Journal of Adhesion Science and Technology

ISSN: 0169-4243 (Print) 1568-5616 (Online) Journal homepage: http://www.tandfonline.com/loi/tast20

Adhesion of dual-polymerized luting cement on superficial and deep dentin using different one-step self-etch mild adhesive systems

Larissa Blarer & Mutlu Özcan

To cite this article: Larissa Blarer & Mutlu Özcan (2018): Adhesion of dual-polymerized luting cement on superficial and deep dentin using different one-step self-etch mild adhesive systems, Journal of Adhesion Science and Technology

To link to this article: https://doi.org/10.1080/01694243.2018.1494399

JOURNAL OF ADHESION SCIENCE AND TECHNOLOGY
https://doi.org/10.1080/01694243.2018.1494399

Adhesion of dual-polymerized luting cement on superficial and deep dentin using different one-step self-etch mild adhesive systems

Larissa Blarer[a] and Mutlu Özcan[b]

[a]University of Zurich, Center for Dental and Oral Medicine, Clinic for Fixed and Removable Prosthodontics and Dental Materials Science, Zurich, Switzerland; [b]University of Zurich, Dental Materials Unit, Center for Dental and Oral Medicine, Clinic for Fixed and Removable Prosthodontics and Dental Materials Science, Zurich, Switzerland

ABSTRACT

This study evaluated adhesion of dual-polymerized resin cement to superficial dentin (SD) and deep dentin (DD) using one-step self-etch adhesives at varying pH. After smear layer was created on third molars ($N = 60$, $n = 15$ per group), adhesive resins, 1- Clearfil S3 Bond Plus-CBP (Kuraray) (pH: 2.3), 2- Bisco All Bond Universal-BAU (Bisco) (pH: 3.2), 3- Single Bond Universal Adhesive-SBU (3M ESPE) (pH: 2.7), 4- Nova Compo-B Plus-NCBP (Imicryl) (pH: 2.5–3), were applied on SD and DD. Resin cement (Variolink II, Ivoclar Vivadent) was adhered incrementally on the SD surfaces using polyethylene molds and photo-polymerized for 40 s from 5 directions (output: 1200 mw/cm^2). After macroshear and microshear test, in order to achieve DD specimens, SD were removed 1 mm in the pulp direction and the same bonding and test procedures were performed. The specimens were kept at 37 °C for 24 h. The adhesion tests were conducted in the Universal Testing Machine and failure types were analyzed. The data were analyzed using Univariate ANOVA, Tukey's, Kruskal-Wallis and Mann-Whitney tests ($\alpha = .05$). Test method, dentin level and the adhesive resin significantly affected the results (MPa) ($p < .05$). After macroshear test, more incidences of cohesive failures in DD were observed with NCBP Plus. On SD, NCBP presented the highest results followed by BAU using macroshear test. On DD, NCBP presented the highest results followed by SBU. Not only the pH but the chemical composition affected adhesion especially to SD while in DD, the difference between the adhesive resins was less significant.

ARTICLE HISTORY
Received 15 March 2018
Accepted 19 June 2018

KEYWORDS
Adhesion; adhesive cementation;
bond strength test;
dentin; macroshear test;
microshear test;
resin luting cement

Introduction

The longevity of indirect fixed-dental-prostheses (FDP) could be affected by multiple factors including the cementation mode that is basically the final stage of consecutive clinical procedures. In principle, the primary function of the cementation is to establish reliable retention of the FDP, a durable seal of the space between the tooth and

CONTACT Mutlu Özcan mutluozcan@hotmail.com Plattenstrasse 11, CH-8032, Zürich, Switzerland

the restoration and to provide adequate optical properties especially for ceramic or polymeric FDPs [1]. Today, adhesive properties of dental cements are of importance as it enables clinicians to perform minimal invasive restorations [2].

Current trends in dental research tend to simplify the clinical procedures, especially for the applications of adhesive resins not only in restorative dentistry for direct applications of resin-based materials but also in prosthetic dentistry in adhesive cementation of FDPs. Advances in these procedures primarily include the development of simplified bonding systems as a substitute for classical three-step systems [1]. On the other hand, traditional "etch and rinse" adhesive systems rely on the application of adhesive monomers to acid-etched dentin or the use of simplified self-etching, self-priming agents that contain hydrophilic and acidic monomers, acidic molecules, diluent monomers, photoinitiators and solvents usually at low pH. Such adhesives simultaneously etch the dentin and infiltrate the adhesive monomers into the dentin [3].

Different bonding strategies may affect the adhesion of resin cements to dentin [4–6]. In fact, long-term survival of FDPs may be very much dependent on the function of the resin cement that adheres both to the restorative materials and the tooth substance [7]. The reliable adhesion of the resin cements to both the dentin and the ceramic becomes particularly important in the application of all-ceramics or surface-retained resin-bonded FDPs where mechanical retention does not dominate [1]. Also, when enamel margins do not exist after tooth preparation as a consequence of caries removal or tissue loss due to trauma, adhesion of the cement is dictated by dentin conditioning through etching and application of the bonding agents. While some previous studies have shown that self-etch adhesives may result in lower bond strength to dentin [7–9], others reported no significant difference with the use of mild universal adhesives with etch-and rinse strategy [10]. In addition, due to the acidic nature of the self-etch adhesives and their permeability, their adhesion to dentin are compromised [9]. Typically, since etching dentin separately would result in higher interaction with dentin [11] and due to their lesser hydrophilicity [12], "etch and rinse" adhesive systems compensate for other factors and lead to higher bond strength of the resin cements to dentin. However, this adds to the increased workflow and achieving balance between dry but at the sometime sufficient humid dentin surface is considered as a clinical challenge when "etch and rinse" adhesive systems are employed [13].

Resin cements, be it conventional or simplified, may perform differently depending on their adhesive systems since the latter is primarily in contact with the dentin [14,15]. Contemporary adhesive systems used in dentistry interact with the enamel/dentin substrate either by removing the smear layer (etch-and-rinse technique) or by partially dissolving the smear layer, decalcifying underlying intertubular dentin, and impregnating any remaining smear layer for the bonding (self-etch technique) [16]. While the etch-and-rinse bonding technique is initiated by a separate etching step using 35–37% phosphoric acid that is later rinsed away, the self-etch/primer agent containing acidic monomers are only air-dried, thus remaining within the modified smear layer. The self-etch approach could also be called as "etch-and-dry" approach [17]. Such adhesives make the application less technique-sensitive for the clinicians [18]. Besides micromechanical interlocking through hybridization, specific functional monomers of 'mild' or 'intermediate' two-step self-etching adhesives were shown to interact chemically with residual hydroxyapatite crystals that remain available in the submicron hybrid layer [19]. While some studies reported higher bond strengths to dentin with two-step self-etching adhesives compared to one-step ones [20], others reported comparable [14] or lower bonding efficacy to dentin [21–23].

Resin-bonded FDPs are usually bonded to enamel or superficial dentin (SD). However, full-coverage FDPs, extensive overlays or endocrowns are cemented to deep dentin (DD) as such restorations require more room for the restorative material. In previous studies, bond strengths of resin composites to dental tissues were found to be higher in SD than in DD [24,25]. Since SD contains more intertubular dentin area and it is rich in collagen fibrils, it makes adhesion with resin-based materials favourable [11]. However, while impaired adhesion was reported on DD due to higher water content compared to SD [24,25], other studies showed no significant difference on both dentin substrates as a function of depth [26]. The controversial results could be in part due to the difference in test methods or the pH effect of the adhesives on dentin [27].

Previous studies using stress distribution analyses have reported that some of the bond strength tests do not appropriately stress the interfacial zone [28,29]. Shear tests have been criticized for the development of non-homogeneous stress distributions at the bonded interface, inducing either underestimation or misinterpretation of the results, as the failure often starts in one of the substrates and not solely at the adhesive zone [28,29]. Conventional tensile tests also present some limitations, such as the difficulty of specimen alignment and the tendency for heterogeneous stress distribution at the adhesive interface. On the other hand, when specimens are aligned correctly, the microtensile test shows more homogeneous distribution of stress, and thereby more sensitive comparison or evaluation of bond performances [22]. However, small deviations in specimen alignment in the jig may cause increase bond strength due to shear component being introduced during deboning bonded joints [30,31]. To the best of our knowledge, no study exists to date where both macro and microshear adhesion tests were employed in one study on both SD and DD.

The objectives of this study therefore were to evaluate the adhesion of conventional resin-based luting cement together with self-etch adhesive resin systems to SD and DD using both macroshear and microshear tests. The hypotheses tested were that a) adhesive strength of resin cement to DD would be less than to SD, b) macroshear bond strength results would be less than microshear tests and c) self-etch adhesive resins with lower pH would deliver higher bond strength results than those of the adhesives with higher pH.

Materials and Methods

Specimen preparation

The brands, types, main chemical compositions, manufacturers and batch numbers of the materials used for the experiments are listed in Table 1. This study comprised four self-etch adhesive resins and one conventional resin-based adhesive cement. Adhesive potential of the cement on SD and DD of third molar teeth was tested.

Human wisdom molars ($N = 60$, $n = 15$ per group), were collected and kept in distilled water at 5 °C until the experiments. All teeth used in the present study were extracted for reasons unrelated to this project. Written informed consent for research

Table 1. The brands, abbreviations, manufacturers, chemical compositions and batch numbers of the materials used for the experiments.

Brand	Manufacturer	Chemical composition	*pH	Batch numbers
Clearfil S3 Bond Plus (CBP)	Kuraray, Kurashiki, Japan	bis-GMA, 2-HEMA, ethanol, sodiumfluoride, 10-methacryloyloxydecyl dihydrogenphosphate, hydrophilicaliphaticdimethacrylate, hydrophobicaliphaticmethacrylate, colloidalsilica, dl-Campherquinone, accelerators, initiators, water	2.3	6E0012
Bisco All Bond Universal (BAU)	Bisco, Schaumburg, USA	Ethanol, bis-GMA	3.2	1300009232
Single Bond Universal (SBU)	3M ESPE AG, Seelfeld, Germany	MDP phosphate monomer, dimethacrylate resins, HEMA, Vitrobond copolymer, filler, ethanol, water, initiators, silane	2.7	497905
Nova Compo-B Plus (NCBP)	Imicryl Dis Malz. GmBH, Konya, Turkey	bis-GMA, 2-HEMA, ethanol,10-methacryloyloxydecyl dihydrogenphosphate, 4-META, silanated high dispersed silica, dl-Campherquinone, accelerators, initiators, water	2.5–3	16224
Variolink II (Dual polymerized resin cement)	Ivoclar Vivadent, Schaan, Liechtenstein	UDMA, inorganic fillers, ytterbium trifluoride, initiators, stabilizers, pigments		S50635T24729

bis-GMA, bisphenol A glycol dimethacrylate; UDMA, Urethane dimethacrylate; HEMA, Hydroxyethyl methacrylate; MDP, 10- Methacryloyloxydecyl dihydrogen phosphate; 4-META, 4-methacryloxyethyl trimellitate anhydride. *pH values of the adhesives are obtained from the manufacturers.

purpose of the extracted teeth was obtained by all donors prior to extraction according to the directives set by the National Federal Council. Ethical guidelines were strictly followed and irreversible anonymization was performed in accordance with State and Federal Law [32–34]. After tissue remnants were removed with a scaler (H6/H7; Hu-Friedy, Chicago, IL), teeth were stored in .5% Chloramin T for 2 weeks. In order to determine the exact location of cusp tips, enamel, dentin and pulp horns of the teeth, a previously describer method was used [22].

The teeth were embedded using polymethylmethacrylate (PMMA, Condular AG, Wager, Switzerland) with their occlusal surfaces exposed in polyethylene rings (diameter: 10 mm, height: 12 mm). The apices of the roots were shortened using a diamond disc (IsoMet 1000, Buehler Ltd, Illinois, USA) under water-cooling when root length exceeded 12 mm. Then, the cusps of the molars were removed in a trimmer (Isomet, Buehler Ltd., Lake Bluff, IL, USA) under water-cooling until flat dentin surfaces were achieved. Dentin level after removal of cusp tips was considered as SD group. One mm below the SD level was indicated and considered as DD [25].

Dentin surfaces were exposed with a low speed disc under water-cooling and ground finished using 600, 800 and 1000-grit, silicone carbide abrasive papers (English Abrasives Ltd, London, United Kingdom) under water in sequence. In both SD and DD groups, flat dentin surfaces were polished with 400 grit silicon carbide papers under water-cooling and then rinsed thoroughly in order to create bonding surfaces covered with smear layers [35,36]. Subsequently, bonding procedures were carried out.

Bonding procedures for adhesion tests

One calibrated operator carried out adhesive procedures throughout the experiments (Table 2). After conditioning the dentin surfaces, the adhesive luting cement

Table 2. Application modes of the adhesive resins tested in this study according to each manufacturer's instructions. See Table 1 for group abbreviations.

CBP	BAU	SBU	NCBP
Apply bond for 10 s	Dispense 1–2 drops of adhesive resin into a well	Place one drop of of the adhesive resin and dual cure activator in a mixing well and mix for 5 s	Dispense 1–2 drops of adhesive resin into a well
Dry with mild pressure air flow for 5 s	Apply two separate coats, scrubbing the preparation with a microbrush for 10–15 s per coat	Apply the adhesive mixture to the prepared tooth and rub it in for 20 s	Apply two separate coats, scrubbing the preparation with a microbrush for 10–15 s per coat
Photo-polymerize for 10 s	Photo-polymerize for 10 s	Gently air dry the adhesive resin for approximately 5 s to evaporate the solvent	Photo-polymerize for 10 s
		Photo-polymerize for 10 s	

(Variolink II, Ivoclar Vivadent, Schaan, Liechtenstein) were adhered incrementally with a hand instrument on the dentin surfaces using translucent polytetrafluoroethylene (Teflon) molds (DuPont, Saint-Gobain, France) (for macroshear bond test-SBT: height: 4 mm, diameter: 2.9 mm; for microshear bond test-μSBT: height: 4 mm, diameter: .8 mm) were stabilized on the dentin specimens in a custom made device. The mold was filled in two increments with the resin cement and polymerized for 40 s from 5 directions from a distance of 2 mm (Bluephase, Ivoclar Vivadent) for 40 s (output: 1200 mw/cm^2). Oxygen inhibiting gel (Oxyguard, Kuraray, Tokyo, Japan) was applied at the bonded margins and rinsed with copious water after 1 minute.

The bonded specimens were kept at 37 °C for 24 hours. After macro and microshear tests on the SD, the dentin surfaces were again ground finished using 600, 800 and 1000-grit, silicone carbide abrasive papers under water in sequence inorder to achive DD and conditioned with the adhesive resins accordingly. The same resin luting cement was adhered incrementally with a hand instrument on the dentin surfaces using polyethylene moulds. The teeth were maintained wet at all times during all procedures except during X-rays.

Macroshear and microshear tests

Specimens were mounted in the apparatus of the Universal Testing Machine (Zwick ROELL Z2.5 MA 18-1-3/7, Ulm, Germany) and the force was applied to the adhesive interface until failure occurred. The load was applied with a 50 kgf load cell to the substrate-adherend interface with the corresponding blades for each test method, as close as possible to the surface of the substrate at a crosshead speed of 1 mm/min and the stress-strain curve was analyzed with the software program (TestXpert®, Zwick ROELL, Ulm, Germany). Bond strength after each test (MPa) was calculated by dividing the maximum load (N) by the bonding surface area of the resin cement.

Failure type evaluation

Failure sites were initially observed using an optical microscope (Stemi 2000-C, Carl Zeiss, Gottingen, Germany) at ×40 magnification and since no mixed failures were observed, the failure types were classified as follows: Score A = adhesive failure between the resin and the dentin; Score C = cohesive failure in dentin, Score CC: cohesive failure in the resin cement.

Statistical analysis

Statistical analysis was performed using Statistica 8.0 software for Windows (StatSoft, Inc., Tulsa, OK, USA). Kolmogorov-Smirnov and Shapiro-Wilk tests were used to test normal distribution of the data. The means of each group were analyzed by Univariate analysis of variance (ANOVA), with bond strength as the dependent variable and adhesive resins (4 levels: CBP, BAU, SBU, NCBP) and dentin level (2 levels: superficial dentin-SD and deep dentin-DD) and test method (2 levels: macroshear vc microshear) (SPSS 11.0 software for Windows, SPSS Inc., Chicago, IL, USA). Multiple comparisons were made by Tukey̓s adjustment test, Kruskal-Wallis and Mann-Whitney tests. Maximum likelihood estimation without a correction factor was used for 2-parameter Weibull distribution, including the Weibull modulus, scale (m) and shape ($_0$), to interpret predictability and reliability of adhesion (Minitab Software V.16, State College, PA, USA) with different self-etch adhesive resins. P values less than .05 were considered to be statistically significant in all tests.

Results

Bond strength test method, depth of dentin and the adhesive resin material significantly affected bond strength (MPa) results ($p < .05$). Interaction terms were also significant ($p < .05$).

While using macroshear test on the SD, NCBP adhesive presented significantly higher bond strength than those of others ($p < .05$), using microshear test no significant difference was found beween all adhesives ($p > .05$) (Table 3a).

As for DD, using macroshear test, NCBP adhesive presented significantly higher bond strength than those of others ($p < .05$), using microshear test, again no significant difference was found between all adhesives ($p > .05$) (Table 3b).

Using macroshear test for SD and DD, Weibull distribution presented higher shape ($_0$) for Groups NCBP (4.39) and BAU (4.02) than those of the other groups

Table 3a. The mean bond strength values (MPa ± standard deviations) of all self-etch adhesive resins tested in conjunction with the dual polymerized resin cement on superficial dentin (SD) after macroshear (SBT) and microshear bond test (µSBT). The same superscript lowercase letters in the same column indicate no significant differences based on the test method ($p < .05$). For test group descriptions see Table 1.

Group	Substrate	Test Method	Bond Strength (Mean ± SD)
CBP	SD	SBT	12 ± 10^{A}
BAU	SD	SBT	15 ± 9^{A}
SBU	SD	SBT	13 ± 9^{A}
NCBP	SD	SBT	19 ± 19^{B}
CBP	SD	µSBT	$.2 \pm 0^{a}$
BAU	SD	µSBT	$.1 \pm 1^{a}$
SBU	SD	µSBT	$.8 \pm 1^{a}$
NCBP	SD	µSBT	$.6 \pm 1^{a}$

Table 3b. The mean bond strength values (MPa ± standard deviations) of all self-etch adhesive resins tested in conjunction with the dual polymerized resin cement on deep dentin (DD) after macroshear (SBT) and microshear bond test (µSBT). The same superscript lowercase letters in the same column indicate no significant differences based on the test method ($p < .05$). For test group descriptions see Table 1.

Group	Substrate	Test Method	Bond Strength (Mean ± SD)
CBP	DD	SBT	13 ± 14^{A}
BAU	DD	SBT	10 ± 9^{A}
SBU	DD	SBT	16 ± 17^{A}
NCBP	DD	SBT	19 ± 14^{B}
CBP	DD	µSBT	$1.5 \pm .6^{a}$
BAU	DD	µSBT	1.4 ± 1^{a}
SBU	DD	µSBT	$1.7 \pm .9^{a}$
NCBP	DD	µSBT	1.5 ± 1.3^{a}

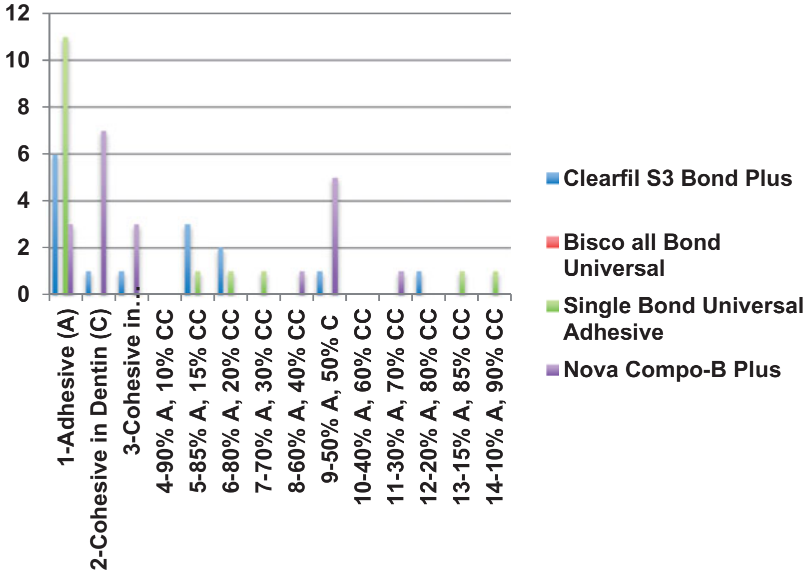

Figure 1. Frequencies of failure modes in percentages after macroshear test in superficial dentin. Score A = adhesive failure between the resin and the dentin; Score C = cohesive failure in dentin, Score CC: cohesive failure in the resin composite.

(CBP: 2.43; SBU: 3.15) on SD but on DD the values were similar (CBP: 2.03; BAU: 2.27; SBU: 2.03; NCBP: 2.46).

Using microshear test for SD and DD, Weibull distribution presented higher shape ($_0$) for Groups SBU (5.1) than those of the other groups (CBP: 3.26; BAU: 2.4; NCBP: 3.1) on SD but on DD, CBP (5.92) showed higher values than those of other groups (BAU: 3.22; SBU: 4.6; NCBP: 2.45).

After macroshear test for SD, except for NCBP where mainly cohesive failures in dentin was observed, with the other adhesive resins the failure types were frequently adhesive in nature (Figure. 1).

On DD, with all adhesives, failure types were exclusively adhesive between the dentin and the resin material with both macro and microshear test methods (Score A).

Discussion

This study was undertaken in order to evaluate the adhesion of conventional resin-based luting cement in conjunction with self-etch adhesive resin systems to SD and DD using both macroshear and microshear tests. Based on the results of this study, the results were dependent on the test method, namely macroshear test resulted in significantly higher results in DD compared to microshear yielding to rejection of the first hypothesis. Macroshear bond strength test resulted in significantly higher bond strength results both on SD and DD compared to microshear test method. Thus, the second hypothesis could be rejected. Furthermore, self-etch adhesive resins with less pH (2.3) did not deliver higher bond strength results than those of the adhesives with higher pH ($\geq$2.5) resulting rejecting the third hypothesis.

Significantly higher results were obtained for bond strength of resin composite to SD and DD when macroshear test method was used. Interestingly, the smaller size of the bonded area did not necessarily resulted in higher bond strength. Although microshear offers bonded areas of 1 to 1.2 mm^2, application of the adhesive through the mould was complex and most possibly led to less good wettability of the adhesive to the dentin surface yielding to practically no adhesion to the dentin substrate. In this study, adhesion procedures were performed on SD and DD in order to simulate clinical indications where restorations are bonded to both types of substrates. In this regard, one important aspect in bonding to dentin is the density and orientation of dentin tubuli. Bonding to DD has been expected to be more challenging than to SD mainly due to the reduced area of solid intertubular dentin associated with the increased water content. There are controversial results supporting this statement. In one study, tubular density and tubular cross-sectional area were not found to be significantly different in deep and coronal dentin [35]. This could be one reason for the insignificant differences between SD and DD when macroshear test was used when the adhesive resins were compared one to one.

In order to measure the bond strength values between an adherent and a substrate accurately, it is crucial that the bonding interface should be the most stressed region, regardless of the test methodology being employed. According to the Griffith's theory [10], the tensile strength of the uniform materials decreases when the specimen size is increased. Overall, adhesion related studies in dentistry, bonded surface areas range from 3 mm^2 to 1 mm^2 in macro- and micro-test methods, respectively [32]. Due to the reduced bonded area and more homogeneous distribution of stresses, micro-test methods tend to show significantly higher bond strength results than the macro-test methods. This could eventually affect the ranking of materials being tested in one study [31]. However, microshear tests resulted in significantly lower values regardless of SD and DD. Since this observation was made for all adhesives used in this study, most likely the wettability of the resin cement was not favourable which was placed through the .8 mm^2 diameter mold. Thus, not only the adhesives but also the rheological properties of the recin cement and in connection to that the technical limitations might be a limitation of microshear tests in adhesion test methods.

One factor affecting the results of shear bond tests is the type of jig. Higher stress concentration was reported with chisel followed by loop type of jigs but where the latter was considered a better test in terms of stress distribution when compared to chisel type of jigs [37]. With the chisel jig, the knife blade may create shear and bending and thus the stress distribution at the interface is not the presumed as solely shear bond strength. In addition, it is almost impossible to achieve homogenous forces at the interface that is in fact also the clinical situation in the majority of the cases. Nevertheless, in order to avoid possible deformation of the stainless steel strip during the tests, a loop type of jig was not considered in this study.

The "etch-and-rinse" adhesives require a moist substrate for optimal bonding [38], making it highly sensitive since the collapse of over-dried, exposed collagen acts as a difficult substrate for the monomer infiltration. However, this also indicates that in the case of increased dentin wetness, which occurs when dentin depth increases, a too wet condition may be created [39]. This makes three-step "etch-and-rinse" adhesives more technique sensitive compared to self-etch adhesive types. Usually, 32–40% ortho-phosphoric acid completely demineralizes 5–8 µm into the intertubular dentin matrix and creates nanometer-sized porosities around collagen fibrils [40]. It also partially demineralizes dentin to a greater depth [41]. Unfortunately, full infiltration of resin monomers within phosphoric acid etched dentin is a complex task. Excessive acid conditioning causes deep demineralization that jeopardizes complete infiltration of resin monomers, thereby resulting in the formation of a weaker and unprotected demineralized dentin zone at the base of these hybrid layers [42]. Adhesives with milder acidity (pH around 2.6) could contribute to the creation of a thinner partially demineralized collagen layer characterized by the presence of mineral precipitation) [41] and less degradation under the hybrid layer. Although the interaction of the etching agentes with dentin is limited by the buffering effect of the mineral and organic phases, there is often a discrepancy between the depth of dentin demineralization versus monomer penetration [43]. Self-etching primers which contain non-rinsing, acidic, polymerizable monomers dissolve the smear layer, or incorporate it into the bonding interface, as it demineralizes the surface and covers collagen fibrils and hydroxyapatite crystals. Demineralization and monomer infiltration occur simultaneously, preventing collagen from collapsing and avoiding the exposure of an unprotected collagen network [43].

The self-etching primers eliminate the technique-sensitive rinsing step to remove the phosphoric acid from dentin. However, the most efficient self-etching adhesives are based on strongly acidic adhesive monomers. Most of the currently available self-etching adhesives are methacrylate-based with a pH value in the range of 1.5–2.5. In this study, two types of pH was of interest, namely pH of <2.3 and pH of 2.5 to 3.5. The results of this study indicated more favourable results with NCBP with pH of 2.5 to 3 whereas BAU with similar pH resulted in lower bond strength values. These results indicate that not only pH but the chemistry might have been influential on the results. The presence of HEMA in NCBP might have increased wettability and the MDP served for the chemical adhesion with the dentin. In addition to that 4-META might have contributed to degree of polymerization [44].

Bond strength results in adhesion studies should be also interpreted with failure types. Cohesive failures in the substrate and combination of adhesive and cohesive failure types in the substrate and bonding agent indicate that bond strength of the adhesive system and the resin cement exceeds that of the cohesive strength of the substrate. After macroshear test for SD, except for NCBP where mainly cohesive failures in dentin was observed, with the other adhesive resins the failure types were frequently adhesive. This finding shows that adhesion remains to be still less reliable on DD.

The presence of pulpal fluid under pressure in vital dentin could be an important factor influencing adhesive bonding to dentin [45]. One limitation of this study was the lack of such a simulation. In future studies, pulpal pressure should also be simulated when comparing the durability of adhesion of the adhesive resins tested [46,47].

Considering bond strength values and the failure types, self-etch adhesives, not only the pH but the chemical composition of adhesive resins on dentin dictates the level of adhesion especially in SD. However, in DD, practically the difference between the adhesive resins appears to be less of significance.

Conclusions

From this study, the following could be concluded:

1. All self-etch adhesives tested presented significantly higher bond strength on superficial dentin compared to deep dentin.
2. Macroshear test presented higher results for both superficial and deep dentin compared to microshear test.
3. Considering Weilbull parameters, characteristics of adhesion and thereby interfacial strength seems to be more reliable with NCBP in macroshear but with SBU in microshear test.
4. After macroshear test, failure types were mainly cohesive in the resin but after microshear test primarily adhesive failures between the material and dentin were common.

Clinical Relevance

Considering bond strength values and the failure types, with the self-etch adhesives tested, not only the pH but the chemical composition dictates adhesion especially to superficial dentin. In deep dentin, the difference between the adhesive resins appears to be less of significance.

Acknowledgement

We would like to acknowledge Imicryl, GmBH, Konya, Turkey for generous provision of resin cements used in this study. The authors further acknowledge Mr. A. Trottmann, University of Zürich, Center for Dental and Oral Medicine, Zürich, Switzerland, for his assistance with the specimen preparation, Dr. M. Roos, from the Division of Biostatistics, Institute of Social and Preventive Medicine, University of Zurich, Switzerland for her support with the statistical analysis.

Disclosure staement

The authors did not have any commercial interest in any of the materials used in this study.

References

[1] Edelhoff D, Özcan M. To what extend does the longevity of fixed dental prostheses depend on the function of cement? Clin Oral Implants Res. 2007;18:193–204.
[2] Manso AP, Carvallho RM. Dental cements for luting and bonding restorations: self-adhesive cements. Dent Clin North Am. 2017;61:821–834.
[3] Van Meerbeek B, De Munk J, Yoshida Y, et al. Adhesion to enamel and dentin: current status and future challenges. Oper Dent. 2003;28:215–235.
[4] Tay FR, Pashley DH. Aggressiveness of contemporary self- etching systems 1: depth of penetration beyond smear layers. Dent Mater. 2001;17:296–308.
[5] Tay F, Pashley D, Yoshiyama M. Two modes of nanoleakage expression in single-step adhesives. J Dent Res. 2002;81:47–476.
[6] Ferracane JL, Stansbury JW, Burke FJT. Self-adhesive resin cements- chemistry, properties and clinical considerations. J Oral Rehabil. 2010;38:1–20.
[7] Armstrong SR, Vargas MA, Fang Q, et al. Microtensile bond strength of a total-etch 3-step, total-etch 2-step, self-etch 2-step, and a self-etch 1-step dentin bonding system through 15-month water storage. J Adhes Dent. 2003;5:47–56.
[8] Melo RM, Özcan M, Barbosa SH, et al. Bond strength of two resin cements on dentin using different cementation strategies. J Esthet Restor Dent. 2010;22:262–268.
[9] Yoshida Y, Nagakane K, Fukuda R, et al. Comparative study on adhesive perform- ance of functional monomers. J Dent Res. 2004;83:454–458.
[10] Rosa WL, Piva E, Silva AF. Bond strength of universal adhesives: A systematic review and meta-analysis. J Dent. 2015;43:765–776.
[11] Suh BI, Feng L, Pashley DH, et al. Factors contributing to the incompatibility between simplified-step adhesives and chemically cured or dual-cured composites. Part III. Effect of acidic resin monomers. J Adhes Dent. 2003;5:267–282.
[12] Spencer P, Wang Y, Walker MP, et al. Molecular structure of acid-etched dentin smear layers-in situ study. J Dent Res. 2007;80:1802–1807.
[13] Choi AN, Lee JH, Son SA, et al. Effect of dentin wetness on the bond strength of universal adhesives. Materials. 2017;10:1224.
[14] Tay FR, Pashley DH, Suh BI, et al. Single-step adhesives are permeable membranes. J Dent. 2002;30:371–382.
[15] Chang J, Platt JA, Yi K, et al. Quantitative comparison of the water permeable zone among four types of dental adhesives used with a dual-cured composite. Oper Dent. 2006;31:346–353.
[16] Tay FR, King NM, Suh BI, et al. Effect of delayed activation of light-cured resin composites on bonding of all-in-one adhesives. J Adhes Dent. 2001;3:207–225.
[17] King NM, Tay FR, Pashley DH, et al. Conversion of one-step to two-step self-etch adhesives for improved efficacy and extended application. Am J Dent. 2005;18:126–134.
[18] Van Landuyt KL, Snauwaert J, De Munck J, et al. Origin of interfacial droplets with one-step adhesives. J Dent Res. 2007;86:739–744.
[19] Pereira PNR, Okuda M, Sano H, et al. Effect of intrinsic wetness and regional difference on dentin bond strength. Dent Mater. 1999;15:46–53.
[20] Sensi L, Lopes G, Monteiro S, et al. Dentin bond strength of self-etching primers/adhesives. Oper Dent. 2005;30:63–68.
[21] Gale MS, Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. J Dent. 1999;27:89–99.
[22] Cheong C, King NM, Pashley DH, et al. Incompatibility of self-etch adhesives with chemical/dual-cured composites: two-step vs one-step systems. Oper Dent. 2003;28:747–755.

[23] Bolhuis PB, de Gee AJ, Kleverlaan CJ, et al. Contraction stress and bond strength to dentin for compatible and incompatible combinations of bonding systems and chemical and light-cured core build-up resin composites. Dent Mater. 2006;22:223–233.

[24] Yoshiama M, Matsuo T, Ebisu S, et al. Regional bond strengths of self-etching/self-priming adhesive systems. J Dent. 1998;26:609–616.

[25] Özcan M, Mese A. Adhesion of conventional and simplified resin-based luting cements to superficial and deep dentin. Clin Oral Investig. 2012;16:1081–1088.

[26] Adebayo OA, Burrow, Tyas M. Bonding of one-step and two-step self-etching primer adhesives to dentin with different tubule orientations. Acta Odontol Scand. 2008;66:159–168.

[27] Cardenas AFM, Siqueira FSF, Bandeca MC, et al. Impact of pH and application time of meta-phosphoric acid on resin-enamel and resin-dentin bonding. J Mech Behav Biomed Mater. 2018;78:352–361.

[28] Della Bona A, Van Noort R. Shear vs. tensile bond strength of resin composite bonded to ceramic. J Dent Res. 1995;74:1591–1596.

[29] Versluis A, Tantbirojn D, Douglas WH. Why do shear bond tests pull out dentin? J Dent Res. 1997;76:1298–1307.

[30] Betamar N, Cardew G, van Noort R. Influence of specimen designs on the microtensile bond strength to dentin. J Adhes Dent. 2007;9:159–168.

[31] Valandro LF, Özcan M, Amaral R, et al. Effect of testing methods on the bond strength of resin to zirconia-alumina ceramic: microtensile versus shear test. Dent Mater J. 2008;27:849–855.

[32] Human Research Ordinance (810.301), Art. 30.

[33] World Medical Association (WMA): Declaration of Helsinki – Ethical Principles for Medical Research Involving Human Subjects. 64th WMA General Assembly, Fortaleza, Brazil, 2013.

[34] Human Research Act (810.30), Art. 2 and 32, Human Research Ordinance (810.301), Art. 25.

[35] Lopes GC, Perdigão J, Lopes Mde F, et al. Dentin Bond strengths of simplified adhesives: effect of dentin depth. Compend Contin Educ Dent. 2006;27:340–346.

[36] Ulker M, Özcan M, Sengün A, et al. Effect of artificial aging regimens on the performance of self-etching adhesives. J Biomed Mater Res B Appl Biomater. 2010;93:175–184.

[37] Özcan M, Kojima AN, Nishioka RS, et al. Effect of jig design and assessment of stress distribution in testing metal-ceramic adhesion. J Prosthodont. 2016;25:665–669.

[38] Carvalho RM, Pegoraro TA, Tay FR, et al. Adhesive permeability affects coupling of resin cements that utilize self-etching primers to dentine. J Dent. 2004;32:55–65.

[39] Paul SJ, Leach M, Rueggeberg FA, et al. Effect of water content on the physical properties of model dentine primer and bonding resins. J Dent. 1999;27:209–214.

[40] Pashley DH, Tay FR, Breschi L. et al. State of the art etch-and-rinse adhesives. Dent Mater. 2011;27:1–16.

[41] Feitosa VP, Bazzocchi MG, Putignano A. et al. Dicalcium phosphate (CaHPO4.2H2O) precipitation through ortho- or meta-phosphoric acid-etching: effects on the durability and nanoleakage/ultra-morphology of resin-dentine interfaces. J Dent. 2013;41:068–1080.

[42] Hashimoto M, Ohno H, Endo K, et al. The effect of hybrid layer thickness on bond strength: demineralized dentin zone of the hybrid layer. Dent Mater. 2000;16:406–411.

[43] Jacques P, Hebling J. Effect of dentin conditioners on the microtensile bond strength of a conventional and a self-etching primer adhesive system. Dent Mater. 2005;21:103–109.

[44] Hanabusa M, Yoshihara K, Yoshida Y, et al. Interference of functional monomers with polymerization efficiency of adhesives. Eur J Oral Sci. 2016;124:204–209.

[45] Caiado AC, de Goes MF, de Souza-Filho FJ, et al. The effect of acid etchant type and dentin location on tubular density and dimension. J Prosthet Dent. 2010;103:352–361.
[46] Griffith AA. The phenomena of rupture and flow in solids. Phil Trans R Soc London, Ser. A 221: 168–98, 1920.
[47] Van Meerbeek B, Peumans M, Poitevin A, et al. Relationship between bond strength tests and clinical outcomes. Dent Mater. 2010;26:100–121.