Comparison of Bonding Performance of Self-etching and Etch-and-Rinse Adhesives on Human Dentin Using Reliability Analysis

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Purpose: To estimate the in vitro reliability of typical self-etching and etch-and-rinse adhesives of various application protocols.

Materials and Methods: The following adhesives were applied on flat dentin surfaces of extracted human teeth (n = 223): self-etching two-step adhesives: AdheSE (AH), Clearfil SE Bond (CL), OptiBond SE (OS); one-step adhesives: Adper Prompt L-Pop (ADP), Adper Prompt (AD), and Xeno III (XE); all-in-one adhesive: iBond (IB); etch-and-rinse three-step adhesives: OptiBond FL (OF), two-step Gluma Comfort Bond (G), Excite (E) and Prime & Bond NT (PB). Composite buildups were prepared using a microhybrid composite, Opticor New. Shear bond strength was determined after 24 h of storage at 37°C in distilled water. The results were analyzed with a nested ANOVA (adhesive, type of adhesive) followed by the Fisher post-hoc tests of group homogeneity at $\alpha = 0.05$. A two-parameter Weibull distribution was used to calculate the critical shear bond strength corresponding to 5% probability of failure as a measure of system reliability.

Results: ANOVA revealed a significant decrease ($p < 0.001$) in the mean shear bond strength as follows: AH=CL=OS=G=E=OF>AD=IB=XE>PB=ADP, but no significant difference ($p > 0.48$) between the etch-and-rinse and self-etching adhesives. The corresponding characteristic bond strength of Weibull distribution ranged between 24.1 and 12.1 MPa, Weibull modulus between 8.3 and 2.1, and the critical shear bond strength varied from 16.0 to 3.0 MPa.

Conclusion: Pronounced differences in the critical shear bond strength suggest reliability variations in the adhesive systems tested, which originate from chemical composition rather than type of adhesive.

Keywords: bond strength, dentin adhesive system, reliability, Weibull analysis.

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The long-term success of composite restorations depends primarily on how reliably they bond to hard tooth tissues. While the bond formation to the enamel by means of enamel surface etching with phosphoric acid[4] and filling of interprismatic space with bonding resins[13] has proved reliable, creation of a bond to dentin is more complicated. Due to the high content of water and organic substances in the dentin and presence of a smear layer on its surface, the application procedure has to consist of three steps:[38] surface etching with acids, which is necessary for smear layer elimination or modification and subsurface dentin demineralization, priming of the demineralized surface with hydrophilic monomers, and application of hydrophobic bonding resins. The complex and time-consuming nature, susceptibility to errors, and sensitivity to a number of variable factors[27] of this procedure have led to efforts to simplify and increase the reliability of this process. With etch-and-rinse adhesives, the goal was achieved by combining priming and bonding into one step and using high-vapor-tension organic solvents to reduce sensitivity of the adhesive to the dentin moisture level, which is difficult to determine.[18] However, even these two-step etch-and-rinse adhesives did not fully eliminate the risk of collagen fiber collapse during drying of the demineralized dentin, which

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decreases bond strength.[35,36,38] An incomplete impregnation of collagen fibers[32] and the need to protect them against degrading effects in the oral cavity environment[5,14] encouraged development of self-etching (SE) systems, in which dentin demineralization and priming take place simultaneously. Theoretically, this procedure ensures that the whole demineralized dentin depth is monomer impregnated. SE adhesives are available as two-step systems, in which the SE primer and the bond are applied separately, or as one-step systems, in which a mixture of self-etching primer and bond is applied, prepared just before application. Recently, one-step all-in-one adhesives have been introduced, which contain a mixture of self-etching primer and bonding resin in one bottle. Although adhesive manufacturers claim that the bonding performance of SE types is less technique-sensitive and thus more reliable thanks to a lower number of working steps, the laboratory and clinical results obtained so far have not conclusively confirmed this claim.[8,10-12,15,25,26]

The reliability of adhesives can be evaluated clinically using American Dental Association criteria, which require that the failure rate of Class V restorations should be no more than 5% after 6 months for "provisional acceptance" and 10% after 18 months for "full acceptance".[1] However, no evaluation procedures have been defined for in vitro conditions. The most common in vitro methods for adhesive system assessment include measurements of the mean bond strength between tooth tissues and composite materials. The tests are carried out in tensile, microtensile, or shear setups in which the strength required for adhesive joint fracture is measured. Despite the fact that results of these tests depend on specimen geometry and experimental setup,[33,39,40] bonded area,[31] preparation tools,[9] operator experience,[34] and other variable factors,[20,24] and furthermore often disagree with other laboratory or clinical methods,[15] they are currently the routine procedures for evaluating adhesives. The evaluation of results is usually based on statistical comparison of the mean bond strengths often obtained with standard deviations of 30% and, in many cases, even up to 100%. Such high variance may mask differences between the adhesive systems. Moreover, when the assumption of a normal data distribution is not met, the mean value may not be suitable for characterization of adhesive performance. In these cases, a different approach can be used. After polymerization, the adhesives display properties of brittle materials, the strength of which is determined by pre-existing defects or flaws present in the specimen rather than the strength of the material. The strength of these materials depends on the probability of occurrence of a critical defect in their structure. Hence, it is more suitable to characterize such materials by means of probability of failure at a certain stress level calculated from the Weibull distribution function. Such an approach is frequently used in engineering to predict the life expectancy of technical systems,[28] but also in dentistry to evaluate the performance of ceramics,[3,29] composite materials,[7] the bond strength of orthodontic brackets to enamel,[21,23] or the bond strength to the tooth tissues,[2,6,19] as recommended by ISO/TS 11405.[17]

As shown elsewhere,[6,19] the probability of failure dependence on adhesive bond strength can be approximated with the two-parameter Weibull distribution curve.[22] The probability P_f that the specimen fails at stress σ is defined as follows:

$$P_f = 1 - \exp\left[-(\sigma/\sigma_o)^m\right]$$

where the scale parameter or the characteristic bond strength, σ_o, is the bond strength at which 63.2% of the samples fail and is thus a measure of adhesive bond strength. On the other hand, Weibull modulus m or the shape parameter reflects distribution of fracture-initiating flaws. High values of m indicate a narrow distribution of defects and more predictable failure behavior, while low m is typical for a high spread of defects and less predictable bond strength. The m parameter is thus a measure of bond strength variability. Probability of failure at a given stress can be calculated as $P_f = i/(N+1)$ where i is its rank number in the ascending order of bond strength data of N samples. The parameters of Weibull distribution can be obtained from the slope and intercept of ln [ln(1-P_f)] vs ln σ data or other mathematical methods. Using characteristic bond strength and the Weibull modulus, the bond strength at a selected probability of failure can be calculated and used to characterize the reliability of adhesive systems.

The objective of this study was to compare, using Weibull reliability analysis, the shear bond strength of typical self-etching (SE) and etch-and-rinse adhesive systems of various application protocols on human dentin, and characterize their performance 24 h after their application. The null hypothesis was that the reduction of the working step number in applying SE adhesives increases the reliability of these adhesives in comparison with multistep etch-and-rinse systems.

MATERIALS AND METHODS

The adhesives included the three-step etch-and-rinse system OptiBond FL, the two-step etch-and-rinse adhesives Gluma Comfort Bond, Excite, and Prime & Bond NT (Table 1). The recommended etching gels from the corresponding manufacturers were used with these systems. The SE adhesives were represented by two-step AdheSE, Clearfil SE Bond, Optibond Solo Plus SE, and one-step Adper Prompt L-Pop, Adper Prompt, Xeno III, and all-in-one iBond (Table 1). The adhesives were combined with a microhybrid composite material Opticor New containing a bis-GMA and TEG-DMA mixture and barium silicate glass filler with an average particle size of 0.7 µm.

Specimen Preparation

With the exception of Adper Prompt L-Pop, all tests were carried out on a series of 20 randomly selected intact human molars and premolars. With Adper Prompt L-Pop, three more specimens were used in order to compensate for specimens that debonded during their preparation. In compliance with the methodology,[17] soft tissues were re-

Table 1 Adhesive and resin composite systems and their application protocols

Adhesive systems	Composition	Batch No.	Application
Etch-and-rinse			
OptiBond FL (Kerr; Orange, CA, USA)	Etchant: Kerr Gel Etchant-phosphoric acid 37.5% Primer: HEMA, GPDM, PAMM, ethanol, water, photoinitiator Adhesive: TEG-DMA, UDMA, GPDM, HEMA, BIS-GMA, barium glass filler, photoinitiator	434362	e (15 s), r (15-30s), d, p (15 s), d (5s), b, c (30s)
Excite (Ivoclar-Vivadent; Schaan, Liechtenstein)	Etchant: Total Etch-phosphoric acid 37% Adhesive: HEMA, dimethacrylates, phosphoric acid acrylate, silicon dioxide, initiators, stabilizers, alcohol	E31825 D57223	e (10-15 s), r (5s), d, b (10 s), d (1-3 s), c (10 s)
Gluma Comfort Bond (Heraeus Kulzer; Hanau, Germany)	Etchant: Gluma Etch 20 Gel-phosphoric acid 20% Bond: HEMA, 4META, polyacid, ethanol, photoinitiators, polyacrylic acids	175091 010054	e (20 s), r, d (1-2s), 3x b (15 s), w (15 s), d, c (20 s)
Prime & Bond NT (Dentsply DeTrey; Konstanz, Germany)	Etchant: Conditioner 36-phosphoric acid 36% Bond: di- and trimethacrylate resins, functionalized amorphous silica, PENTA, photoinitiators, stabilizers, cetylamine hydrofluoride, acetone	209000319 206000202	e (15 s), r (15 s), d, b (20 s), d (5s), c (≥ 10 s)
Self-etching			
AdheSE (Ivoclar Vivadent)	Primer: phosphoric acid acrylate, bis-acrylamide, water, initiators, stabilizers Bond: dimethacrylates, HEMA, silicon dioxide, initiators, stabilizers	H19794	p (≥ 30 s), d, b, l, d, c (10 s)
Adper Prompt L-Pop / Adper Prompt (3M ESPE; Seefeld, Germany)	A: Red blister/liquid 1: methacrylated phosphoric esters, bis-GMA, initiators, stabilizers B: Yellow blister/liquid 2: water, HEMA, polyalkenoic acid, stabilizers	153454/ 162938	m (A+B), a (15 s), d, a , d, c (10 s)
Clearfil SE Bond (Kuraray; Osaka, Japan)	Primer: MDP, HEMA, hydrophilic dimethacrylate, camphorquinone, N,N-diethanol-p-toluidine, water Bond: MDP, bis-GMA, HEMA, hydrophobic dimetacrylate, camphorquinone, N,N-diethanol-p-toluidine, silanated colloidal silica	41265	p (20 s), d, b, d, c (10 s)
OptiBond Solo Plus SE (Kerr)	Self-etching primer: ethanol, water, alkyl dimethacrylate resin, stabilizers and activators Bond: ethanol, alkyldimethacrylate resin, barium aluminoborosilicate glass, fumed silica, sodium hexafluorosilicate	430870	p (15 s), d (3 s), b (15 s), d (3 s), b (15 s), d (3 s), c (20 s)
Xeno III (Dentsply DeTrey)	Liquid A: HEMA, water, ethanol, BHT, highly dispersed silicon dioxide Liquid B: Pyro-EMA, PEM-F, urethane dimethacrylate, BHT, camphorquinone, ethyl-4-dimethylaminobenzoate	0305001039	m (A+B, 5s), a (≥ 20 s), d (≥ 2s), c (≥ 10 s)
iBond (Heraeus Kulzer)	4-META, UDMA, glutaraldehyde, acetone, water, photoinitiators, stabilizers	010062	3 x a, w (30s), d, c (20 s)
Composite resin			
Opticor New (Spofa-Dental; Jicin, Czech Republic)	Bis-GMA, TEG-DMA, UDMA, barium fluoride glass, silicon dioxide, initiators, stabilizers, pigments	862616-2/A2	c (20 s)

BHT: butylated hydroxy toluene; bis-GMA: bisphenol diglycidyl methacrylate; GPDM: glycerol phosphate dimethacrylate; HEMA: hydroxyethyl methacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; 4-META: 4-methacryloxyethyl trimellitanhydride; PAMM: phtalic acid monoethyl methacrylate; PEM-F: monofluorophosphazene modified methacrylate; PENTA: dipentaerythritol penta acrylate monophosphate; pyro-EMA: phosphoric acid modified methacrylate; TEG-DMA: triethylene glycol dimethacrylate; UDMA: urethane dimethacrylate.
a-application, b-bonding, c-curing, d-drying/spreading, e-etching, m-mixing, p-priming, r-rinsing, w-waiting.

moved from the extracted teeth and the teeth were stored in 0.5 wt% chloramine T solution for up to 1 week and then in distilled water at 4°C. The teeth were used within six months after extraction. After the roots were removed, the teeth were fixed in a stainless steel ring using the self-curing acrylic resin Spofacryl (Spofa-Dental; Jicin, Czech Republic). The oral or buccal enamel layer was removed by means of a low-speed saw (Isomet Buehler; Lake Bluff, IL, USA) equipped with a diamond wafering blade under water cooling, and the dentin surface was polished with a wet SiC paper P 1200 (Buehler). Subsequently, an experienced operator applied adhesives to the dentin surface following manufacturers' instructions. The Adper Prompt and Xeno III adhesives were mixed for each specimen separately; a new blister was used for each specimen with Adper Prompt L-Pop. It was necessary to apply the latter adhesive two or three times in order to obtain the recommended glossy dentin surface in some specimens. Composite buildups were made on the dentin surface using a circular transparent polyethylene mold with an inner diameter of 3.5 mm and height of 2 mm. The mold was filled with one layer of composite material and polymerized for 20 s. An Elipar TriLight (3M ESPE) halogen lamp with a light output intensity of 850 mW/cm^2 was used for polymerization of the composite and adhesive materials. Its light output was periodically checked using a calibrated radiometer.

Shear Bond Measurement

Shear bond strength (SBS) was determined after 24-h storage of specimens in water at 37°C using a universal testing machine (Shimadzu AGS-G, Shimadzu; Kyoto, Japan) at the strain rate of 0.75 mm/min. Stainless steel rings were fixed in a Bencor Multi T device (Danville Engineering; Danville, CA, USA) equipped with a flat shearing blade positioned within 0.5 mm from the adhesive interface. SBS was calculated from the force at specimen failure, divided by the bonded area. A fractographic surface analysis was performed using a Nikon SMZ 2T stereomicroscope (Nikon; Tokyo, Japan) with 20X to 30X magnification. The failure mode was classified according to the prevailing failure type – it was adhesive where the fracture path was located between the composite and the dentin, and mixed where the fracture path included both the interface region and the dentin or the composite.

Statistical Analysis

As Weibull analysis automatically excludes zero values, the specimens that failed during specimen preparation were replaced and their number was registered. A nested ANOVA and Fisher Least Significant Difference (LSD) post-hoc tests of group homogeneity were performed at $\alpha = 0.05$ to test bond strength differences between individual adhesive systems and differences between SE and etch-and-rinse systems. The Weibull distribution parameters were calculated using the maximum likelihood estimation method at 95% confidence level. All the calculations including the critical shear bond strength at 5% probability of failure, $\sigma_{0.05}$, were performed using STATISTICA 7.1 (StatSoft; Tulsa, OK, USA).

RESULTS

A nested ANOVA and post-hoc tests revealed significant differences ($p < 0.001$) in the mean SBS of individual adhesive systems (Table 2). On the other hand, there was no significant difference ($p > 0.48$) between groups of etch-and-rinse and SE adhesives. As seen from Table 2, the highest SBS was found for a group of adhesives including etch-and-rinse three-step OptiBond FL, two-step Excite, Gluma Comfort Bond and SE 2-step AdheSE, Clearfil SE Bond, OptiBond Solo Plus SE systems. A high percentage of mixed fractures including dentin corresponded to the high bond strength values. The one-step SE adhesives Adper Prompt, Xeno III and iBond (Table 2) displayed significantly lower SBS. While with Adper Prompt some specimens exhibited mixed fractures, only adhesive fractures were observed in the rest of the group. The lowest SBS and exclusively adhesive fractures were found for two-step etch-and-rinse Prime & Bond NT and one-step SE Adper Prompt L-Pop, in which some pre-testing failures occurred (Table 2).

The parameters of Weibull distributions, their 95% confidence intervals, and correlation coefficients are summarized in Table 3. The fit of experimental data with Weibull distribution (Figs 1 to 3) was acceptable as indicated by a high correlation coefficient $r > 0.920$. The Weibull distribution parameters varied for different adhesive systems. The characteristic bond strength values ranged from 24.1 to 12.1 MPa and Weibull modulus from 8.3 to 2.1 (Table 3). Pronounced differences were also found for $\sigma_{0.05}$, which ranged from 16.0 to 3.0 MPa.

DISCUSSION

The focus of the study was an in vitro evaluation of some representatives of diverse adhesive systems with different application protocols. Similar to other studies,[11,25,30] statistical processing of results based on the mean bond strengths showed significant differences in the bond performance of the adhesive systems tested, but no significant difference between etch-and-rinse and SE adhesives with a simplified application protocol. From the clinical point of view, the mean bond strength values should be less relevant than the likelihood that a high adhesion will be achieved with a given system or, conversely, that its bond strength will be low and the risk of an adhesive failure high. This probabilistic approach to adhesive evaluation is made possible by using the Weibull distribution to calculate the critical shear bond strength, $\sigma_{0.05}$, corresponding to 5% of failed specimens. The highest $\sigma_{0.05}$ values were in the range of 14.8 to 16.0 MPa in the group of two-step etch-and-rinse Gluma Comfort Bond and SE two-step AdheSE, Clearfil SE Bond and OptiBond Solo Plus SE adhesive systems (Table 3). These high values resulted not only from the high characteristic bond strengths but also from high Weibull modulus values, ranging from 8.3 to 6.5. These values of the Weibull parameter indicated a steep slope of probability of failure on failure stress dependence (Fig 1), and thus a lower scatter of bond strength.

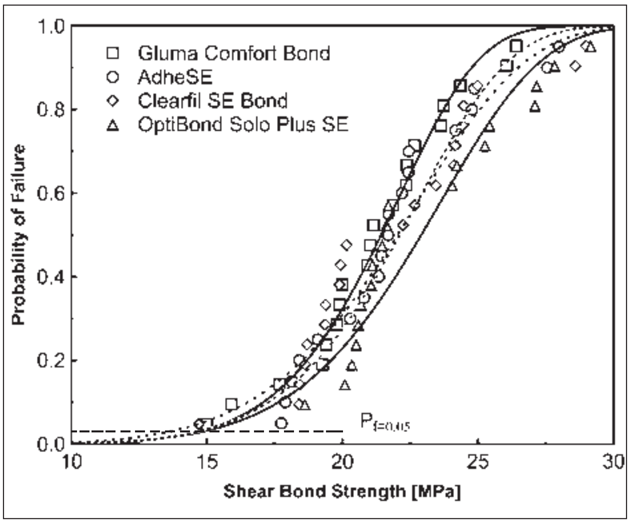

Fig 1 Probability of failure vs the shear bond strength for AdheSE, Clearfil SE Bond, Gluma Comfort Bond and OptiBond Solo Plus SE. The dotted line is drawn at 5% probability of failure.

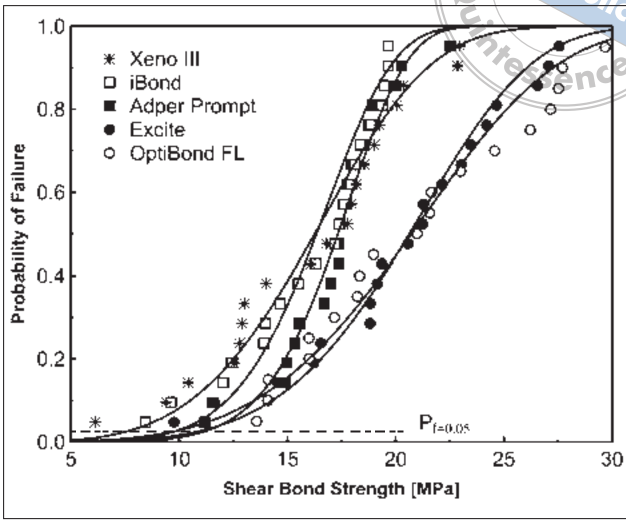

Fig 2 Probability of failure vs the shear bond strength for Adper Prompt, Gluma Comfort Bond, iBond, Xeno III and OptiBond FL. The dotted line is drawn at 5% probability of failure.

Table 2 Descriptive statistics of the adhesive shear bond strength and specimen failure mode ranked from the highest to the lowest bond strength

Adhesive	Mean ± SD (MPa)	Interval (MPa)	Failure mode Mixed/ Adhesive (%)
Clearfil SE Bond	22.8[a] ± 3.6	14.7-29.0	75/25
OptiBond Solo Plus SE	22.6[a] ± 3.5	15.0-29.1	65/35
AdheSE	21.8[a] ± 3.0	17.7-28.0	35/65
Gluma CB	21.2[a] ± 3.0	15.0-26.4	65/35
OptiBond FL	20.5[a] ± 5.4	12.5-29.7	35/65
Excite	20.2[a] ± 4.9	9.8-27.6	45/55
Adper Prompt	17.1[b] ± 2.7	11.2-22.5	40/60
Xeno III	16.1[b] ± 4.5	6.1-23.0	10/90
iBond	16.0[b] ± 3.4	8.4-19.7	0/100
Prime&Bond NT	12.0[c] ± 3.1	5.7-18.8	0/100
Adper Prompt L-Pop	11.1[c] ± 5.6	2.3-20.6	0/100

Identical superscript letters indicate no significant difference ($p > 0.05$)

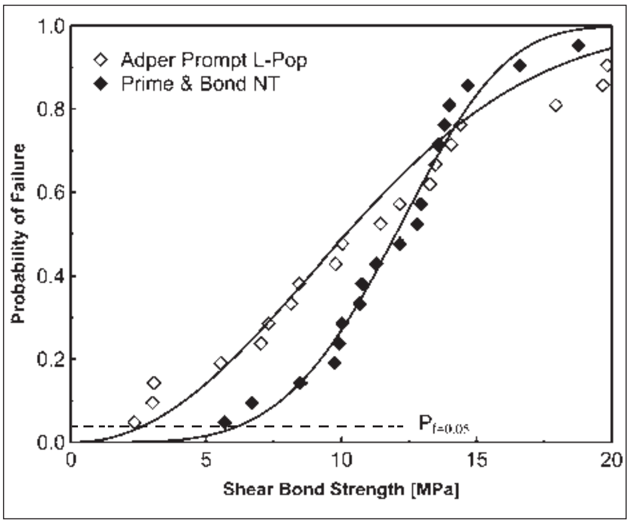

Fig 3 Probability of failure vs the shear bond strength for Adper Prompt L-Pop and Prime & bond NT. The dotted line is drawn at 5% probability of failure.

With high probability, it can be assumed that these systems create a reliable bond to the tooth tissues.

Lower $\sigma_{0.05}$ values, ranging from 10.9 to 12.4 MPa, were found for SE one-step Adper Prompt and iBond, and two-step Excite and three-step OptiBond FL systems from the etch-and-rinse adhesive group. With Excite and Optibond FL adhesives, whose characteristic bond strength values of 22.2 and 22.5 MPa, respectively, were comparable with the preceding systems, the lower critical bond strengths were caused mainly due to lower Weibull modulus values, 5.1 and 4.4, respectively (Table 3, Fig 2). In contrast, with Adper Prompt and iBond, the characteristic bond strengths were lower, 18.2 and 17.3 MPa, respec-

Table 3 Critical bond strength, $\sigma_{0.05}$, corresponding to 5% probability of failure, the characteristic bond strength σ_o and Weibull modulus m. The adhesives are ranked from the highest to the lowest critical bond strength

Adhesives	$\sigma_{0.05}$ [MPa]	σ_o [MPa]	CI[1)] [MPa]	m	CI[1)]	r[2)]
OptiBond Solo Plus SE	16.0	24.1	22.6–25.7	7.2	5.2–10.1	0.960
Gluma CB	15.7	22.4	21.2–23.7	8.3	5.9–11.6	0.989
AdheSE	15.7	24.1	21.7–24.7	7.6	5.5–10.6	0.947
Clearfil SE Bond	14.8	23.3	21.7–25.0	6.5	4.7–9.1	0.920
Excite	12.4	22.2	20.3–24.2	5.1	3.6–7.3	0.960
Adper Prompt	12.2	18.2	17.1–19.3	7.5	5.4–10.4	0.974
OptiBond FL	11.4	22.5	20.2–25.0	4.4	3.1–6.2	0.990
iBond	10.9	17.3	16.1–18.6	6.4	4.4–9.4	0.977
Xeno III	8.9	17.7	15.9–19.7	4.3	3.0–6.2	0.984
Prime & Bond NT	6.6	13.1	11.8–14.6	4.3	3.1–6.1	0.988
Adper Prompt L-Pop	3.0	12.1	10.7–14.9	2.1	1.5–2.9	0.985

[1)] CI – confidence interval ± 95%, [2)] r – correlation coefficient.

tively, but the Weibull modulus values were higher, 7.5 and 6.4, respectively. This indicated that the slightly lower bonding reliability of Excite and Optibond FL could be caused by a higher scatter of bond strength values and thus their increased susceptibility to defect introduction. Since the bond strength evaluation took place 24 h after application, the defects may be caused, for example, by imperfect dentin demineralization, incomplete impregnation of collagen fibers and subsurface dentin,[32] incomplete polymerization of adhesive as a result of the presence of organic solvent residues in the adhesive layer,[16,18,41] or monomer-solvent phase separation in the adhesive layer.[36,37] However, the lower characteristic bond strength values of Adper Prompt and iBond indicated lower bond strength rather than their sensitivity to defect introduction. Unlike these systems, one-step SE Xeno III showed a lower level of both the characteristic bond strength, 17.7 MPa, and Weibull modulus, 4.3. This system also showed a lower critical bond strength, 8.9 MPa, as a result of deteriorated adhesion and increased bond strength variability. The last group included Prime & Bond NT and Adper Prompt L-Pop, whose bond strength and adhesive mode of fracture confirmed decreased adhesion. ANOVA analysis and comparison of characteristic bond strength values, 13.1 and 12.1, respectively, did not reveal any significant differences between these systems. On the other hand, the Weibull modulus values were significantly different between these systems. While Prime & Bond NT showed, like many other systems, an acceptable value of 4.3, the Adper Prompt L-Pop value of 2.1 indicated a considerable scatter of bond strength and thus a low predictable behavior (Fig 3). The probability that a reliable bond to dentin can be created with this adhesive is significantly lower due to decreased adhesion to dentin and susceptibility to defects, as manifested also by the presence of pre-test failures.

For clinical purposes, such adhesive systems are suitable that provide with high probability a bond strength higher than the stress acting in the adhesive layer as a result of polymerization contraction, mastication forces, or thermal expansion variances. Although such minimal bond strength between dentin and composite restoration material has not been defined clearly, from comparison of these results with the clinically successful adhesive systems Optibond FL or Clearfil SE Bond,[8,26] it can be deduced that the critical bond strength should not fall under approximately 11 to 14 MPa under the given measurement conditions. This value is in compliance with the shear bond strength requirements of 10 to 12 MPa, estimated on the basis of clinical and laboratory comparison.[15]

Within the limitations of this study, the bond strengths obtained 24 h after application confirmed differences in reliability of the adhesive systems tested. These differences follow from the specific chemical composition of each adhesive system. The null hypothesis that SE systems with a simplified application protocol are generally more reliable than etch-and-rinse adhesives must therefore be rejected.

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Clinical relevance: There are pronounced differences in the reliability of contemporary adhesives. To a great extent, these differences depend on the specific compositions of adhesives, and it cannot be assumed that SE systems with a simplified application protocol are generally more reliable than etch-and-rinse adhesives.