

Prime&Bond active™

Universal Adhesive



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1 Introduction

Today's one-bottle dental adhesives market is more complex than ever before. New products were constantly put on the field, several new players have entered the game. A new class of one-step universal solutions has emerged recently, simultaneously competing with the established etch&rinse (E&R) and self-etch (SE) adhesives.^[1-10]

Until today, there is no standardized definition of the term «universal» naming the new class of the so called universal adhesives. One way to define it is to refer to the full set of possible etching modes covered by the product including E&R, SE and selective enamel-etch (SEE). In these terms, Xeno[®] Select is a representative of this class.

An extended definition refers to the use for direct and indirect restorations in combination with dual- and self cure materials, including additional primer properties on zirconia oxide ceramics.^[11] In this broadest sense, the term «universal» covers different etching modes and different dental substrates. Here, Prime&Bond active[™] with its Active-Guard[™] technology is introduced.

As there are several one-bottle universal adhesives available on the market; what exactly makes Prime&Bond active[™] so unique regarding its technology and handling? Why is Prime&Bond active[™] NOT just another universal adhesive?

During the development of Prime&Bond active[™], we focused on dentists' expectations: a versatile, robust product, being easy in use and application, providing a precise and economical (pre)dose and finally, offering an additional advantage. Therefore, we developed a new one-hand bottle. Overall, Prime&Bond active[™] provides more than versatility and bond strength. Since we would like to support dental practitioners in their daily work by offering them a new and convenient adhesive, we put special emphasis on what can be defined as “*pro-active handling properties*”.

2 Prime&Bond active™ - product description

Prime&Bond active™ is a one-component universal dental adhesive. It is available in:

- Tri-layer bottle with flip-top-cap
- Disposable containers for single patient use

Prime&Bond active™ is indicated for:

- Direct, light cured composite and compomer restorations
- Composite, ceramic and amalgam repairs
- Cavity varnish for use with fresh amalgam
- Indirect restorations and endodontic posts cemented with Calibra® Ceram
- Direct dual cure / self cure composite restorations and core build-ups¹
- Cementation of indirect restorations and endodontic posts using dual cure / self cure resin cements¹

2.1 General Composition

Prime&Bond active™ is a mild-etch (pH value > 2.5) adhesive formulation containing newly developed hydrolysis stable crosslinker, phosphoric acid esters, isopropanol and water. An overview of the composition and the general function of each component is given in the following Table 1.

Component	General function
Bi- and multifunctional acrylate	Surface active crosslinker
Phosphoric acid modified acrylate resin	Etchant, adhesion promoter, primer
Initiator	Photoinitiator system
Stabilizer	Stabilize monomers upon storage
Isopropanol	Solvent for the resins, polarity adjustment
Water	Solvent for the resins, etching aid

Table 1 Composition of Prime&Bond active™, general function of components.

¹ In combination with Dentsply Sirona Self Cure Activator.

Prime&Bond active™ does not contain inorganic filler particles. Usually, such particles were used to increase the viscosity or to ensure a uniform, sufficiently thick adhesive layer. Both, the low viscosity of Prime&Bond active™ (<30 mPa*s) and its low film thickness after polymerization (<10 µm) are considered optimal. The required film forming properties were already ensured by the resin composition. Thus, additional fillers were obsolete and would just unnecessarily complicate the mixture design.

To realize the desired “*pro-active handling properties*”, a thorough evaluation of all commercially available adhesive components was conducted prior to development.

2.2 Acidic Monomers

Components with a clear advantage, good penetration behavior such as the clinically proven phosphoric acid modified acrylate resins PENTA (dipentaerythritol pentacrylate phosphate) and MDP (10-methacryloyloxydecyl dihydrogen phosphate) were considered and included into the mixture design, see Figure 1.

Due to the phosphoric acid ester moieties of MDP and PENTA, their concentration is of great importance. We combined both monomers in the proper way to give the final adhesive formulation the desired pH value of around 2.5 (mild-etch). Besides the well-balanced hydrophilicity/hydrophobicity of the formulation, such a careful pH control is crucial for universal adhesives. It ensures the desired full compatibility with other resin based dual cure/self cure composites and cements.^[8]

Our combination of the widely used MDP monomer with the proprietary Dentsply Sirona monomer PENTA in Prime&Bond active™ unites the benefits of both structures.

Both monomers reliably etch the dental substrate releasing solvated ions of calcium. These ions were instantly retained in the hybrid layer by forming irreversible calcium-phosphate complexes. As MDP and PENTA contain polymerizable double bonds, their various calcium-phosphate complexes were subsequently integrated into the polymer network upon curing. In particular, MDP contains a long, quite hydrophobic alkyl linker chain and one double-bond per molecule. It forms self-assembled nanolayer structures which further strengthen the hybrid layer of the adhesive.^[12-17]

The PENTA molecule shows a calcium ion complexation similar to that of MDP but due to its bulky structure, is unlikely to show nanolayering.^[18] In contrast, PENTA exhibits intrinsic advantages that are not covered by MDP. It contains a hydrophilic core and five double-bonds per molecule. Thus, it is not only a highly effective crosslinker but also a powerful wetting aid – and renders Prime&Bond active™ this unique.

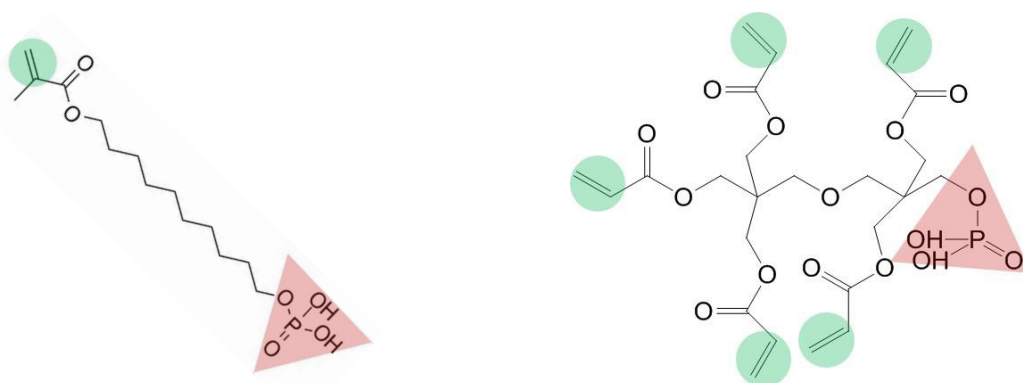


Figure 1 Structure of the acidic monomers MDP (left) and PENTA (right) used in Prime&Bond active™. Reactive double bonds (circles) and acidic moieties (triangles) are marked.

2.3 Resin Components

Commercially available bi- and multifunctional acrylate resins appeared suitable at first sight. On a closer look, they soon revealed undesired limitations and consequently, were excluded from further development.^[6]

A conventional acryl resin mixture in classical dental adhesives preferably consists of two dedicated parts A and B: A) a crosslinker such as *bis*GMA (Bisphenol A diglycidyl methacrylate) or (D)UDMA ((di)urethane dimethacrylate) in combination with B) a reactive diluent such as the low molecular HEMA (2-hydroxyethyl methacrylate) or the highly flexible TEGDMA (triethyleneglycol dimethacrylate).

Whereas the main role of the mostly hydrophobic A is to ensure long-term durability and clinical longevity by forming a dense and stable network upon polymerization, the main role of the mostly hydrophilic B is to reduce the high viscosity and increase matrix solubility of A. Although B is very suitable to ensure high dentinal penetration rates of

the resulting A plus B mixture (A+B), both parts A and B introduce disadvantages. A for example, might form strong intermolecular hydrogen bonds – resulting in a very high intrinsic viscosity ($>> 100 \text{ Pa}\cdot\text{s}$) and poor water solubility. B on the other hand, has an excellent water solubility and a very low viscosity ($< 0.01 \text{ Pa}\cdot\text{s}$) that might result in a high water sorption, high leaching rates of non-polymerized B from the cured material and as a consequence, in undesired water trees found in the hybrid layer.

It is certainly possible to generate a well-balanced, A+B based resin composition. Under best conditions, a stable state of equilibrium can be established resulting in good quality products. However, an A+B resin system remains a binary concept wherein the single components A and B still have opposite chemical and physical properties. Thus, A+B systems are intrinsically sensitive towards external influences that *introduce a deviation from the preset state of equilibrium*. For example, selective evaporation of one component A or B shifts the A/B concentration in the formula. The character of the counterpart B or A in the formulation becomes predominant, destabilizing its balance. Addition of a third component C such as water, also disturbs the state of equilibrium which can end up in three different scenarios: I. water is actively incorporated, but leads soon to a phase separation of A, B or mixed phases thereof (*detrimental*). II. water is not incorporated at all or shows just marginal incorporation (*passive*). With increasing water amounts, a collapse of the homogenous A+B+C system and in consequence, irreversible phase separation to an A+B and C system may occur. Or, the A+B mixture/adhesive and added water phase C remain unaffected, see Figure 2.

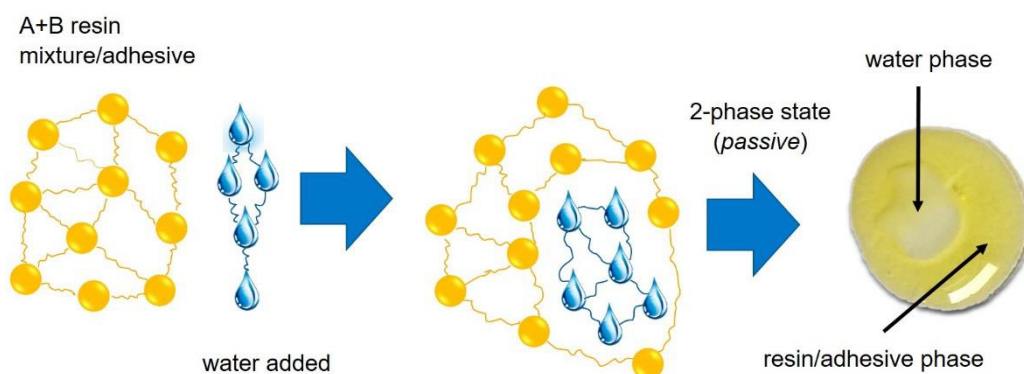


Figure 2 *Passive* behavior of A+B type resin/adhesive when contacted with a small amount of water C; remaining a heterogeneously separated A+B and C phase system.

Consequently, A+B based adhesive systems in heterogeneous scenarios I. and II. cannot be evenly, uniformly distributed on a surface and would not give a homogeneous adhesive layer. Their application on an overly wet substrate would have the same result as shown in Figure 2. In contrast, their application on a strongly absorbent substrate such as over-dried dentin could result in a detrimental shift in the A/B concentration, observed as insufficient wettability and/or penetration behavior.

Both, this undesired behavior and the strategy for its prevention are a matter of molecular thermodynamics, mixture kinetics and wetting properties of liquids on surfaces. The most desired *active* scenario III, was pursued with Prime&Bond active™.

In contrast to the classical A+B approach discussed above, we followed an innovative A–B approach when developing Prime&Bond active™. This new A-B concept would unify all the desired beneficial features within one molecule and simultaneously minimize or even exclude undesired properties. The result culminated in the Active-Guard™ technology. Increased water compatibility could successfully be realized within one single monomer of the A–B type. A new liquid, bifunctional acryl crosslinker containing N-allyl functions revealed a high level of polymerization reactivity ($> 150 \text{ kJ/mol}^{-1}$). The exclusion of hydrogen bonds resulted in a very low viscosity ($< 0.5 \text{ Pa}\cdot\text{s}$).

Such a design thoroughly balances the hydrophobic and hydrophilic contributions of A and B, which were built-in inseparably in one single molecule. As a result, the Active-Guard™ technology helps to achieve an optimized surface tension and low contact angle on dental substrates. Finally, the quite high surface tension of water (72.8 mN/m^{-1}) could effectively be overcome. A “*pro-active*”, spontaneous incorporation of small amounts of water is realized avoiding undesired phase separation; see Figure 3. The chemical structure of the new crosslinker is shown in Figure 4.

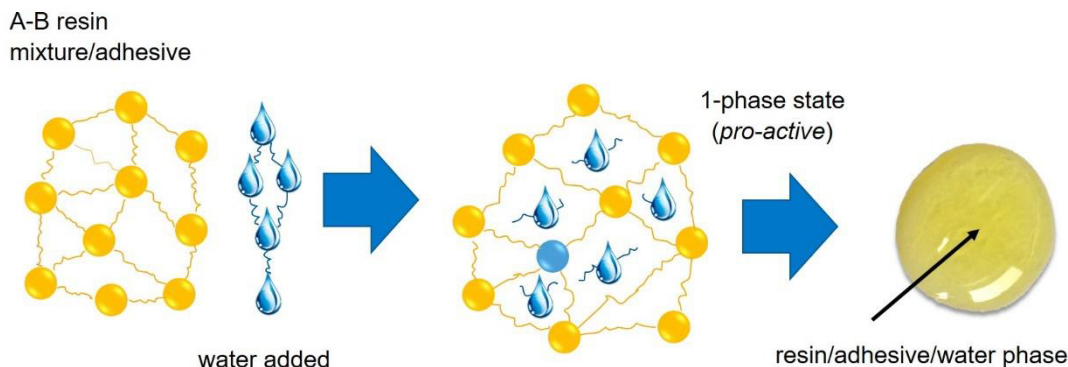


Figure 3 “*Pro-active*” behavior of A–B type resin mixture/adhesive when contacted with a small amount of water (Dentsply Sirona Active-Guard™ technology, quickly resulting in one homogenous phase).

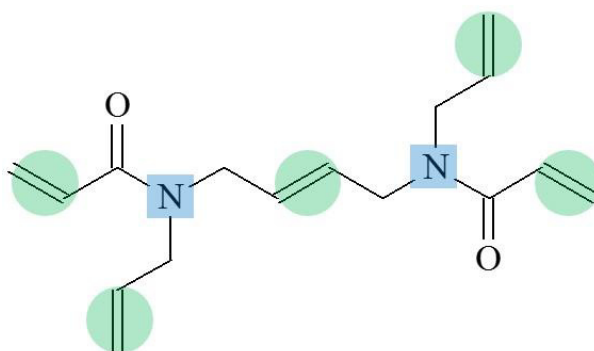


Figure 4 Structure of the new crosslinker, part of the Active-Guard™ technology. Reactive double bonds (circles) and N-allyl centers (squares) are marked.

2.4 Solvent System

A careful selection of the solvent system is a key step in adhesive development. However, there is no general answer which solvent system should be taken for a given resin composition. After the decision for a preferred resin mixture was made, the solvent system needs to be adjusted individually and under consideration of the targeted evaporation times.^[7] Thereby, pros and cons of each (co)solvent and their possible influences should be taken into account, see Figure 5.

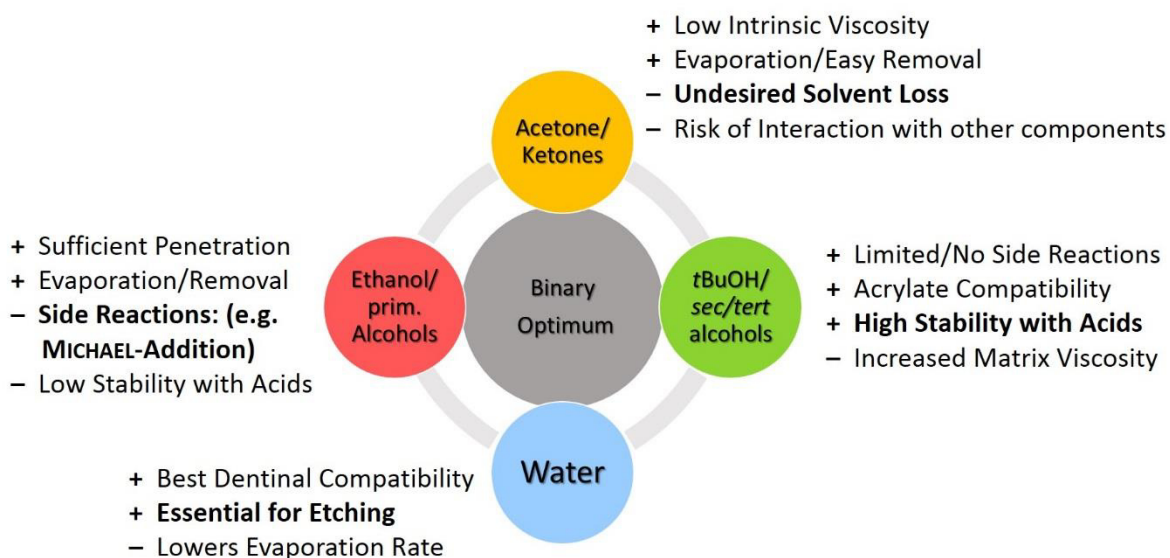


Figure 5 Overview of common solvent systems used in dental adhesives including the respective pros and cons.

The starting point is certain: water is an essential ingredient of the formula. It ensures sufficient etching performance when the universal adhesive is applied in SE and SEE mode. The high relative polarity of water (1.000) in combination with its low vapor pressure makes it hard to remove upon air-blow procedures prior to light curing. That is why it is usually combined with a second co-solvent in a so called binary mixture.

One possible candidate, isopropanol is fully soluble with water in every ratio. Its low viscosity (2.4 mPa*s at 20°C) is more than 400-fold lower than that of water (1000 Pa*s at 20°C). The relative polarity of isopropanol (0.546) is lower when e.g. compared with the primary alcohol ethanol (0.654). The viscosity of isopropanol (2.4 mPa*s) is twice as high as those of ethanol (1.2 mPa*s). Although isopropanol has a similar boiling point (82°C) to that of ethanol (78°C), they may result in different solubility, wetting and evaporation properties when combined with water in more complex adhesive mixtures.

Despite the fact that isopropanol is less hydrophilic and has a slightly higher viscosity than ethanol, we found it to be perfectly suitable to be combined with our new, low viscous Active-Guard™ technology. As co-solvent, isopropanol offers the best balance between polarity and surface wetting properties under various amounts of residual moisture. It revealed advantages in terms of the storage stability of the formulation (e.g. reduced risk of Michael addition reaction when compared to ethanol), economical dosing and in terms of *on-demand removability* of the water-alcohol vapor. Thanks to

the optimized water-isopropanol solvent system used in Prime&Bond active™, you may conveniently use it even after 30 min of storage in a closed CliXdish™.

Prime&Bond active™ offers high application robustness even on over-dried and over-wet dentin. It is robust towards various degrees of moisture and offers active moisture control on teeth, which results in spontaneous surface coverage and virtually no post-operative sensitivities. In indirect scenarios, its low viscosity in combination with its low film thickness forms a uniform, consistent film layer and ensures best fitting results. Its formula is free of HEMA, UDMA, TEGDMA and Bisphenol-based chemistry.

3 In vitro Investigations

As the clinical performance of an adhesive highly depends on the operator and other influencing factors, a first step in evaluating a novel adhesive should be in vitro testing under conditions as controlled as possible in order to allow comparison to controls. As there is no single in vitro method allowing a conclusive prognosis of the clinical outcome, a comprehensive testing was conducted in order to evaluate the potential of the universal adhesive Prime&Bond active when used in different etching modes (etch&rinse, self-etch, and selective enamel-etch). This included bond strength to enamel and dentin, micromorphology, marginal adaptation, and investigations of the adhesive layer.

3.1 Bond strength

3.1.1 Shear bond strength to enamel and dentin

(PROF. MARK LATTA, Creighton University, USA)

Exposed dentin and enamel surfaces of extracted human teeth were ground (600 grit) flat in order to standardize the smear layer. Four groups of 12 specimens each were tested per substrate. Testing was carried out on both enamel and dentin using either etch&rinse or self-etch application per directions for use (DFU) of the adhesives. After light-curing of the adhesives, composite cylinders were bonded to these surfaces using the Ultradent jig and light-cured. Specimens were stored in water for 24 h at 37°C and subjected to thermocycling (6000 cycles at 5/55°C). After further 24 h of storage, shear bond strength was determined in a test machine using an Ultradent notched blade and a crosshead speed of 1 mm/min. Statistical analysis was done by parametric tests at $p < 0.05$.

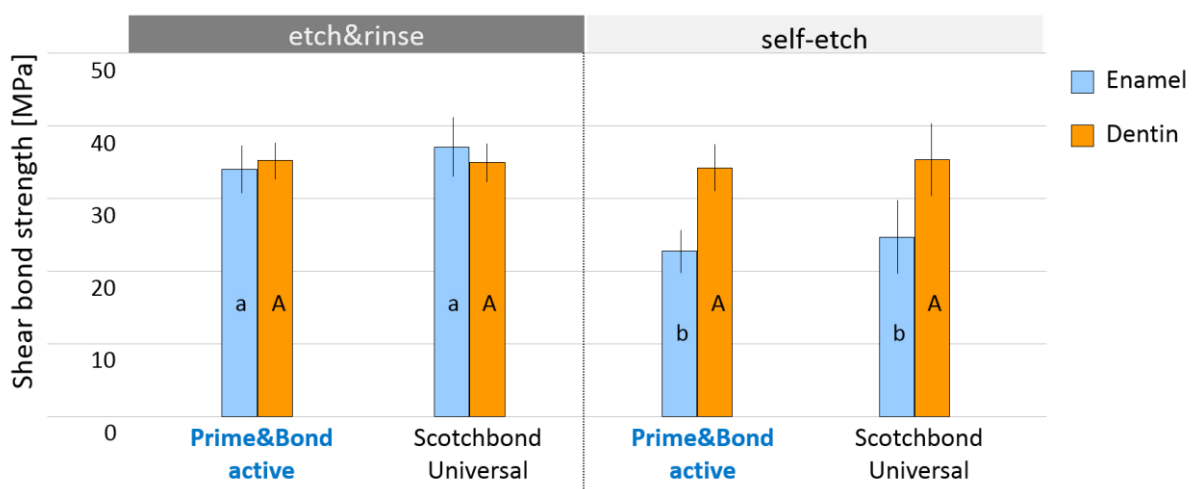


Figure 6 Enamel and dentin bond strength of adhesives in either etch&rinse or self-etch mode after thermocycling. Bars with different letters are significantly different.

Both universal adhesives showed significantly higher mean bond strengths to enamel in the etch&rinse mode than in the self-etch mode. These findings support the use of the selective enamel-etch mode, i.e. etching the enamel margins with phosphoric acid. In dentin, however, the etching mode (etch&rinse versus self-etch) had no significant influence on the bond strength (Figure 6).

3.1.2 Shear bond strength to differently moist dentin

(PROF. MARK LATTA, Creighton University, USA)

Extracted human molars were mesiodistally cut in half, mounted and wet ground (4000 grit) to a flat dentin surface to standardize the smear layer. Dentin surfaces were conditioned according to the etch&rinse technique and divided into 15 groups of 15 specimens each. Dentin bonding was tested either under ideal conditions (i.e. moist dentin) or challenged by different degrees of moisture. Over dried dentin was achieved by thorough air drying for 10 s. Over wet dentin was simulated by application of 2.5 μ L distilled water onto a 4 mm round area. After application and light-curing of the adhesives according to the DFU, brass moulds were used to bond composite cylinders to the treated dentin. The composite was condensed and light-cured, followed by storing the specimens in water for 24 h at 37°C. Shear bond strength was determined using a test machine at a crosshead speed of 1 mm/min. Statistical analysis was carried out at $p < 0.05$.

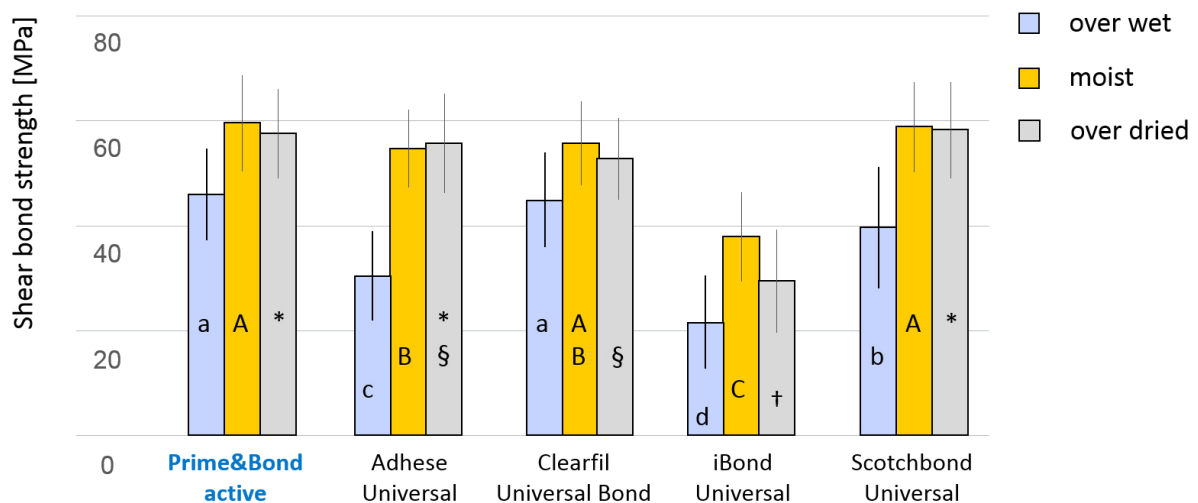


Figure 7 Bond strength of adhesives in etch&rinse mode on differently moist dentin. Bars with different letters or symbols are significantly different.

There were significant differences among the adhesives. Under ideal moist conditions, Prime&Bond active was statistically equal in mean bond strength to Clearfil Universal and Scotchbond Universal. However, under suboptimal conditions, Clearfil Universal had significantly lower bond strengths on over dried dentin, while Scotchbond Universal was significantly more sensitive to over wet dentin (Figure 7).

3.1.3 Shear fatigue strength to differently moist dentin

(PROF. MARK LATTA, Creighton University, USA)

Specimens and bonding procedures for each group (n = 15) were prepared in the same way as described for the bond strength testing in the previous chapter. After 24 h of water storage at 37°C, shear fatigue strength was determined in a test machine by using sinusoidal loading of the specimens at a frequency rate of 10 Hz until 50,000 cycles or until failure. The load was incrementally adjusted upward or downward depending on the survival or failure by approximately 10% of the initial load. The initial maximum load was 50-60% of the shear bond strength determined for each of the adhesives tested (cf. chapter 3.1.2). The lower load limit was set to near zero (0.4 N). Statistical analysis was carried out at $p < 0.05$.

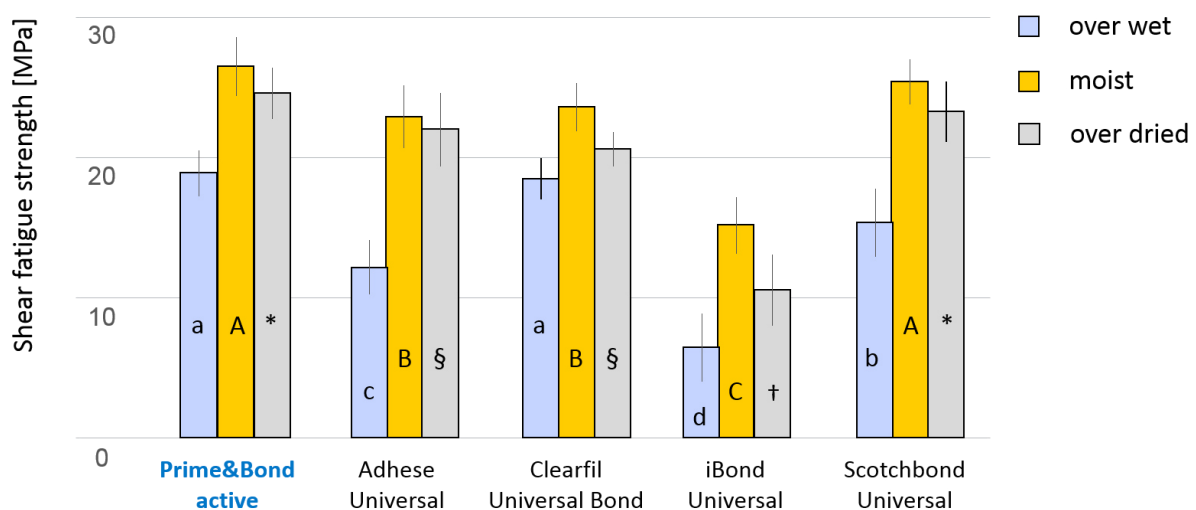


Figure 8 Fatigue strength of adhesives in etch&rinse mode on differently moist dentin. Bars with different letters or symbols are significantly different.

Prime&Bond active applied under ideal moist dentin conditions was found to have fatigue strengths in the range of Scotchbond Universal. Moreover, Prime&Bond active demonstrated high mean fatigue strengths under wet and dry bonding, whereas some other adhesives showed a significant reduction in fatigue strength on over wet or dried dentin surfaces (Figure 8).

3.1.4 Shear bond strength to dentin with varying water content

(R&D DENTSPLY SIRONA, Konstanz, Germany)

Two operators tested the bond strength of five adhesives to dentin with varying water content. Flat dentin surfaces were prepared from extracted human teeth and divided into 15 groups with 30 specimens each. Dentin was conditioned according to the etch&rinse technique, and dentin bonding was tested either under ideal conditions (i.e. moist dentin) or challenged by varying water content. Over wet conditions were achieved by applying either 2.5 μL (25 vol%) or 4 μL (40 vol%) distilled water onto a 4 mm round area. After application and light-curing of the adhesives according to the DFU, composite cylinders were bonded to these surfaces using the Ultradent jig and light-cured. After 24 h of water storage at 37°C, shear bond strength was determined in a test machine using an Ultradent notched blade and a crosshead speed of 1 mm/min. Statistical analysis was done by parametric tests at $p < 0.05$.

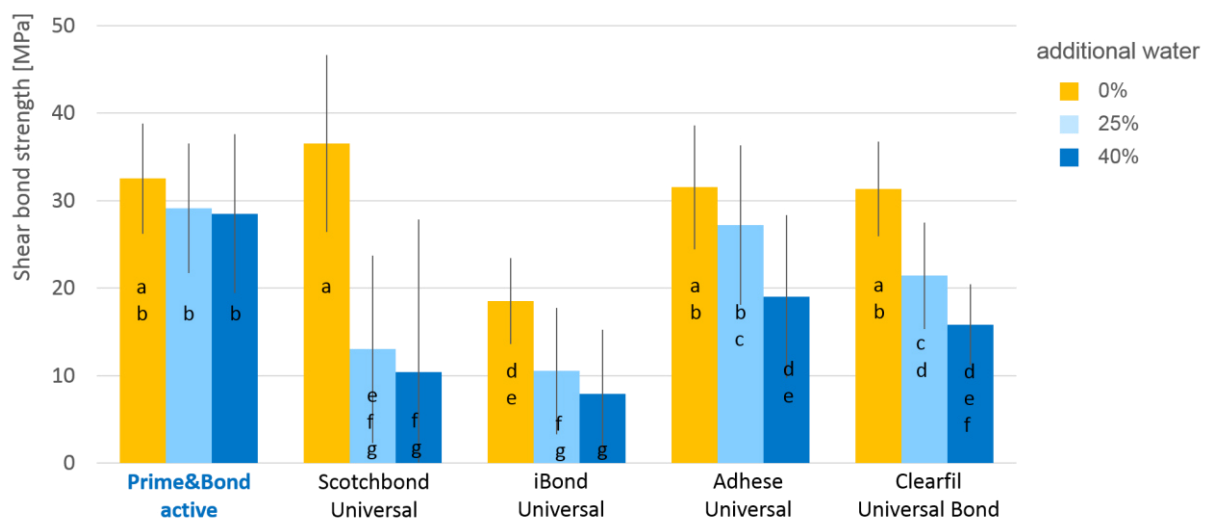


Figure 9 Bond strength of adhesives in the etch&rinse mode on dentin with varying water content. Bars with different letters are significantly different.

Except iBond Universal, no significant differences were found among the tested adhesives under ideal moist conditions. With increasing water content, Prime&Bond active showed bonding robustness towards over wet dentin surfaces, while the mean bond strengths of the other tested adhesives significantly decreased showing less tolerance to excess water (Figure 9).

3.1.5 Microtensile bond strength to dentin under indirect restorations

(Prof. ROLAND FRANKENBERGER, University of Marburg, Germany)

A total of 120 Class I cavities (4 x 4 mm, depth 3 mm) were prepared in extracted human molars. Cerec glass ceramic inlays (Celtra Duo) were hydrofluoric acid etched, silanated, and bonded with different adhesive/luting composite combinations according to the respective DFU. Following the etch&rinse or self-etch technique, the adhesives were either light-cured before seating the ceramic inlay (pre-cured) or cured together with the luting composite through the restoration (not pre-cured). Prime&Bond active was either combined with self-cure activator (SCA) and Calibra Automix or with Calibra Ceram without SCA. After water storage for 24 h at 37°C, the specimens were cut into resin-dentin sticks with a cross-sectional area of 0.5 mm². After further 24 h of water storage at 37°C, 20 sticks per group were subjected to microtensile bond strength testing. Fracture analysis was performed under a scanning electron microscope (SEM). Statistical analysis was done by non-parametric tests at $p < 0.05$.

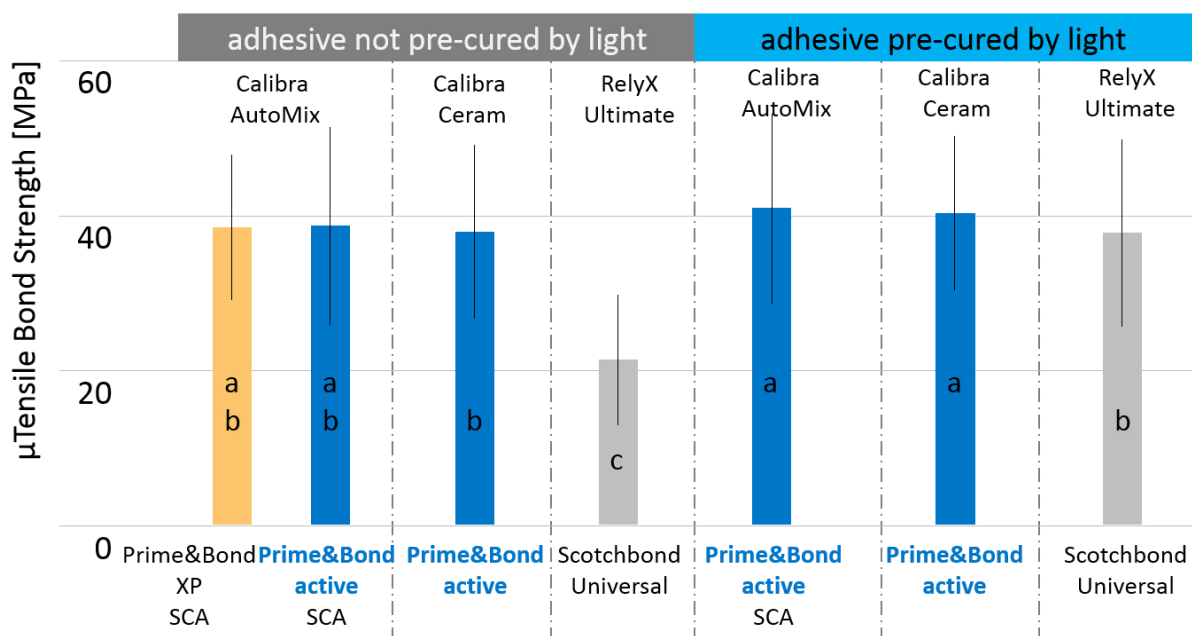


Figure 10 Dentin bond strength of luting systems in the **etch&rinse mode** under ceramic inlays. Bars with different letters are significantly different. SCA = self-cure activator (Prime&Bond XP was used as etch&rinse control).

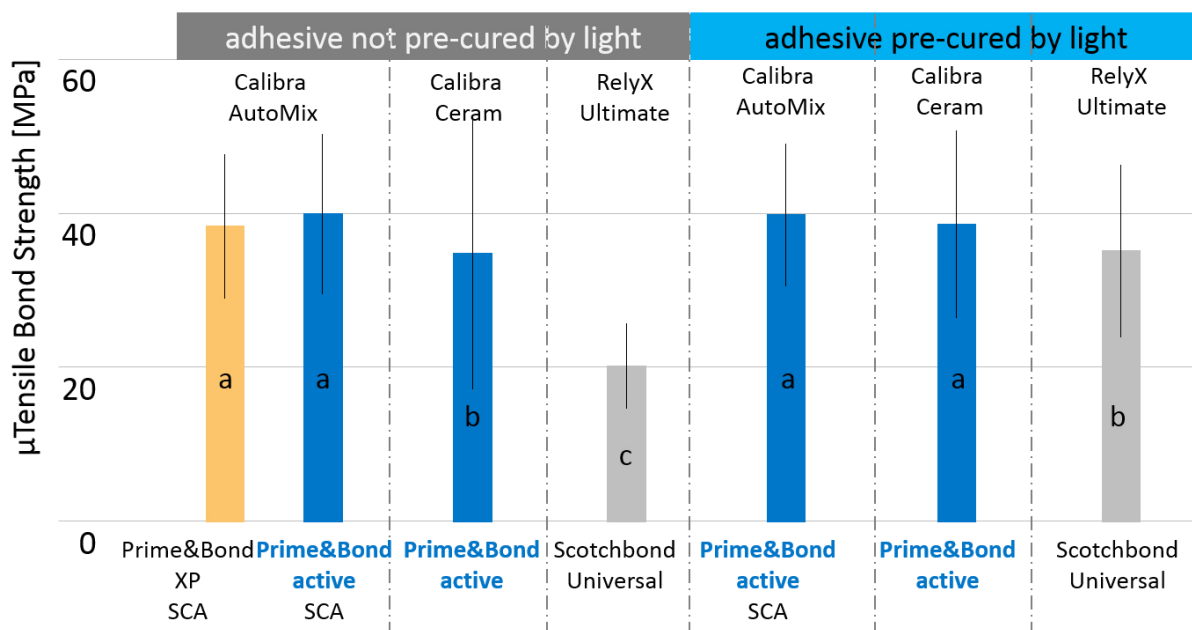


Figure 11 Dentin bond strength of luting systems in the **self-etch mode** under ceramic inlays. Bars with different letters are significantly different. SCA = self-cure activator (Prime&Bond XP was used as etch&rinse control).

Without pre-curing of the adhesive by light, Prime&Bond XP (in combination with SCA and Calibra Automix) and Prime&Bond active (either combined with SCA and Calibra Automix or with Calibra Ceram without SCA) showed significantly higher mean bond strengths than the Scotchbond Universal/RelyX Ultimate luting system. However, bond strengths of the luting systems in which the adhesive layer was pre-cured by light before seating the restoration were statistically equal or significantly higher than when the adhesive was cured at the same time as the luting composite under the thick ceramic (Figure 10 and Figure 11). Therefore, light-curing of Prime&Bond active should be accomplished before seating restorations. Optionally, the adhesive may be light-cured together with the luting composite in case of light-transmissible restorations (limited to ceramic thickness ≤ 2.5 mm).

3.1.6 Shear bond strength to restorative materials

(R&D DENTSPLY SIRONA, Milford, USA)

The bond strength of two luting systems to various restorative materials were compared. Five substrates with at least six specimens each were tested per luting system: glass ceramic (IPS e.max), zirconia (Cercon), cured composite (TPH Spectra LV), base and noble metal alloys (Duceranum U, SMG-CF). Surface treatments consisted of hydrofluoric acid etching and silanization for glass ceramic or sandblasting with 50- μ m aluminium oxide followed by cleaning (ultrasonic in distilled water for 10 min) for the other restorative materials. The adhesives and appropriate luting composites were applied to the treated surfaces according to their respective DFU. Stainless steel rods with 3.2 mm diameter were sandblasted, ultrasonically cleaned and bonded to these surfaces. After self-curing of the luting systems at 37°C for 10 min under controlled seating force, the specimens were stored in water for 24 h at 37°C. Shear bond strength was determined using a test machine at a crosshead speed of 1 mm/min.

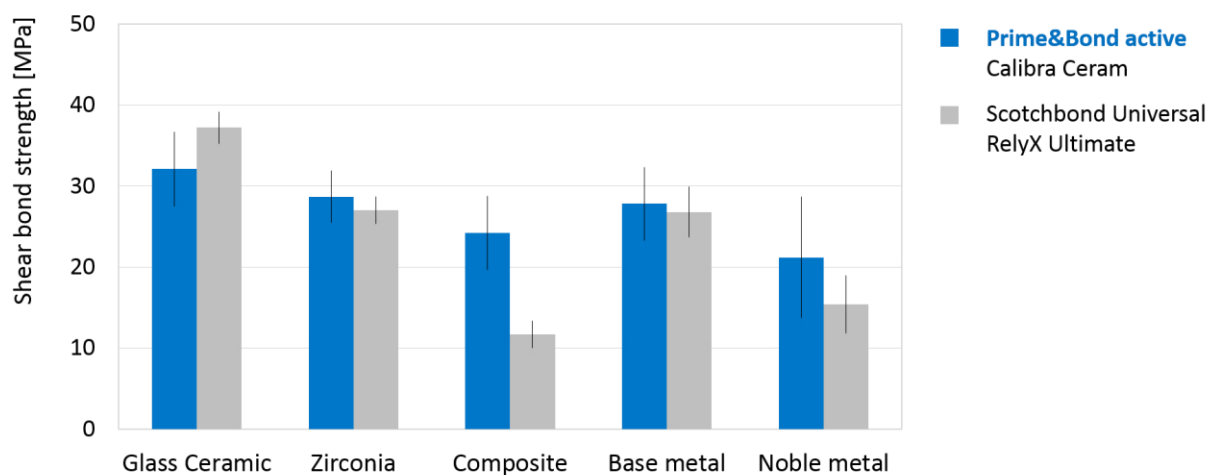


Figure 12 Shear bond strength of luting systems to various restorative materials.

Prime&Bond active in combination with Calibra Ceram showed bond strengths on glass ceramic, zirconia, cured composite, base and noble metal alloys similar to or higher than Scotchbond Universal combined with RelyX Ultimate.

3.1.7 Immediate shear bond strength to dentin

(R&D DENTSPLY SIRONA, Milford, USA)

In each group, six dentin surfaces of extracted human molars were ground (600 grit) flat in order to standardize the smear layer. The tested adhesives were applied on dentin according to the DFU for the self-etch application. Stainless steel rods with 3.2 mm diameter were sandblasted, ultrasonically cleaned and dried. After curing of the adhesives per DFU (Prime&Bond active was light-cured, Scotchbond Universal and Multilink Primer A/B were not light-cured), the appropriate luting composites were applied to the surface of the steel rods and placed onto treated dentin. Specimens were allowed to self-cure at 37°C for 5.5 min under controlled seating force and tested immediately (6 min) after application of luting composites. Shear bond strength was determined in a test machine at a crosshead speed of 1 mm/min. Statistical analysis was done by parametric tests at $p < 0.05$.

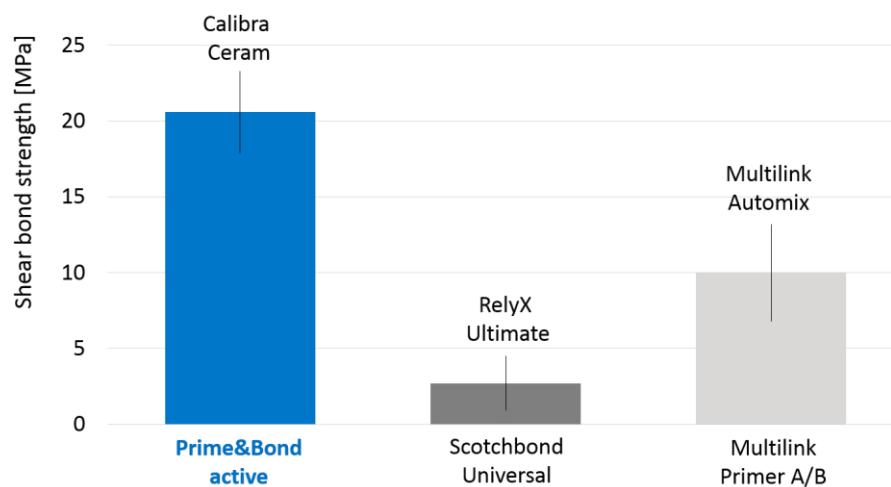


Figure 13 Immediate bond strength of three luting systems on dentin (Prime&Bond active was light-cured, Scotchbond Universal and Multilink Primer A/B were not light-cured per their DFU).

Prime&Bond active in combination with Calibra Ceram showed mean bond strengths on dentin significantly higher than the other tested luting systems six minutes after application of the luting composite.

3.1.8 Retentive strength to crown preparations

(Prof. JOHN BURGESS, University of Alabama, USA)

In each group, 10 extracted human premolars were embedded in acrylic resin and placed into a lathe to standardize crown preparation (22° convergence angle, 3 mm height). The occlusal surface was prepared flat. Preparations were scanned and the surface area was measured for each tooth. Cerec glass ceramic crowns (Celtra Duo, IPS e.max) were hydrofluoric acid etched, silanated, and bonded with different adhesive/luting composite combinations. The adhesives were applied in self-etch mode and cured according to their respective DFU, and the ceramic crowns were bonded with the appropriate luting composite and allowed to self-cure under controlled seating force. After water storage for 24 h at 37°, the specimens were subjected to thermocycling (10,000 cycles at 5/50°C). Next, specimens were held in a special jig to attach them to the test machine and loaded in tension at a crosshead speed of 0.5 mm/min until failure. Failure mode was recorded as adhesive, cohesive or mixed.

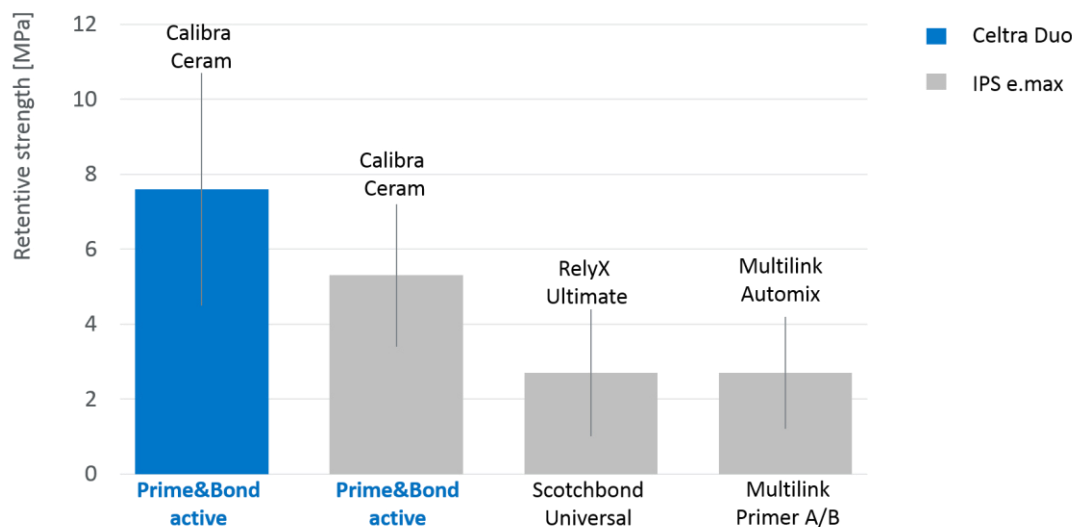


Figure 14 Retentive strength of ceramic crowns using different luting systems. The results for Prime&Bond active/Calibra Ceram come from a separate report, using exactly the same methods by the same operators, as detailed above (Prime&Bond active was light-cured, Scotchbond Universal and Multilink Primer A/B were not light-cured per their DFU).

The combined use of Prime&Bond active and Calibra Ceram resulted in better crown retention compared to the other luting systems under investigation.

3.2 Micromorphology

Incomplete infiltration and sealing of dentin is one of the critical conditions leading to postoperative sensitivity and poor bond strength. After phosphoric acid etching, the dentin should remain moist to avoid collapse of collagen fibers and thus incomplete infiltration of the adhesive. As the moist bonding technique is clinically sensitive, it would be desirable for the adhesive to present a similar bonding performance when applied on moist or over dried dentin. In order to visualize the capability to properly wet and infiltrate such dentin surfaces field emission scanning electron microscopy (FE-SEM) and transmission electron microscopy (TEM) were applied to investigate the resin-dentin interface.

3.2.1 Hybrid layer of moist and over dried dentin

(Prof. ANDRE REIS, Guarulhos University, Brazil)

The exposed dentin surface of 24 extracted human molars were ground (600 grit) flat in order to standardize the smear layer and divided into eight groups. Dentin was conditioned according to the etch&rinse technique, and dentin bonding was tested either under ideal moist conditions or after air drying for 10 s to simulate over dried dentin. After application of four different universal adhesives, the bonded surfaces were covered with a thin layer of SDR composite. Adhesive and composite were light-cured separately. After 24 h of water storage, each specimen was cut into two resin-dentin slabs. One of the slabs was used for FE-SEM, the other for TEM preparation. Slabs for the FE-SEM analysis were embedded in epoxy resin and deproteinized in sodium hypochlorite. Next, they were dehydrated in ascending concentrations of ethanol and sputter coated. Slabs for the TEM analysis were stained and dehydrated in ascending concentrations of ethanol. Then, they were embedded in epoxy resin and prepared in ultra-thin sections. For both microscopic evaluations, representative images of resin-dentin interfaces produced by the adhesives on moist and over dried dentin were recorded.

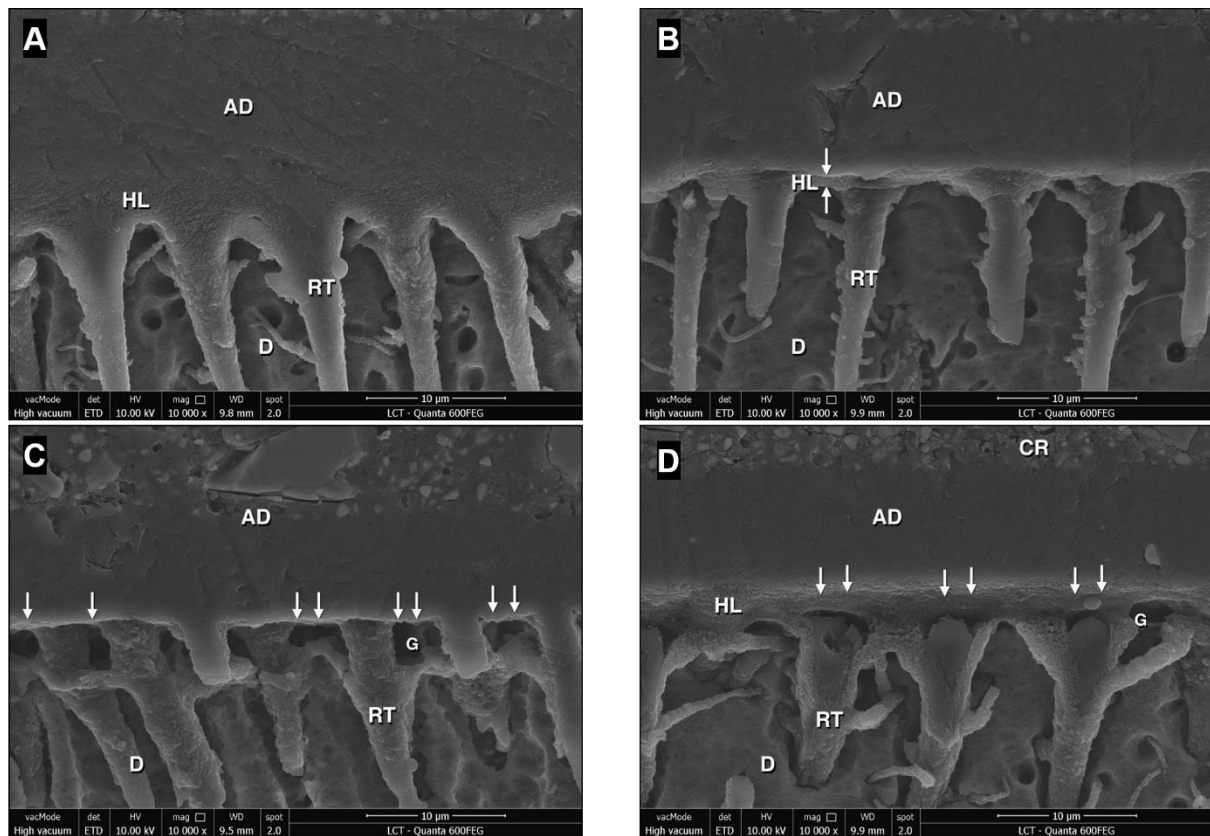


Figure 15 Representative FE-SEM images of resin-dentin interfaces produced by four adhesives applied on etch-and-rinse mode to over dried dentin. While Prime&Bond active produced a well-formed hybrid layer (A), the hybrid layer of Adhese Universal was considerably thinner (B). Arrows point defects and gaps within the resin-dentin interface produced by Futurbond U (C) and Scotchbond Universal (D). AD = adhesive layer, CR = composite resin, D = dentin, G = gap, HL = hybrid layer, RT = resin tag.

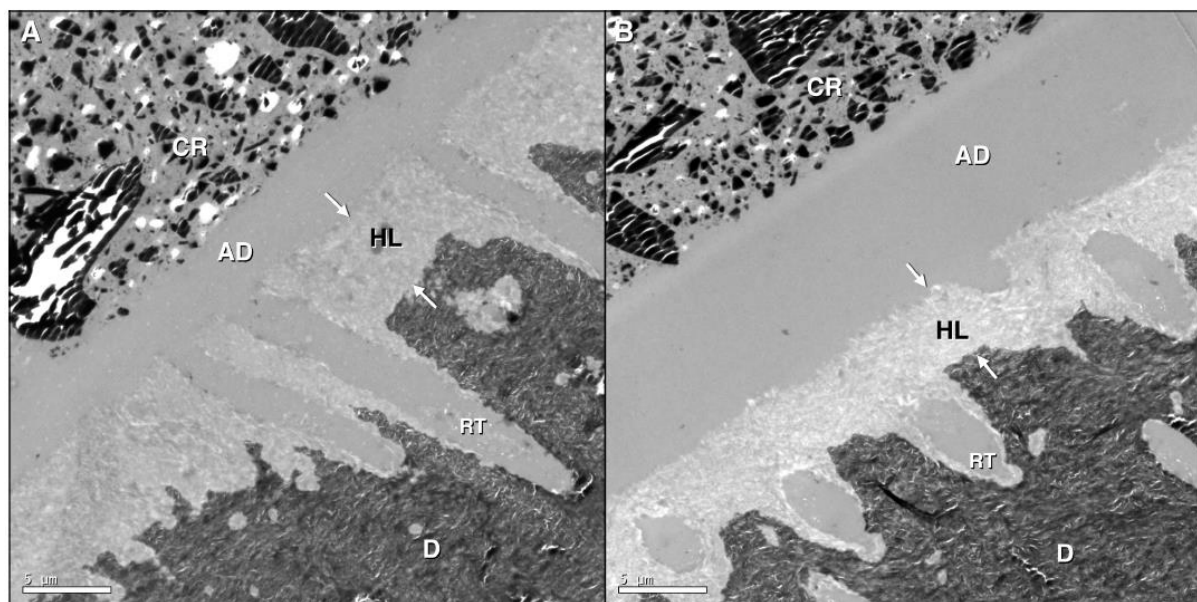


Figure 16 Representative TEM images of a well-formed hybrid layer produced by Prime&Bond active applied on etch-and-rinse mode to moist (A) and over dried (B) dentin. AD = adhesive layer, CR = composite resin, D = dentin, HL = hybrid layer, RT = resin tag.

The investigator summarized the findings as follows: “For moist dentin, a well-formed hybrid layer was observed for all universal adhesives tested. However, when the adhesives were applied to over dried dentin, remarkable differences were observed in comparison to interfaces produced on moist dentin. Defects, gaps and reduced hybrid layer thickness were observed when Adhese Universal, Futurabond U, and Scotchbond Universal were applied to over dried dentin” (Figure 15). The findings from the micromorphological investigation of Prime&Bond active support the robust bond strengths when dentin was over dried as the investigator emphasized: “Prime&Bond active does not seem to be sensitive to the degree of moisture, and presented well-formed hybrid layers when applied to either moist or over-dried dentin” (Figure 16).

3.3 Marginal adaptation

Clinically, marginal adaptation is claimed to be predictable to a certain degree by in vitro chewing simulation. Though there is no correlation to clinical outcome published, using dye penetration to evaluate marginal sealing allows differentiating among adhesives and/or etching modes. Therefore, chewing simulation and microleakage were investigated.

3.3.1 Marginal adaptation of class II composite restorations

(Prof. ROLAND FRANKENBERGER, University of Marburg, Germany)

In each group, eight extended MOD cavities (with cervical margins in enamel and dentin) were prepared in extracted human molars and incrementally filled with composite. The tested universal adhesives were used according to the DFU for either etch&rinse or self-etch application. After finishing and water-storage for 21 days at 37°C, the specimens were subjected to thermocycling (2500 cycles at 5/55°C) and mechanical loading (TML) in a chewing simulator (1000,000 cycles, 50 N). In order to investigate the marginal adaptation of the composite restorations before and after TML, epoxy resin replicas were sputter coated and analyzed under a scanning electron microscope (SEM) at 200x magnification. Results were statistically analyzed by non-parametric tests at $p < 0.05$.

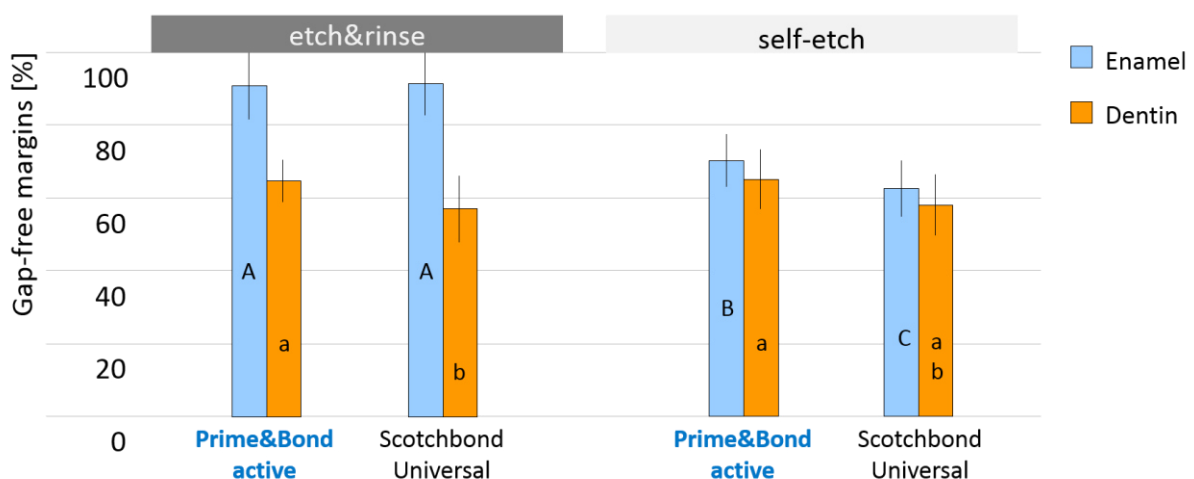


Figure 17 Percentage of gap-free margins of Class II restorations after TML. Bars with different letters are significantly different.

In enamel and dentin, 100% gap-free margins were initially identified for all adhesives and etching modes (data not shown). Comparing the percentage of gap-free margins after TML, however, significant differences were found (Figure 17). In enamel, the etch&rinse mode led to significantly better marginal adaptation than the self-etch mode. In dentin, however, the etching mode had no significant influence on the performance of the adhesives. These findings once again support the use of the selective enamel-etch mode (i.e., etching the enamel margins with phosphoric acid).

3.3.2 Marginal adaptation of class V composite restorations

(Dr. UWE BLUNCK, Charité Berlin, Germany)

In each group, eight Class V cavities (with margins in enamel and dentin) were prepared in extracted human incisors and incrementally filled with the composite ceram.x universal. The universal adhesives were applied according to the DFU in the etch&rinse mode and the self-etch mode, respectively. After finishing and water-storage for 21 days, epoxy resin replicas were taken before and after thermocycling (2000 cycles at 5/55°C) and analyzed under SEM at 200x magnification for marginal adaptation. Statistical analysis was carried out by using non-parametric tests at $p < 0.05$.

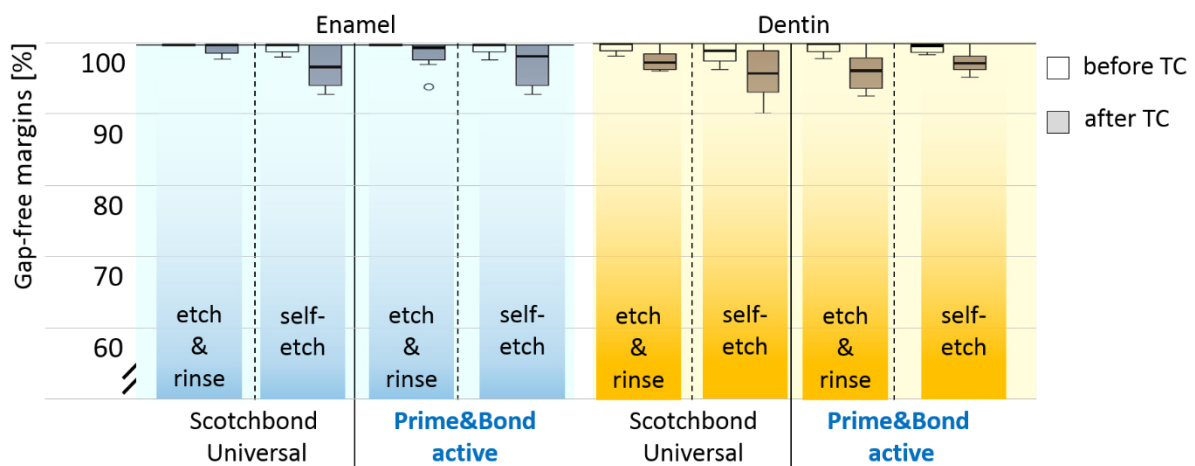


Figure 18 Percentage of gap-free margins of Class V restorations before and after thermocycling (TC). No significant differences were found.

Both adhesives demonstrated comparable high percentages of gap-free margins in enamel and dentin prior and after thermocycling. Moreover, the etching mode of the tested adhesives had no significant influence on the quality of marginal adaptation (Figure 18).

3.3.3 Microleakage of class V composite restorations

(Prof. JUAN IGNACIO ROSALES LEAL, University of Granada, Spain)

Two class V cavities (with margins in enamel and dentin) each were prepared in five extracted human molars per group and filled with composite. The tested universal adhesives were used as per DFU for either etch&rinse or the self-etch application. After 24 h of water-storage at 37°C, the specimens were subjected to thermocycling (4000 cycles at 5/55°C) and sectioned longitudinally. Analysis of microleakage was performed engaging the dye penetration test (0.5% basic fuchsin solution, 24 h, 37°C). The penetration depth was expressed as percentage of the total interfacial length and ranked between hermetic seal and massive microleakage. Additionally, it was measured whether any dye penetrated in the dentinal tubules. Results were statistically analyzed at $p < 0.05$.

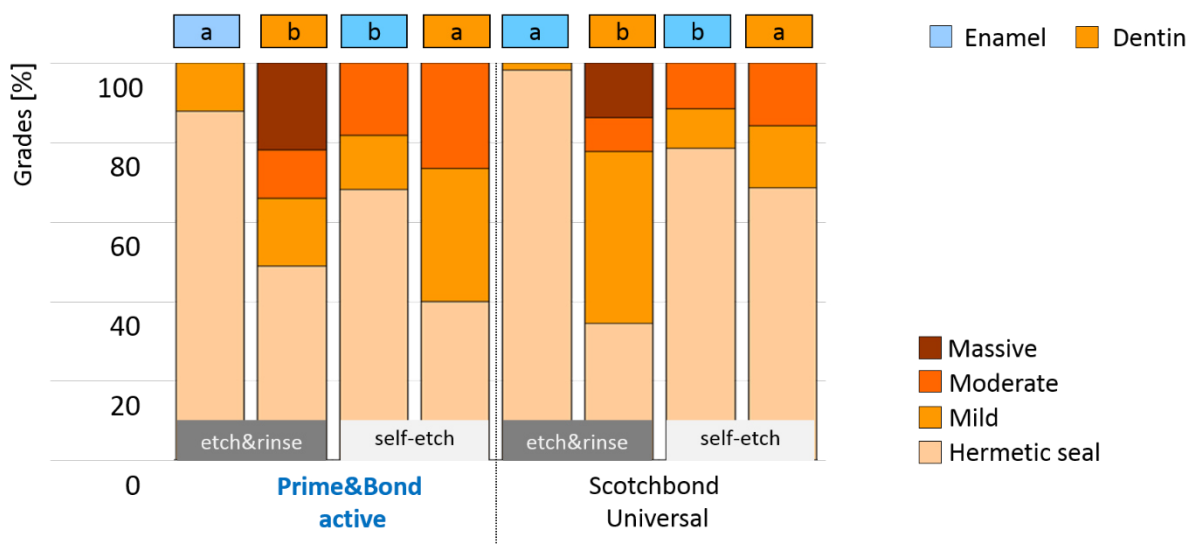


Figure 19 Percentage of dye penetration in Class V restorations after thermocycling. Columns with different letters are significantly different.

Microleakage was not significantly different between the tested adhesives. Both adhesives showed significantly tighter margins in enamel when used in the etch&rinse mode. The self-etch mode, however, resulted in a significantly lower penetration depth in dentin than the etch&rinse mode (Figure 19). Both adhesives showed only slight penetration of the dentinal tubules in the etch&rinse mode (5-10%), which significantly decreased in the self-etch mode (data not shown).

3.3.4 Marginal adaptation of indirect restorations

(Prof. ROLAND FRANKENBERGER, University of Marburg, Germany)

In each group, eight extended MOD cavities (with cervical margins in enamel and dentin) were prepared in extracted human molars. Cerec glass ceramic inlays (Celtra Duo) were hydrofluoric acid etched, silanated, and bonded with different adhesive/luting composite combinations according to the respective DFU. The adhesives were either pre-cured by light before seating the ceramics or applied without any light-curing in a dark cure scenario. Except for the etch&rinse control (Prime&Bond XP combined with SCA and Calibra Automix), all luting groups were used in the selective-etch mode. Specimens were subjected to thermocycling (2500 cycles at 5/55°C) and mechanical loading (TML) in a chewing simulator (1000,000 cycles, 50 N). Marginal quality was analyzed under the SEM at 200x magnification using replicas before and after TML. Statistical analysis was done by non-parametric tests at $p < 0.05$.

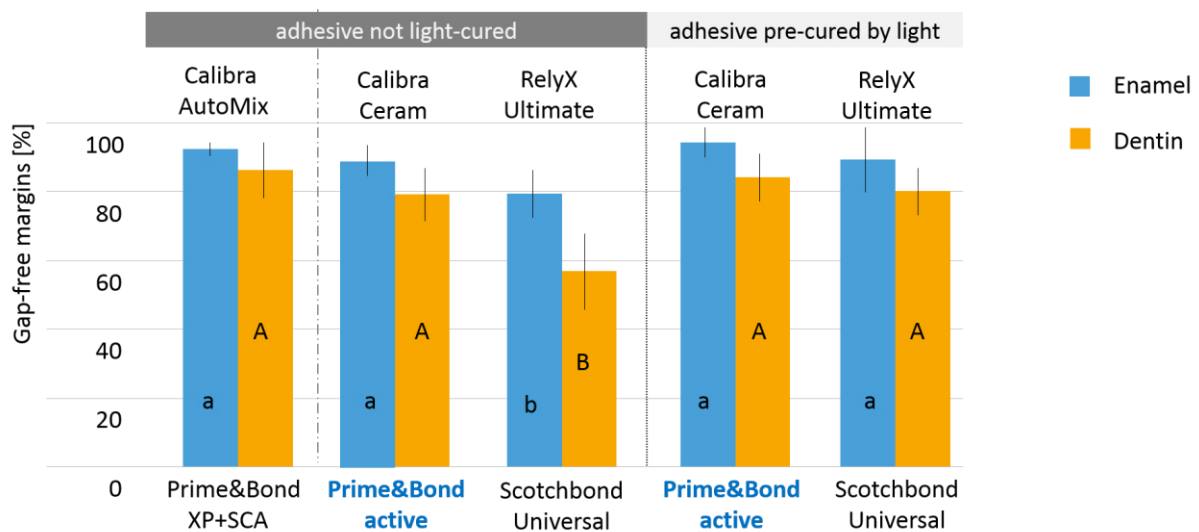


Figure 20 Percentage of gap-free margins of MOD ceramic inlays after TML. Bars with different letters are significantly different. SCA = self-cure activator.

After TML, marginal adaptation significantly decreased for the tested luting systems. When light-curing the adhesives, no significant differences were found between the luting systems. However, in the dark cure scenario, the Scotchbond Universal/RelyX Ultimate system showed a significant decrease of marginal adaptation to enamel and dentin (Figure 20). Although no such differences were found for Prime&Bond active/Calibra Ceram, as a safeguard it is advisable to light-cure this luting system – as indicated in the DFU.

3.4 Adhesive layer thickness and polymerization

Light-curing the adhesive may interfere with the fit of indirect restorations. Although a low film thickness avoid possible misfits of the restoration, it can be questioned whether thin adhesive layers polymerize sufficiently, because of the inhibition layer. Therefore, investigations on the film thickness and its polymerization were conducted.

3.4.1 Film thickness

(Prof. ANDRE REIS, Guarulhos University, Brazil)

The exposed dentin surface of 36 extracted human molars were ground (600 grit) flat in order to standardize the smear layer. After application of the adhesives according to the DFU, the bonded surfaces were covered with a thin layer of SDR composite. Adhesive and composite were light-cured separately. Except for the etch&rinse adhesive Optibond Solo Plus, the adhesives were applied in the self-etch mode. After 24 h of water storage, the specimens were cut into resin-dentin slabs and embedded in epoxy resin. Next, they were dehydrated in ascending concentrations of ethanol and sputter coated. The film thickness of the adhesive layers was measured by means of FE-SEM, while the hybrid layer was not included in the measurements. For each adhesive, five images with three measurements on each image were evaluated using an image analysis software. Results were analyzed by parametric tests at $p < 0.05$.

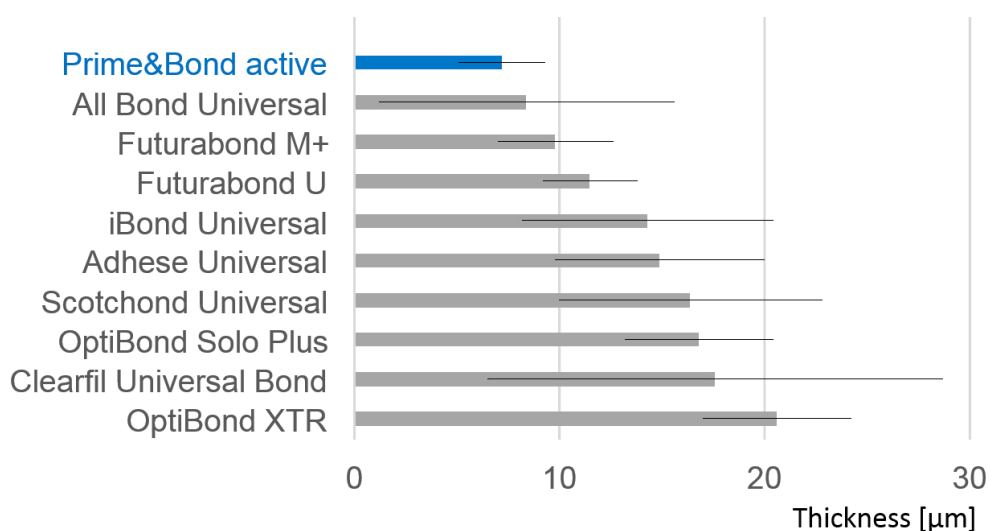


Figure 21 Mean values of film thickness in μm and standard deviation for the tested adhesives.

The results showed that Prime&Bond active provides a lower film thickness than a number of other adhesives enabling separate light-curing of the adhesive layer without possible misfits of the seated restoration (Figure 21).

3.4.2 Conversion degree

(Prof. JACQUES LALEVÉE, Mulhouse Institute, France)

The conversion degree of different universal adhesives was monitored by real-time Fourier transform infrared spectroscopy (RT-FTIR). The method is well established to determine polymerization profiles, i.e. the monomer conversion versus the curing time by the infrared beam of the spectrometer. All adhesives were deposited on a sample surface (barium fluoride pellet) and light-cured under air using a SmartLite Focus LED curing light with 300 mW/cm² irradiance at a distance of 18 mm between the LED and the sample. RT-FTIR was calibrated to an adhesive layer thickness of 10 µm. Each of the adhesives was tested without any additional water and in the presence of 26 wt% and 40 wt% distilled water, respectively.

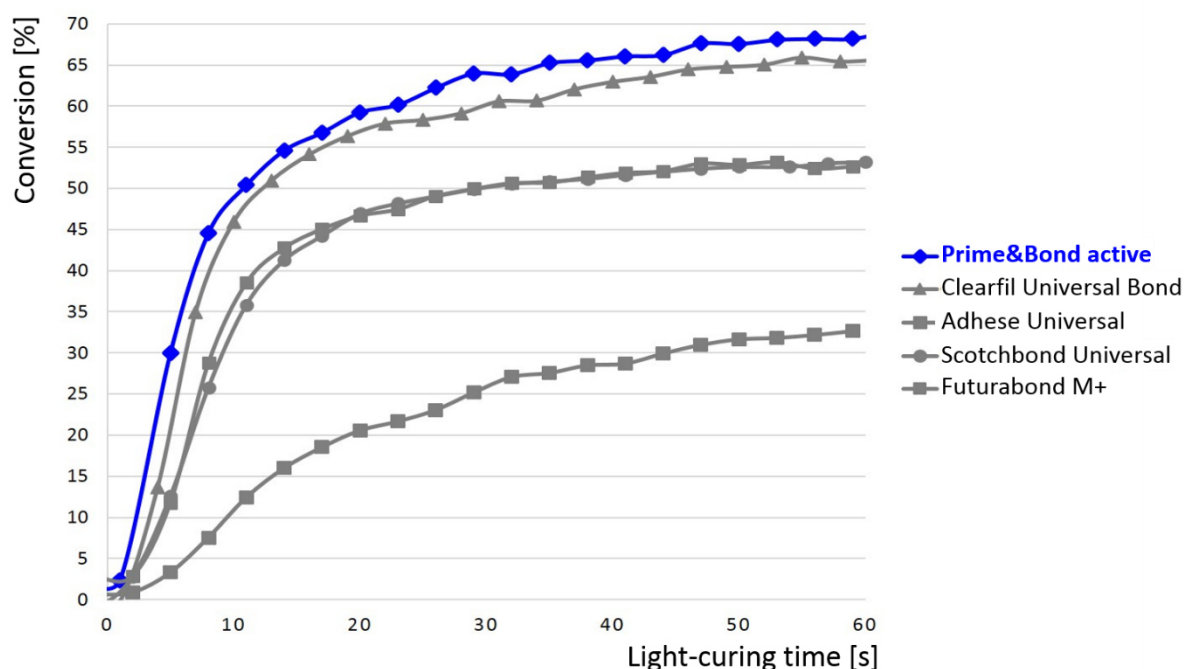


Figure 22 Percentage of conversion as a function of curing time under air for various adhesives. Polymerization profiles are shown without any additional water.

Without any additional water, Prime&Bond active demonstrated a faster and higher degree of conversion than the other adhesives tested (Figure 22).

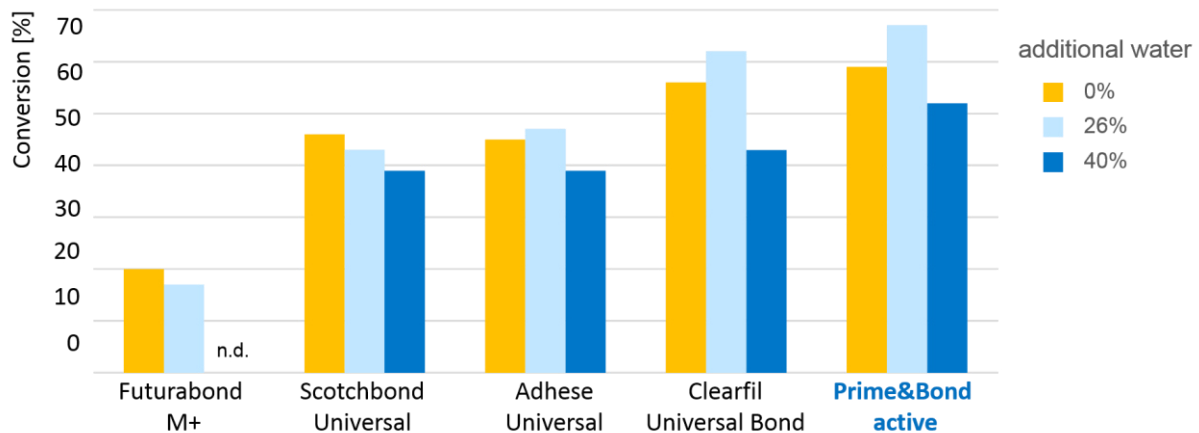


Figure 23 Percentage of conversion as a function of water content after light-curing the adhesives for 20 s under air, n.d. = not determined as no homogeneous mixture with that water content was possible.

Prime&Bond active showed also a high conversion degree in the presence of additional amounts of water (Figure 23). The study showed that the initiators of Prime&Bond active lead to a reliable curing even of thin adhesive layers and even if the cavity is too wet.

3.5 Summary of in vitro studies

Prime&Bond active performed equal or better compared to the controls independent of the etching mode. Furthermore, it showed high robustness towards different degrees of moisture in dentin. Based on the shown in vitro data Prime&Bond active is a universal adhesive providing high performance and technique robustness.

4 Clinical Data

As described in chapter 3 in vitro investigations have proven equal or superior performance regarding bond strength and marginal integrity compared to leading universal adhesives.

Though this high performance in laboratory settings is the fundamental base when developing a new adhesive, it is crucial that the handling of the adhesive is well accepted in a clinical setting, too.

Therefore, two user evaluations were conducted in order to evaluate the handling properties of the adhesive itself and the new Flip-Top-Cap bottle.

4.1 User evaluation on handling properties – adhesive

General dental practitioners (GDPs) were asked to use Prime&Bond active for at least 4 weeks for a minimum of 20 procedures. A total of 79 GDPs participated in this user evaluation and placed more than 3000 restorations (Table 2).

Total	Min	Max	Mean	Median
3176	20	210	40	31

Table 2 Number of restorations placed in 4 weeks by 79 GDPs

As universal adhesive Prime&Bond active may be used in all etching techniques. Figure 24 shows how often each etching technique was used in this user evaluation.

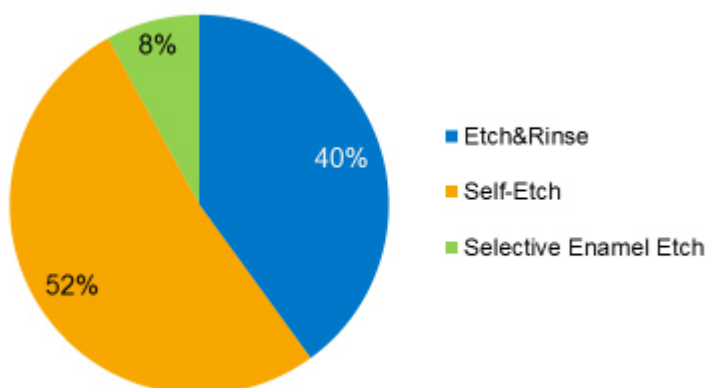


Figure 24 Distribution of applied etching techniques (n=79)

An important parameter while applying an adhesive to the cavity is its viscosity. Though there is no defined optimum the personal preference is important regarding user acceptance. The overall rating of the viscosity is shown in Figure 25.

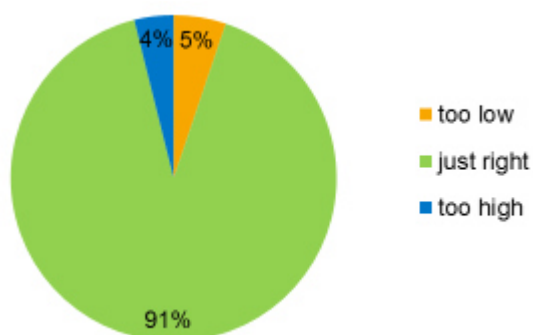


Figure 25 Rating of the viscosity (n=77)

Furthermore, the users were asked to compare the viscosity of Prime&Bond active to their currently most often used adhesive (Figure 26).

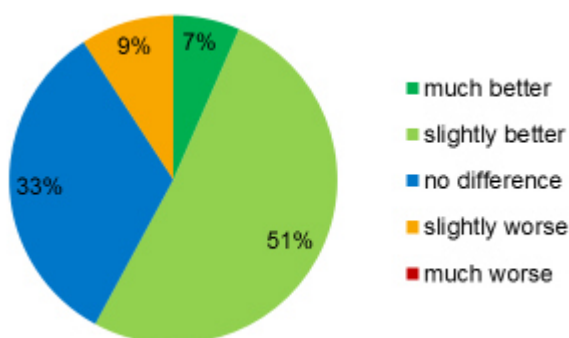


Figure 26 Rating of viscosity compared to currently most often adhesive (n=76)

Finally regarding handling properties, the overall rating is shown in Figure 27.

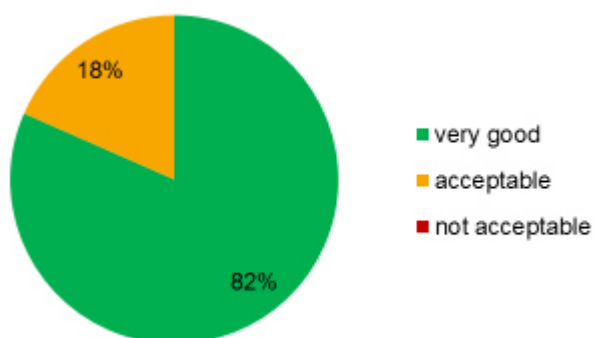


Figure 27 Overall rating of handling properties (n=79)

As described in chapter 3.1.2 to 3.1.4 Prime&Bond active is less sensitive to the degree of moisture in dentin leading to a technique robustness. Figure 28 shows how important such a feature is in the eyes of GDPs.

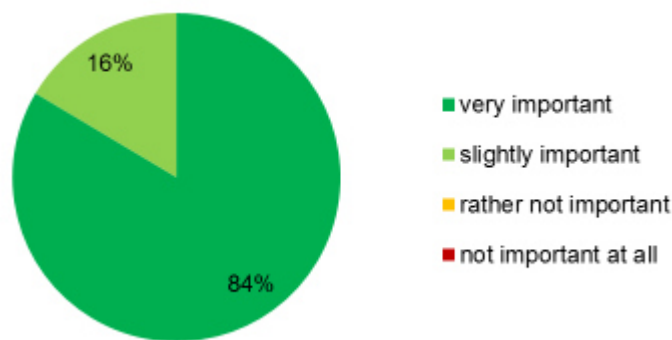


Figure 28 Importance of robustness towards varying degree of dentin moisture (n=79)

4.1.1 Postoperative sensitivity in daily practice

Participating GDPs were asked whether they observed cases of postoperative sensitivity and if so in how many cases this was observed.

Three participating dentists reported together five cases of postoperative sensitivity. Two dentists reported that the observed frequency was equal to what they had observed with their currently used adhesive. The other dentist didn't specify whether the observed incidence was higher or lower.

Overall, this resulted in **less than 0.2% postoperative sensitivity** reported by GDPs from daily practice.

4.2 User evaluation on handling properties – new Flip-Top-Cap bottle

Prime&Bond active is offered in a newly designed bottle with a cap allowing opening and closing in one hand (Figure 29).



Figure 29 Newly developed bottle for adhesives (Flip-Top-Cap bottle)

In addition to the above mentioned user evaluation focusing on the adhesive, a second user evaluation was performed with focus on the Flip-Top-Cap bottle. 30 GDPs participated and were asked to clinically use Prime&Bond active in the new bottle for at least 40 applications on a minimum of 20 patients. Furthermore, additional bottles were provided to test specific aspects of handling the bottle. Table 3 shows the number of clinical procedures.

Total	Mean	Max	Min	Median
1774	59.1	100	40	52

Table 3 Number of clinical procedures using Prime&Bond active in the new bottle (n=30)

To ensure stability of the adhesive after first usage, the proper closing of the bottle to avoid evaporation of volatile components (e.g. solvent) is mandatory. Therefore, the newly designed cap provides tactile and acoustic feedback which was rated good or very good by all participants (Figure 30).

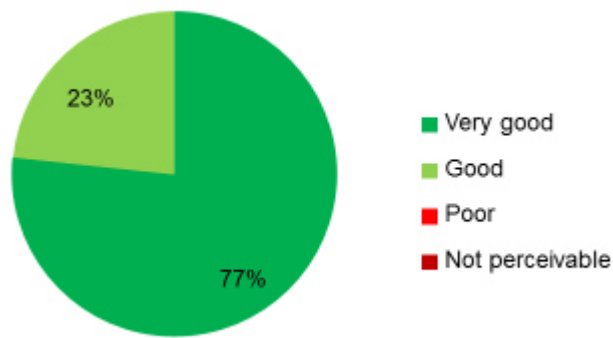


Figure 30 Rating of tactile and acoustic feedback while closing the cap in one hand (n=30)

Finally, the users were asked for rating their personal preference comparing the new bottle of Prime&Bond active with the delivery system of their currently used adhesive. The overall rating of the handling properties was very positive as shown in Figure 31.

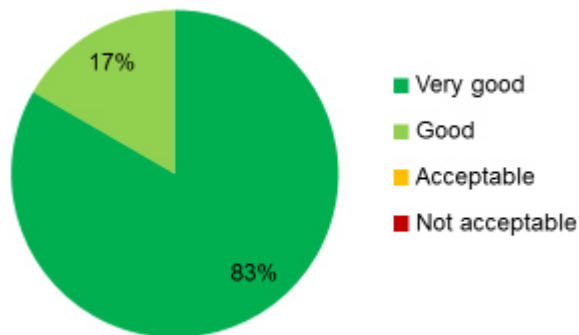


Figure 31 Overall rating of handling properties (n=30)

4.3 Summary of user evaluations

Prime&Bond active was evaluated in daily practice with a total of 4950 restorations being placed by GDPs. Important aspects of this user evaluation were the optimal viscosity of the adhesive, the very low incidence of postoperative sensitivity and the importance that an adhesive shows robustness towards different degrees of moisture in dentin. And finally, the highly rated overall handling of the new Flit-Top-Cap bottle.

5 Instructions for Use

The up-to-date version can be found in all European languages on www.dentsply.eu.

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7 Glossary and Abbreviations

A+B	Binary mixture of components A and B
A+B+C	Ternary mixture of components A and B and C
A-B	Single component with properties of A and B
°C	Degree Celsius, temperature
<i>bis</i> GMA	Bisphenol A diglycidyl methacrylate
DFU	Directions for Use
(D)UDMA	(Di)urethane dimethacrylate
ER	Etch&rinse
FE-SEM	Field emission scanning electron microscopy
GDP	General Dental Practitioner
HEMA	2-hydroxyethyl methacrylate
$\text{kJ}\cdot\text{mol}^{-1}$	Kilojoule per mol, molar energy, reactivity
MDP	10-methacryloyloxydecyl dihydrogen phosphate
$\text{mN}\cdot\text{m}^{-1}$	Milinewton per meter, surface tension
MPa	Megapascal
$\text{Pa}\cdot\text{s}$	Pascal seconds, viscosity
PENTA	Dipentaerythritol pentacrylate phosphate
RT-FTIR	Real-time Fourier transform infrared spectroscopy
SCA	Self-cure activator
SE	Self-etch
SEE	Selective enamel-etch
SEM	Scanning electron microscopy
TC	Thermocycling
TEGDMA	Triethyleneglycol dimethacrylate
TEM	Transmission electron microscopy
TML	Thermomechanical loading
Vol%	Volume percent
Wt%	Weight percent

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Futurabond M+ (VOCO)

Futurabond U (VOCO)

iBond Universal (Heraeus Kulzer)

IPS e.max (Ivoclar Vivadent)

Multilink Automix (Ivoclar Vivadent)

Multilink Primer A/B (Ivoclar Vivadent)

OptiBond Solo Plus (Kerr)

OptiBond XTR (Kerr)

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