Evaluating the advancements in a recently introduced universal adhesive compared to its predecessor

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Abstract *Background/purpose:* The dental adhesive market is constantly evolving to meet the demands of dentists and patients, but new products and upgrades should be rigorously evaluated before being used in clinical practice. This study investigated the physicomechanical properties and dentin bonding efficacy of a newly upgraded universal adhesive compared to its predecessor.

Materials and methods: Twenty-four molars were divided into four groups (n = 6/group) based on adhesive (new vs. predecessor) and application mode [self-etch (SE) vs. etch-and-rinse (ER)] for evaluating their dentin microtensile bond strength (μTBS), failure pattern, and bonding interface. Additional thirty-six molars' crowns were perpendicularly sectioned to obtain flat mid-coronal dentin discs. The opposing dentin surfaces of each disc received contrasting treatments (new/predecessor adhesive applied in SE/ER mode), resulting in six

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interventions. The bonded discs (n = 6/intervention) were used to assess the adhesives' survival probability employing a double-sided μTBS test. The other physicomechanical properties examined were adhesives' oxygen inhibition layer (OIL), viscosity, hardness, elastic modulus, degree of conversion (DC), and in-situ DC.

Results: Both adhesive versions showed similar μTBS ($P > 0.05$), failure pattern ($P > 0.05$), and survival probability ($P > 0.008$). ER mode promoted resin tag formation and exhibited a slender adhesive layer for both adhesives. The newer adhesive version showed a thinner adhesive layer in general with narrower OIL ($P < 0.001$), less viscosity ($P < 0.001$), higher hardness ($P < 0.05$), elastic modulus ($P < 0.05$), DC ($P < 0.001$), and in-situ DC ($P < 0.001$).

Conclusion: While the newly updated adhesive had superior physicomechanical properties with more fluidity, its dentin bonding efficacy and survival probability were comparable to its predecessor.

Introduction

Dental bonding systems have undergone significant developments in recent years, primarily driven by the increasing demand for adhesive restorations. The latest trend in this field is the universal or multi-mode adhesive system. Although the term 'universal adhesive' appeared in a few articles in the early 1990s, it now refers to an adaptable, multi-mode adhesive system that can be used in etch-and-rinse, self-etch, or selective-etch modes.[1,2] In addition, it claims versatile applicability in direct or indirect restorations.

Scotchbond™ Universal Adhesive (also known as Single Bond Universal Adhesive in different regions of the world) was the first multi-mode or universal adhesive system launched in 2011.[3] By definition, this universal adhesive can be applied on enamel or dentin with or without etching.[3] The selection of etch-and-rinse or self-etch application modes lies in their unique bonding mechanisms, hybrid layer characteristics, and the formulation of the adhesives, emphasizing the crucial role of both application techniques and material composition in achieving effective bonding.[4] While etching exposes the dentinal collagen network, allowing adhesive monomers to diffuse and form a $3-5$ μm thick hybrid layer resulting in diffusion-based micromechanical interlocking, self-etch mode utilizes the mild etching effect of acidic functional monomers to form a submicron hybrid layer where the adhesive adheres through microretention and chemical interaction.[5] However, most universal adhesives contain a delicate mixture of hydrophilic and hydrophobic components in a single container, which is less of a problem for etched enamel than for more hydrated, less mineralized dentin.[2] Unlike the two-step self-etch adhesives, degradation of dentin bonding for universal adhesives has been commonly observed over time.[6] Despite this, the universal adhesive market is escalating, with new products or upgrades to meet the overwhelming influence of versatility and convenience.

Scotchbond Universal Plus Adhesive is a newly upgraded version of the Scotchbond Universal Adhesive.[7] According to the manufacturer, it has several advantages over its predecessor, such as a longer silane chain that eliminates the need for a separate primer in ceramic restorations, a built-in dual cure activator, and a Bisphenol A-free radiopaque cross-linking resin.[7] However, studies found conflicting results regarding the effectiveness of glass ceramic bonding when the adhesive was applied without a primer.[8] Also, transitioning from the commonly used bisphenol A-glycidyl methacrylate (Bis-GMA) to another resin may impact the mechanical properties and viscosity of the adhesives.[4] Therefore, despite marketing claims, a newly developed dental adhesive cannot be guaranteed to behave predictably across various laboratory and clinical situations unless evaluated extensively.[9] Given the limited available information on the direct dentin bonding efficacy and other physicomechanical characteristics of the new Scotchbond Universal Plus Adhesive, this study sought to compare these with those of its predecessor, Scotchbond Universal Adhesive. Since the predecessor version exhibited lower susceptibility to various application modes,[4] the upgraded version should be subjected to a similar bonding outcome assessment, considering its compositional alterations. The microtensile bond strength test is a widely established and highly regarded method for assessing the bonding outcome.[10] Although this method offers several advantages, including reduced defect formation, localized bond strength assessment, and mitigated stress distribution heterogeneity over other methods, the results vary significantly among researchers despite similar bonding systems and experimental conditions.[11] These variations stem from substrate variability, particularly in dentin and material properties. The design of the double-sided microtensile adhesion strength test, which is derived from the principles of the sporting game "tug-of-war," can allow simultaneous evaluation of adhesives while maintaining uniform experimental conditions with the aim of determining the direct comparison in terms of adhesive survival probability.[12] In addition, evaluating the physicomechanical behavior of adhesives can provide insights into the performance of recently marketed materials. This information is precious for clinicians to decide whether to retain the previous version of an adhesive or replace it with a new one.

Therefore, the study aimed to assess the bonding outcome and physicomechanical properties of two versions

of universal adhesives from the same manufacturer according to their application mode. The null hypotheses tested were that there would be no difference in (1) dentin bonding efficacy and the survival probability for direct resin composite restoration, considering different application modes, and (2) other physicomechanical characteristics between both adhesive versions.

Materials and methods

Study design

This research was a quantitative, qualitative, and prospective laboratory study. The independent variables were adhesives (2 levels) and application modes (2 levels), and the dependent variables were microtensile bond strength (μTBS), failure pattern, survival probability, oxygen inhibition layer (OIL), viscosity, hardness, elastic modulus, and degree of conversion (DC). Sound human third molars were collected with the patients' informed consent and approved by the local Ethics Committee (protocol # 2018-9). All the teeth were cleaned and stored in a 0.5 wt% aqueous chloramine-T solution at 4 °C and used within six months after extraction. The teeth and the restorative materials were removed from the refrigerator and kept at room temperature for at least 1 h before testing. The details of the adhesives and their application modes are shown in Table 1.

Microtensile bond strength test and failure pattern analysis

Twenty-four molars were cut to expose the occlusal dentin and then grounded with 600-grit silicone-carbide papers

(SiC) to produce standardized smear layers.[13] The adhesives were then applied to dentin in self-etch (SE) and etch-and-rinse (ER) modes according to the manufacturer's instructions (Table 1), leading to four groups (six teeth per group). Four mm of light-cure resin composite (Clearfil AP-X, Shade A3, Kuraray Dental, Tokyo, Japan) restorations were then built up in increments of no more than 1.5 mm, and each layer was cured for 20 s. The light-curing unit used was an LED unit (Pencure 2000, J. Morita Corp., Tokyo, Japan), having a light-wave range of 430—490 nm and power output (irradiance) of 1000 mW/cm². After storing the specimens in distilled water at 37 °C for 24 h, the μTBS of the bonded specimens were determined with a universal testing machine (EZ-S, Shimadzu Co., Kyoto, Japan) using the non-trimming technique following a standard protocol and adhering to the Academy of Dental Materials guidance.[13]

After the μTBS test, the failure patterns were observed with a stereomicroscope (SMZ-171-TLED, Shimadzu, Kyoto, Japan) at 50× magnification and were classified into interfacial failure between dentin/resin composite and adhesive (adhesive failure), cohesive failure exclusively in dentin (dentin failure), or resin composite (composite failure) and, mixed adhesive-cohesive failure (mixed failure).[14] To simplify, dentin, composite, and mixed failures were reclassified as non-adhesive failures.[15]

Adhesives' survival probability with double-sided microtensile bond strength test

Flat dentin discs (1.0 ± 0.02 mm thick) were obtained by cutting the coronal portion of thirty-six molars perpendicular to their long axes. Both sides of the discs were ground with 600-grit SiC. The contrasting adhesives (SP/SB) were

Table 1	Dental adhesives tested in the study.			
Adhesives (codes; manufacturers; LOT no.)	pH[a]	Composition[a,b]	Application modes[c]	
			Self-etch (SE)	Etch-and-rinse (ER)
Scotchbond Universal Plus Adhesive (SP; 3M Oral Care, Seefeld, Germany; 6944803)	2.7	10-MDP, 1,3-benzenediol 2-(2-hydroxyethoxy)ethyl 3-hydroxypropyl diethers, HEMA, 2-Propenoic acid, 2-methyl-, 3-triethoxysilyl)propyl ester, reaction products with silica and APTES, ethanol, water, CQ, a copolymer of acrylic and itaconic acid, copper (II) acetate monohydrate	1. Apply adhesive and rub for 20 s. 2. Gently air dry for approximately 5 s until the adhesive no longer moves and the solvent evaporates. 3. Light cure for 10 s.	1. Apply etchant[d] for 15 s. 2. Thoroughly rinse with water for 30 s and dry with a laboratory wipe. 3. Apply adhesive in SE application mode.
Scotchbond Universal Adhesive (SB; 3M Oral Care, St. Paul, MN, USA; 00203A)	2.7	10-MDP, Vitrebond copolymer, Bis-GMA, HEMA, filler, silane, CQ, ethanol, water		

MDP: 10-methacryloyloxydecyl dihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate; APTES: 3-Aminopropyl triethoxysilane; CQ: camphorquinone; Bis-GMA: bisphenol-A-glycidyl methacrylate.
[a] Safety data sheet
[b] Technical product profile
[c] Manufacturer's instruction
[d] K-ETCHANT Syringe (Kuraray Noritake Dental, Niigata, Japan)

then applied in different application modes (SE/ER) on the opposing sides of each disc and restored with resin composite, similar to the μTBS test specimens. Each dentin disc thus received either of the six interventions (SP-SE/SP-ER, SP-SE/SB-SE, SP-SE/SB-ER, SP-ER/SB-SE, SP-ER/SB-ER, and SB-SE/SB-ER). Six discs per intervention were used (n = 6). Fig. 1 shows the test specimen assembly for the double-sided μTBS test. After storing them in distilled water at 37 °C for 24 h, the specimens were cut into approximately one mm² of non-trimmed resin-dentin sticks. Then, they were subjected to the μTBS test, and the failure patterns were determined similarly to the μTBS test described in the previous section. The percentage of adhesive failures/group/intervention was employed for survival analysis.

Micromorphology of resin-dentin interface

Three peripheral resin-dentine slices per group were obtained from bonded teeth before cutting into sticks for the μTBS test and embedded in epoxy resin. First, the medial surfaces of the slices were wet-polished with a series of ascending grits of SiC papers (#800-, #1000- and #1200-grit; Sankyo-Rikagaku, Saitama, Japan) and descending grits of diamond pastes (6-, 3-, 1- and, 0.5-μm; DP-pastes, Struers, Ballerup, Denmark). Two mins of ultrasonic cleaning was done after each polishing step. Next, the polished specimen interfaces were treated with 5 wt% HCl for 10 s and deproteinized with 5.25 wt% aqueous solution of NaOCl for 5 mins. Water irrigation was done after both treatments. The specimens were dried overnight under ambient conditions, ion-sputtered with Pt−Pd, and observed under a scanning electron microscope (S4800 FE-SEM, Hitachi, Tokyo, Japan) at 2000× magnification.

Oxygen inhibition layer thickness of the adhesives

OIL thickness was obtained from three specimens per adhesive following a procedure described by Endo et al.[16] The adhesive droplet was placed on a bare glass slide, delimiting its two lateral peripheries with 0.17 mm thick cover glass, leaving the other two peripheries free. The specimen top was then covered with another cover glass and light-cured for 10 s. Five minutes after curing, the OIL depth was measured from the free peripheral sides of the specimen under an optical microscope (Olympus BX50, Olympus, Tokyo, Japan) at 400× magnification.

Viscosity of the adhesives

100 μL adhesive was injected into a microfluidic viscometer (microVISC, Rheosense Inc., San Ramon, CA, USA) using a pipette and was tested under standardized conditions (470 rpm speed, 1 s⁻¹ shear rate, and 15 s runtime). The thermostatic chamber of the viscometer maintained a temperature of 25 °C during the measurement. The measurement of each adhesive was repeated five times.

Determination of hardness and elastic modulus of the adhesive layer

The hardness and elastic modulus of the adhesives were tested following a standard protocol described previously.[17] The specimens were tested in a nanoindentation device (ENT-1100a; Elionix, Tokyo, Japan) using a three-sided pyramidal diamond Berkovich indenter. The indentations were done at 28 ± 0.2 °C; RH = 40 ± 5 %. Nine locations (spaced at least 20 μm apart) of the adhesive layer per specimen were indented at a constant speed of 0.1 mN/s up to a maximum load of 2 mN with a 5 s holding time at maximum load. The Poisson's ratio was 0.30. The adhesive layers' hardness and elastic modulus values were retrieved from the ENT-1100a device's built-in software.

Determination of degree of conversion of the adhesives

A modular confocal Raman spectrometer (NRS-5100, Jasco Inc, Easton, MD, USA) with a diode laser (785 nm) kept at
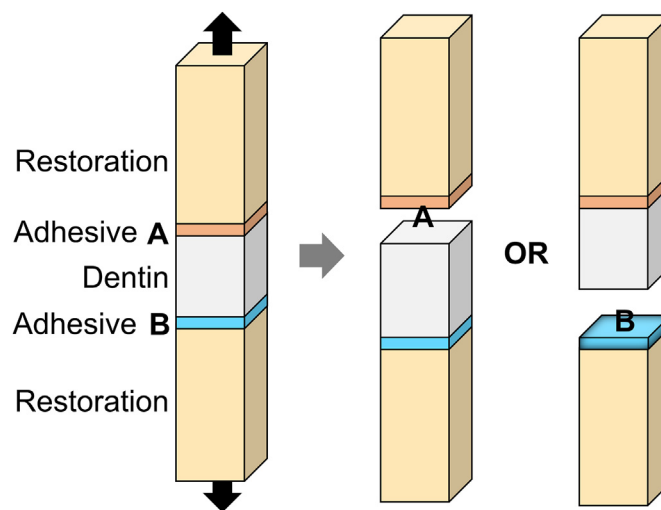

Figure 1 Diagrammatic representation of a double-sided bonded dentin specimen used to determine the survival probability of adhesive systems A and B under tensile stress (adhesive A/B intervention).

500 mW was used to obtain the spectra immediately before and after light-curing of the adhesives. Spectra were acquired between 1000 and 1800 cm^{-1} using an exposure time of 5 s and ten accumulations with an average spectral resolution of 1.6 cm^{-1}. The adhesives were light polymerized for 10 s using an LED curing unit (1200 mW/cm^2; Bluephase 20i, Ivoclar-Vivadent, Schaan, Liechtenstein). A spectral region between 1590 and 1660 cm^{-1} was selected for spectrometric analyses. The DC values were determined by the change of peak area ratio of the absorbance aliphatic C=C at 1638 cm^{-1} and the internal reference peak of aromatic C=C at 1608 cm^{-1}.[18] After baseline correction, the DC values of the two adhesives were calculated from the peak area using the formula —

$$DC\ (\%) = 1 - \frac{(Cured\ C{=}C\ 1638\ cm^{-1}/\ Cured\ C{=}C\ 1608\ cm^{-1})}{(Uncured\ C{=}C\ 1638\ cm^{-1}/\ Uncured\ C{=}C\ 1608\ cm^{-1})} \times 100.$$

Determination of in-situ degree of conversion of the adhesives

The in-situ DC of the adhesives was measured following a procedure described previously, using a NIR spectrometer attached to a NIST-developed tensometer (NIST SRI 6005b, Gaithersburg, MD, USA), which allows monitoring the real-time C=C conversion in transmission.[19] 1 mL solvent-freed adhesive was poured into a clear tube. The tube was mounted to a tensometer to facilitate the curing and spectroscopy procedure. As the curing light was transmitted through the specimen (10 s), the simultaneous measurement of the C=C (6165 cm^{-1}) conversion was done by guiding the NIR signal via 1 mm diameter optical fiber cables. By using the peak spectral area value of the sample before starting the curing (Area$_{monomer}$) and at each time point during the polymerization (Area$_{polymer}$), the real-time fractional DC was determined with the following equation: $DC\ (\%) = \frac{1 - Area_{polymer}}{Area_{monomer}} \times 100$. The DC values were continuously recorded for 600 s.

Statistical analyses

SPSS 25.0 (IBM, Chicago, IL, USA) was used to analyze the data at a 5 % significance level ($\alpha = 0.05$). Individual teeth served as the statistical units for evaluating the bonded specimens. Different groups' normal and non-homogeneous µTBS data were analyzed by Welch's t-test with Tukey's HSD test. A chi-square (χ^2) test was done to analyze the failure patterns. Survival probability based on the occurrence of adhesive failure rates among groups was estimated with the Kaplan-Meier estimator and Mantel-Cox log-rank test with Bonferroni adjustment. Non-normal and non-homogeneous hardness, elastic modulus, and in-situ DC data of the adhesives were analyzed using the Mann-Whitney U test. The normally distributed homogeneous viscosity and micro-Raman DC data were tested by independent samples t-test.

Results

Microtensile bond strength and failure patterns

The result is graphically represented in Fig. 2A. No pretest failures were observed in the study. A two-way ANOVA showed significant effects of interaction between adhesives and application modes on the µTBS (F = 12.498, $P < 0.001$). However, the µTBS of SP and SB did not differ statistically ($P > 0.05$). The application mode did not affect the µTBS of SP ($P > 0.05$). SB applied in ER mode exhibited the highest mean among all the groups but was statistically higher only when compared to its SE mode ($P > 0.05$).

This study did not observe any composite failure. Furthermore, no association was found between the four groups of adhesives and their failure patterns ($\chi^2(3) = 1.892$, $P > 0.05$). Fig. 2B shows that the failure patterns for both the adhesives in either application mode are mostly non-adhesive failures.

Survival probability of the adhesive groups

There were no pretest failures. The survival probability based on the fracture incidences in the double-sided µTBS test is shown in Fig. 3. All the curves crossed each other in the same pattern, indicating a similar survival tendency for all the groups. The log-rank test adjusted with Bonferroni correction (Fig. 3 inset) confirmed that all the groups had statistically equal survival rates regardless of adhesives and application modes ($P > 0.008$).

Bonding interface under scanning electron microscope

The representative SEM images of the test groups are shown in Fig. 4. All the groups showed intact adhesive layers continuous with resin composite and hybrid layers on both sides and devoid of bubbles.

A homologous resin tag distribution pattern was observed for the same application mode regardless of adhesives. In general, the adhesive layer of SB was noticeably thicker than that of SP. However, ER mode adhesives demonstrated thinner adhesive layers and abundant longer tags with lateral branching than those in SE mode. SP applied in ER mode showed more lateral tags than its SB counterpart.

Oxygen inhibition layer thickness of the adhesives

Representative microscopic images of the incompletely polymerized OIL adjacent to the polymerized adhesive layer are shown in Fig. 5. SB showed a thicker ($P < 0.001$) OIL (35.4 ± 4.3 µm) compared to SP (12.2 ± 1.4 µm).
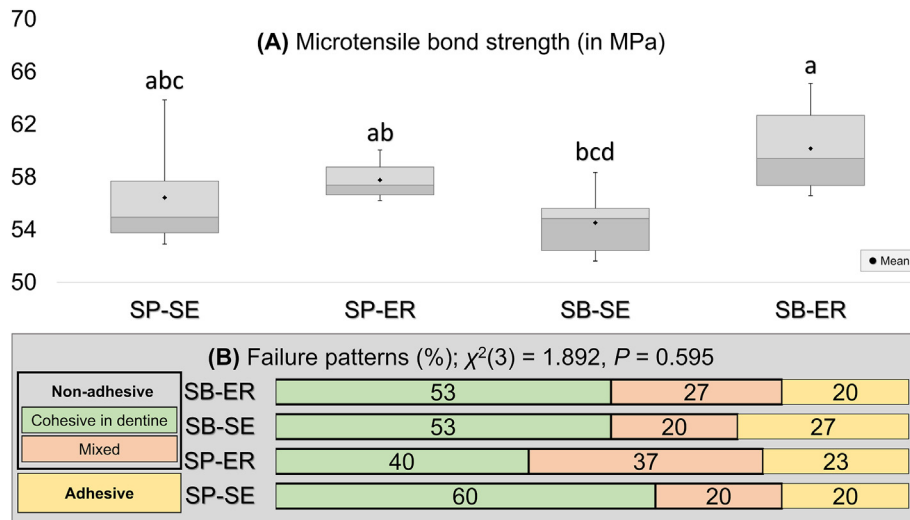
Figure 2A Box-whisker plots [minimum-(lower quartile-median-upper quartile)-maximum] of microtensile bond strength (in MPa) of the adhesives − Scotchbond Universal Plus Adhesive (SP) and its predecessor Scotchbond Universal Adhesive (SB) applied in self-etch (SE) and etch-and-rinse (ER) mode. Different lowercase letters above the plots indicate significant differences ($P < 0.05$). SB applied in ER mode showed higher bond strength than its SE counterpart ($P < 0.05$), and no significant difference ($P > 0.05$) was found when compared to its successor SP. **2B** Failure pattern analysis of the tested groups. The predominantly observed non-adhesive failures include cohesive failures in dentine or mixed failures. Cohesive failure in resin composite was not observed. Failures within the adhesive layer, at the composite-adhesive interface or the adhesive-dentine interface, were considered adhesive failures. SP and SB demonstrated a similar failure pattern ($P > 0.05$), regardless of application mode.
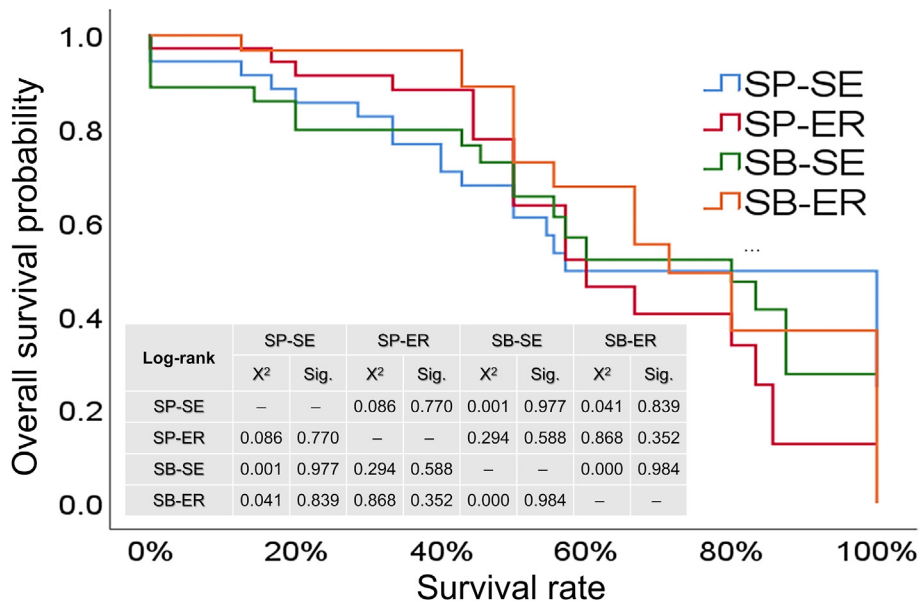

Figure 3 Survival curve and log-rank test result of the tested groups. The specimens were stressed to fracture with a double-sided microtensile bond strength test. The four different colored curves represent their survival tendency based on the rate of their adhesive failure events. The higher the curve, the more the survival tendency. The survival probability of Scotchbond Universal Plus Adhesive (SP) and Scotchbond Universal Adhesive (SB) was similar ($P > 0.008$), regardless of application mode - self-etch (SE) and etch-and-rinse (ER). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

Viscosity of the adhesives

A significant difference ($P < 0.001$) in viscosity was observed between the two adhesives. SP showed a mean viscosity of 50.2 ± 0.3 mPa S, and for SP, the mean value was 115.5 ± 0.6 mPa S, indicating a higher fluidic nature of SP compared to its predecessor, SB.
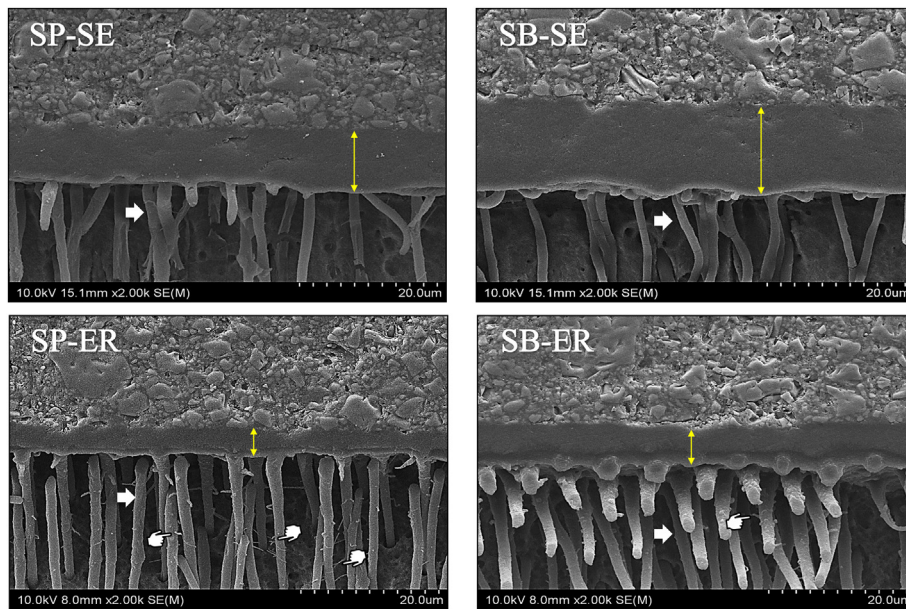
Figure 4 Representative photomicrographs (SEM) of the interfacial structures from the groups tested (2000X). Both Scotchbond Universal Plus Adhesive (SP) and Scotchbond Universal Adhesive (SB), when applied on etched dentin (ER), produced thinner adhesive layer and abundant, more extended resin micro tags with lateral tags compared to when used in self-etch (SE) mode, where SP-ER had the thinnest layer. In general, SB's adhesive layer was thicker than SP's. The thinner double-headed arrows indicate the adhesive layer thickness, the thicker white arrows indicate the resin tags, and the hand signs indicate the lateral tags.
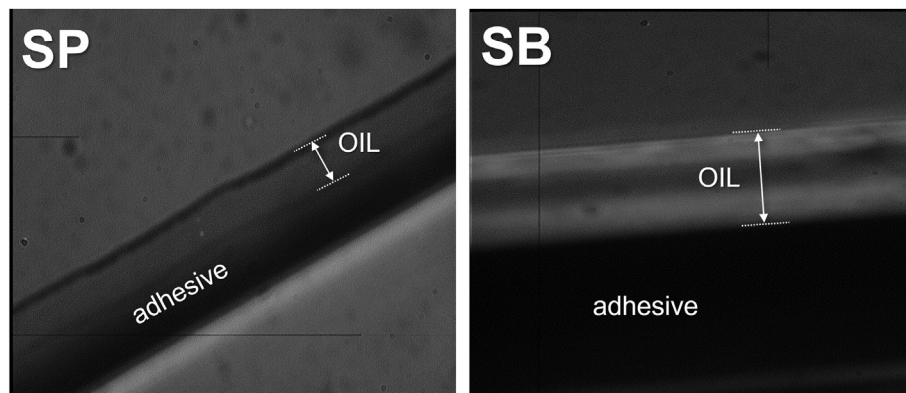

Figure 5 Microscopy images (400X) of oxygen inhibition layers (OIL) formed at the light-polymerized adhesive surfaces of Scotchbond Universal Plus Adhesive (SP) and Scotchbond Universal Adhesive (SB) exposed to the atmosphere. SP obtained a thinner OIL.

Hardness and elastic modulus of the adhesive layer

The hardness and elastic modulus results are illustrated in Fig. 6. The hardness of SP (329.6 ± 50.3 MPa) was significantly higher ($Z = -2.425$, $P = 0.015$) than that of SB (292.8 ± 31.5 MPa). A statistically higher ($Z = -3.356$, $P = 0.001$) elastic modulus was also observed in SP (7.7 ± 1.1 GPa) compared to that of SB (6.7 ± 0.7 GPa).

Degree of conversion of the adhesives

The Raman spectral regions (1590-1660 cm^{-1}) for the spectrometric measurements of the adhesives have been illustrated in Fig. 7. The Raman shift-intensity curve showed different ratios for the aromatic C=C internal reference peak at 1608 cm^{-1} and the aliphatic absorbance C=C peak at 1638 cm^{-1} before and after light polymerization for both adhesives. The t-test revealed that the DC value calculated from the spectral areal data of SP (92.6 ± 0.4 %) was significantly higher ($t_{(140)} = -5.747$, $P < 0.001$) compared to that of SB (81.4 ± 2.4 %).

In-situ degree of conversion of the adhesives

As shown in Fig. 8, the polymerization process for both adhesives started instantly with light curing. It peaked at the end of curing (at 10 s). The 'during polymerization' inset (Fig. 8a) shows that the polymerization rate of SP
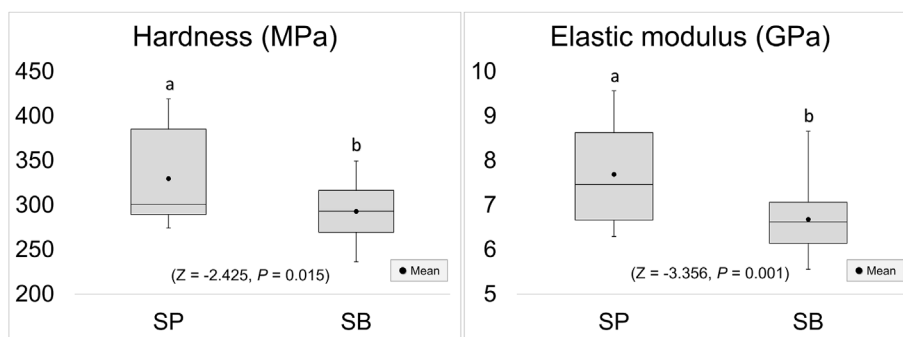
Figure 6   Box-whisker plots [minimum-(lower quartile-median-upper quartile)-maximum] of hardness and elastic modulus (mechanical properties) of the adhesive layers produced by Scotchbond Universal Plus Adhesive (SP) and Scotchbond Universal Adhesive (SB). Different lowercase letters over the plots indicate statistical variation. SP showed significantly higher mechanical properties than SB ($P < 0.05$).
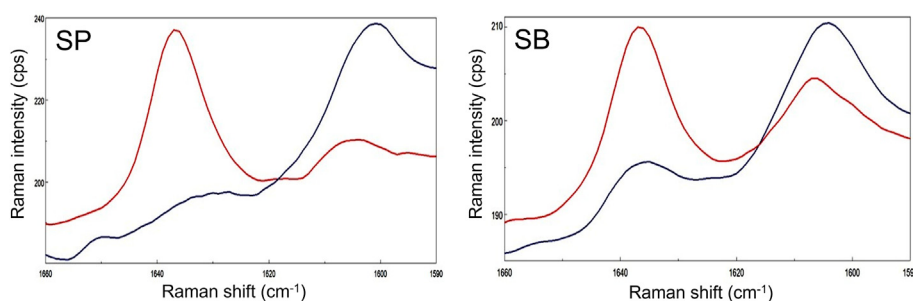

Figure 7   Raman spectra region (1590−1660 cm^{-1}) for the measurement of the degree of conversion (DC) for Scotchbond Universal Plus Adhesive (SP) and Scotchbond Universal Adhesive (SB). Raman scattering changes due to the polymerization of the adhesives; the red curve indicates the spectra immediately before light polymerization, and the blue curve represents the conversion after polymerization. The aliphatic absorbance C=C peak at 1638 cm^{-1} was noticeably higher for SP, and the DC was significantly different ($P < 0.001$) between SP and SB. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)
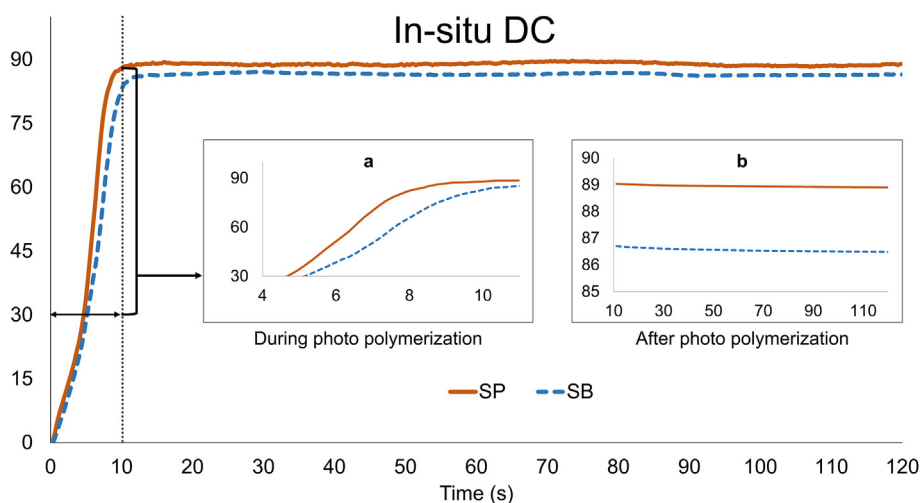

Figure 8   In-situ degree of conversion (DC) during and after 10 s light polymerization of Scotchbond Universal Plus Adhesive (SP; solid line) and Scotchbond Universal Adhesive (SB; broken line) for up to 120 s measured with near-IR spectroscopy. Inset' a' shows the corresponding initial polymerization stage, and inset' b' illustrates the post-polymerization stage of the adhesives. SP had a significantly higher DC ($P < 0.001$) than SB.

surpassed SB at a constant rate approximately 4 s after light exposure. The 'post-polymerization' inset (Fig. 8b) focused on the DC difference between the adhesives, showing that after 10 s, both the adhesives reached a plateau similarly. The paired comparison showed significantly higher ($P < 0.001$) in-situ DC of SP (90.1 ± 1.7 %) compared to that of SB (86.4 ± 2.1 %).

Discussion

Dental adhesives have evolved from multi-step systems to simplified universal versions, but resin composite restorations still fail over time. While caries, fractures, and esthetic concerns are the leading causes of restoration failure, operator skill and patient compliance play equally important roles.[20] Resin composite restorations are exposed to oral fluids, temperature variations, pH fluctuations, and mechanical stresses. Dental adhesives must withstand these challenges without compromising the restoration's properties.[20] Therefore, the mechanical response of dental adhesive systems is paramount for the long-term survival of resin composite restorations.[21]

In this study, both adhesive versions, SP and SB, demonstrated a statistically similar μTBS result. The presence of functional monomers 10-MDP and a copolymer of polyacrylic acid (Vitrebond copolymer) in both SP and SB's compositions and the same rubbing application steps (Table 1) might have led to their similar bonding outcomes.[2,5] The similarity in failure pattern with a predominance of non-adhesive failure, regardless of application mode (Fig. 2B), also supports the comparability of their dentin bonding efficacy. Non-adhesive failures require high stress to fracture dentin or resin composite cohesively or partially (mixed failures). Despite the proven usefulness of the μTBS test for interpreting adhesive's performance, it remains uncertain whether bond strength data from samples that had not failed due to adhesive issues should be considered.[22] Nevertheless, most current dental adhesives have improved bonding ability, causing increased non-adhesive failures.[22] Therefore, in this study, we included the assessment of the adhesives' survival tendencies in different application modes in a tug-of-war confrontation with the double-sided μTBS test (Fig. 1) following a previously reported method.[12,23] However, the present study used dentin as the substrate, focusing on the occurrence of adhesive failures, disregarding bond strength values and non-adhesive failure cases. No differences in survival tendencies were found between the two adhesive versions (Fig. 3). These findings align with the results of the μTBS test, supporting the influence of similar functional monomers and application techniques in adhesives' survival tendency.

Thus, the first null hypothesis that there would be no difference in bonding efficacies and survival probabilities between the two adhesives in different application modes was accepted.

According to the SEM observations (Fig. 4), SP obtained a thinner adhesive layer (2.9 ± 0.2 μm for ER; 6.1 ± 0.8 μm for SE) than SB (6.1 ± 0.4 μm for ER; 10.6 ± 2.7 μm for SE). Research has shown a positive relationship between adhesives' bond strength and thickness.[24,25] However, the

indifferent bond strength of the two adhesive versions observed in this study suggests that SP's composition might be the dominating sec3.1factor contributing to its bonding outcome.[26] According to the manufacturer, SP's viscosity and handling characteristics were adjusted using non-setting silica fillers and solvents similar to SB.[7] We found that the viscosity of SP was less than half of its predecessor SB. Such thinner viscosity of SP likely facilitated its infiltration into etched dentin, as indicated by the numerous lateral branches visible under SEM (Fig. 4). A low viscous adhesive can flow easily and spread evenly on a surface, which might explain SP's thin adhesive layer.[27] Nevertheless, low viscosity would be undesirable if overflown during air-drying, leaving no or excessively thin adhesive film on the prepared walls to withstand the resin composite contraction stress and maintain the bonding integrity.

The photoinitiators in light-cure resin generate propagating reactive free radicals that convert the monomers.[28] Atmospheric oxygen has a greater affinity to these free radicals than monomer molecules and can readily react to form a gel-like oxygen inhibition layer (OIL), impeding the conversion.[29] OIL in dental adhesives can extend to a certain depth, making a thin adhesive layer more prone to polymerization inhibition.[30] In terms of dimensional ratios between the adhesive layer and the OILs, the thicker SB and the thinner SP did not exhibit much of a difference (Figs. 4 and 5). According to the manufacturer's recent safety data sheet, both adhesives consist of the photoinitiator combination of camphorquinone (CQ) and accelerating tertiary amine (EDMAB) at an undisclosed ratio (< 2 wt%). The manufacturer only revealed that the newly upgraded SP's CQ concentration is higher than its predecessor to achieve a high DC.[7] According to Musanje L et al., an optimal CQ/amine concentration is critical to a material's composition because excessive amounts can compromise the material's mechanical properties.[31] Therefore, evaluating SP's mechanical properties (hardness and elastic modulus) was crucial.

As observed in this study, SP showed higher mechanical properties compared to its predecessor (Fig. 6). Despite the general trend of higher viscosity leading to enhanced mechanical properties in photopolymerizable resins, factors such as monomer composition, cross-linking density, and filler content can alter this relationship.[32,33] The observed contradiction between the lower viscosity of SP and its superior hardness and elastic modulus necessitates further investigation of its undisclosed BPA-free resin's cross-linking density and filler-polymer matrix interactions in a dedicated study. In addition to compositional variations, adhesive layer thickness has also been shown to influence the mechanical properties of universal adhesives.[24] Therefore, the discrepancies in adhesive thickness between SP and SB could have also contributed to their distinct mechanical behavior. Because higher mechanical properties of a photopolymerizable dental resin indirectly indicate its higher vinyl conversion, we evaluated the DC of the adhesives using different spectroscopic methods.[34] The micro-Raman findings confirmed the higher DC of SP compared to SB with a higher aliphatic absorbance C=C peak at 1638 cm^{-1} (Fig. 7). Combining the NIR in-situ DC measurement with micro-Raman was done to validate the results and better understand the kinetics. As illustrated in

Fig. 8, the in-situ spectroscopy also confirmed SP's higher polymerization ability after photopolymerization. The high CQ concentration might have resulted in increased mobility of the reactive molecules in SP's more resistance-free fluidic environment, leading to a higher DC than that of SB.[35] Therefore, SP's claimed modified CQ/amine ratio either has been made more optimized than before or an undisclosed synergistic copolymerizing agent might have been introduced to the formulation to accelerate the propagating reaction.[36]

Thus, the significant differences among all the tested physicomechanical properties between both adhesives made the second null hypothesis void.

Notwithstanding the comparable dentin bonding effectiveness, the two versions of the adhesive exhibit distinctions in some physicomechanical and micromorphological characteristics. Consequently, the bonding outcomes may vary depending on the specific tooth preparations and clinical scenarios. It is important to acknowledge two limitations of this current study: the absence of alternative adhesive systems and long-term water storage evaluation. In the future, assessing the long-term dentin bonding performance of the enhanced universal adhesive compared to multiple adhesive systems is imperative.

Within the limitations of this study, it can be concluded that the newly upgraded Scotchbond Universal Plus Adhesive: (1) has an immediate dentin bonding efficacy and survival probability similar to its predecessor Scotchbond Universal Adhesive, (2) is more fluidic and, therefore, precautions should be taken during air drying to avoid excessive thinning, (3) possesses higher mechanical properties and degree of vinyl conversion ability.

Declaration of competing interest

The authors have no conflicts of interest relevant to this study.

Acknowledgments

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