bonding of G2 was presumably ensured by its lower hydrophilicity. While the manufacturer did not disclose which monomers G2 contains, it stated that G2 is free of HEMA and contains no functional monomers in the bonding agent, as opposed to CSE2 which contains HEMA and 10-MDP in both of its components. We assume that the presence of hydrophilic monomers contributed to the fact that CSE2 exhibited a significantly higher water sorption than G2. The extent of water solubility was low for both adhesives, which may be attributed to a high degree of conversion.¹⁹ The manufacturer of CSE2 claims that it contains a new, undisclosed catalyst system, and given that GPB contains a phosphine oxide initiator,³⁶ we speculate that it is also contained in G2.

The last test performed in this study was the observation of ABRZ, which is believed to play an important role in preventing recurrent caries by increasing the resistance to acid attacks at restoration margins.²⁴ ABRZ is created by SEAs, but the interfacial morphology after the acid-base challenge is known to be influenced by functional monomers²² and the number of application steps.²³ The present results showed no appreciable difference between the ABRZ of G2 and CSE2, and the erosion beneath ABRZ, which had been observed previously with 1-SEAs²³ or 2-SEAs without 10-MDP in the bonding agent,²⁰ was no longer apparent. This suggests that the application of a hydrophobic bonding agent can improve the quality of the resin-dentin interface of 1-SEAs, as the composition G2's primer is similar to GPB. It is also noteworthy that phase separation was not observed, although it is common in HEMA-free 1-SEAs. Based on the results of previous studies, it seems that this phenomenon is eliminated when a bonding agent is applied in the second step.^{1,37,43}

CONCLUSION

Further tests are necessary to confirm the effectiveness of G2, but within the limitations of this study, it can be concluded that it exhibited durable bonding to dentin. The performance of G2 was similar or even better than that of the gold-standard CSE2, possibly due to the absence of hydrophilic monomers.

ACKNOWLEDGEMENTS

This work was supported by the Ministry of Education, Culture, Sports, Science, and Technology of Japan (Grant Numbers 18K09571 and 19K10106) and Charles University (project PROGRES Q29/1LF). Kuraray Noritake and GC are gratefully acknowledged for providing the materials used in this study. The authors would also like to thank Dr. Ahmed Abdou and Dr. Pavel Bradna for their assistance with the statistical analyses.

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Clinical relevance: Restorations bonded using the new two-step self-etch adhesive free of hydrophilic monomers, G2-Bond Universal, may have improved marginal adaptation, and their adhesion to dentin should be stable in the long term due to increased hydrophobicity.