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Prevention of erosions by a surface sealant and adhesives under abrasive conditions

KEYWORDS

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SUMMARY

It was the aim of the current in vitro study to test the erosion-preventive potential of an experimental surface sealant (K-0184) and currently marketed self-etch adhesive systems under abrasive conditions. Ninety-six dentine specimens were randomly allocated to eight groups (n = 12): (1) positive control (no treatment), (2) K-0184, (3) Shield Force Plus, (4) Xeno Select, (5) Scotchbond Universal, (6) Adhese Universal, (7) OptiBond All-In-One, and (8) Clearfil SE Bond. Before and after sealing, dentine specimens were measured profilometrically to determine the layer thickness of the protective agents. During 12 days, the specimens were demineralized daily for 5 min using hydrochloric acid (pH = 3) and abraded by applying 600 brushing strokes

(F = 2.5 N). Calcium content in the hydrochloric acid was determined using atomic absorption spectroscopy.

Calcium concentration was consistently highest in the positive control group (p < 0.05 compared to all other groups). The lowest cumulated Ca release was measured for K-0184; it differed significantly (p < 0.05) from that determined in groups 4, 6, and 7. K-0184 also had the highest layer thickness (p < 0.05 compared to all other groups). It was concluded that the experimental surface sealant K-0184 and the adhesive systems Xeno Select, Scotchbond Universal, Adhese Universal, and Clearfil SE Bond prevent dentine erosion under the current abrasive conditions with a total of 7,200 brush strokes.

Introduction

Erosive changes of dental hard tissues have gained attention in dental research and daily clinics (JAEGGI & LUSSI 2014). A literature search by Lussi und Carvalho highlighted the importance of this topic. While a mere ten publications were found for the year 1980 using the search term “tooth erosion”, the year 2012 yielded 100 respective hits (LUSSI & CARVALHO 2014). Non caries-related tooth defects in general gain importance in the scientific literature, which can be appreciated by entering the terms “tooth wear”, “tooth attrition”, or “tooth abrasion” in PubMed.

Dental erosion is defined as a non-carious tooth defect, which occurs without bacterial impact (LUSSI & CARVALHO 2014). Erosion etiology is multifactorial and may be related to either exogenous (food, medicaments) or intrinsic (bulimia, reflux) acids, which dissolve minerals from the dental hard tissues (ZIPKIN & McCLURE 1949). Without preventive or therapeutic measures, dental erosion may lead to untoward long-term effects such as loss in vertical dimension of occlusion and aesthetic problems (LAZARCIK & FILLER 1997). Preventive measures include the local application of fluoride or more novel products containing tin chloride (GANSS ET AL. 2010) or cerium chloride (WEGEHAUPT ET AL. 2010). In severe cases of dental erosion, especially when the underlying causes remain unclear or cannot be treated, a mechanical intervention may be indicated to prevent further loss of dentine (AZZOPARDI ET AL. 2004). Next to time-consuming and costly restorative measures, minimally invasive approaches have emerged over the recent years. These are based on the application of surface sealants or adhesive systems to affected areas (BRUNTON ET AL. 2000). Irrespective of their intended usage, adhesives are categorized into the “etch and rinse” or the “self-etch” type. With etch and rinse adhesive systems, the dental hard tissues are pretreated using an acid. In contrast, the self-etch systems contain acidic monomers, which demineralize and infiltrate dental hard tissues concomitantly (VAN MEERBEEK ET AL. 2011). Because of their milder acidity in comparison to etch and rinse adhesive systems, the exposed collagen network in the dentine does not collapse, allowing for an optimized interaction between dentine and the resins (VAN MEERBEEK ET AL. 2011). In addition, self-etch adhesives cause less postoperative pain in the context of restorative dentistry (VAN MEERBEEK ET AL. 2011). Consequently, self-etch adhesives appear to be ideally suited to protect against dental erosions and abrasions (AZZOPARDI ET AL. 2004; ITOH ET AL. 2010). The surface sealants that were used in various studies (AZZOPARDI ET AL. 2004; WEGEHAUPT ET AL. 2012A; WEGEHAUPT ET AL. 2012B; WEGEHAUPT ET AL. 2013A; WEGEHAUPT ET AL. 2013C) such as Seal&Protect and K-0184 also interact directly with the dental hard tissues without any preconditioning, so that they are comparable to self-etch adhesives in that respect. The experimental surface sealant K-0184 was assessed in former studies regarding its abrasion resistance and biocompatibility (WEGEHAUPT ET AL. 2013B; WEGEHAUPT ET AL. 2014). In a recently published investigation, a surface sealant and two etch and rinse adhesives were studied regarding their resistance to erosion/abrasion (WEGEHAUPT ET AL. 2013C). However, hitherto there is sparse information on the performance of self-etch adhesives under erosive/abrasive conditions.

It was the goal of the current study to test the durability of the antierosive effects of a surface sealant and universally available self-etch adhesives under abrasive conditions. The null hypothesis was that erosive/abrasive conditions would not alter the protective properties of the materials under investigation.

Material and methods

Preparation and erosive pretreatment of dentine specimens

Ninety-six dentine specimens were prepared for the current study from 16 bovine mandibular incisors. Soft tissue remnants were removed using a scalpel subsequent to the postmortem extraction of these teeth. The crowns were removed using a rotary saw (IsoMet® Low Speed Saw, Buehler, Illinois, USA) under water-cooling. Subsequently, 6 cylindrical dentine specimens were prepared from each root using a water-cooled diamond trephine bur. These specimens were embedded in resin (Pal-adur®, Heraeus Kulzer GmbH, Hanau, Deutschland). To remove remnants of this resin and to obtain plain, standardized dentine surfaces, specimens were polished (Planopol-2, Struers GmbH, Birmensdorf, Switzerland) at 150 rotations/min using silica carbide paper (Struers GmbH, Birmensdorf, Switzerland) of decreasing grain size (1,200, 2,500 und 4,000 Grit). Finally, the specimens were randomly assigned to similar groups (1–8; n = 12). A stratification process made sure that there were not two dentine specimens from the same bovine root in any group. The specimens were then de- and remineralized as follows: they were exposed to hydrochloric acid (pH = 3.0) for 6 times 5 min. After each of these acidic exposures, the specimens were washed in tap water to arrest the erosive process. Between acidic exposures (30 min) and overnight, specimens were stored in artificial saliva (KLIMEK ET AL. 1982).

Sealing of dentine specimens

The surfaces of the pretreated dentine specimens were sealed using either surface sealant or self-etch adhesives (Groups 2–8). Group 1 served as positive control, in which no surface protection was applied. Light polymerization was performed using an LED lamp (Bluephase, mode: low, 1,200 mW/cm²; Ivoclar Vivadent, Schaan, Liechtenstein) at a working distance of 0.5 mm.

The specimens in Group 2 were treated with the experimental surface sealant K-0184 (Dentsply DeTrey GmbH, Konstanz, Germany). The sealant was applied to the surface in a first step. It was allowed to infiltrate the dentine for 20 s. Subsequently, it was blown out gently for 5 s and light-polymerized for 10 s. A second layer of the surface sealant was applied by following the steps described for the first layer. This procedure followed the instructions by the manufacturers.

The specimens in Group 3 were sealed using the self-etch adhesive Shield Force Plus (Tokuyama Dental Corporation, Tokyo, Japan). The adhesive was rubbed into the dentine using a microbrush, and then blown out gently for 5 s followed by a stronger blow for 10 s. Subsequently the specimen was light-polymerized for 10 s.

In Group 4, the dentine specimens were sealed using Xeno® Select (Dentsply DeTrey GmbH). This adhesive system can be applied using an etch and rinse protocol or in a self-etch manner. In the current study, we used the self-etch protocol. To this end, the adhesive was rubbed into the dentine for 20 s using a microbrush, then blown out gently for at least 5 s (until no more movement of the adhesive layer was detectable), and finally light-polymerized for 10 s.

The specimens in Group 5 were treated using Scotchbond™ Universal (3M ESPE, Neuss, Germany). In a first step, the adhesive was rubbed into the dentine for 20 s and then blown out gently for 5 s. Subsequently, the material was light-polymerized for 10 s.

In Group 6, Adhese® Universal (Ivoclar Vivadent, Schaan, Liechtenstein) was used to seal the dentine specimens. It was applied following a similar protocol as described for the other self-etch adhesives under investigation: 20 s of application, blowing out gently using compressed air, light-polymerization for 10 s.

As requested by the manufacturer, the specimens in Group 7 were treated twice with the OptiBond™ All-In-One (Kerr Dental, Scafati, Italy) adhesive. Again, the adhesive was applied for 20 s, blown out gently, and polymerized for 10 s. Subsequently, the second layer was applied according to the application of the first layer.

Specimens in Group 8 were treated using the 2-bottle system Clearfil™ SE Bond (Kuraray, Okayama, Japan). The primer was applied for 20 s to the dentine and then blown out as described for the other adhesive systems under investigation. The adhesive was applied subsequently and light-polymerized for 10 s.

The chemical composition of the seven materials used to seal the dentine in the current study is summarized in Table I.

Tab. I Composition of the sealant/adhesives (as provided by manufacturers)

Sealant/adhesive	Contents
K-0184	UDMA, trimethacrylate, PENTA, highly dispersed silica, camphorquinone, ethyl-4(dimethylamino)benzoate, BHT, cetylaminhydrofluoride, acetone
Shield Force Plus	2,6-di-tert.butyl-4-methylphenol, 2-HEMA, Bis-GMA, diphenyl-(2,4,6-trimethylbenzoyl)-phosphin oxide, mequinol, methacryloxyalicylic acid phosphate, propan-2-ol, TEGDMA, camphorquinone, water
Xeno® Select	Bifunctional acrylates, acidic acrylates, phosphoric acid ester, water, T-butanol, initiators, stabilizers
Scotchbond™ Universal	10-MDP, dimethacrylate resins, HEMA, Vitrebond™ copolymer, fillers, ethanol, water, silan, initiators
Adhese® Universal	2-HEMA, Bis-GMA, methacrylate phosphoric acid ester, 2-dimethyl-aminoethylmethacrylate, camphorquinone, ethanol, water
OptiBond™ All-In-One	HEMA, ethanol, disodium hexafluorsilicate, acetone, water
Clearfil™ SE Bond	Primer: 10-MDP, 2-HEMA, hydrophilic aliphatic dimethylacrylate, camphorquinone, N,N-diethanol-p-toluidine, water Bonding: 10-MDP, 2-HEMA, Bis-GMA, hydrophobic aliphatic dimethylacrylate, camphorquinone, N,N-diethanol-p-toluidine, colloidal silica
Abbreviations: UDMA = urethandimethacrylate, PENTA = phosphoric acid-modified acrylic resin, BHT = butylhydroxytoluol, HEMA = 2-hydroxyethylmethacrylate, Bis-GMA = bisphenol-A-diglycidylmethacrylate, TEGDMA = triethyleneglycoldimethacrylate, 10-MDP = 10-methacryloyloxydecyl dihydrogenphosphate, Vitrebond™ Copolymer = methacrylate-modified polyalkanoate acid copolymer	

Determination of material thickness

To determine the thickness of the applied material layers, an initial measurement before their application was performed using a Stylus profilometer (MAHR Oberflächenmessgerät, Mahr GmbH, Göttingen, Deutschland). To this end, 5 profiles were recorded per specimen in a distance of 250 µm from each other. To prevent possible shrinkage of the dentine specimens during profilometric measurements, they were dried in ambient air for 10 min prior to the profilometry procedure. In addition, the resin margins in each specimen were covered using adhesive tape (Scotch® Magic™, 3M AG, Rüschlikon, Switzerland) to be able to reposition each specimen for the measurement and to superimpose the resulting profiles (ATTIN ET AL. 2009). The initial measurement was performed after demineralization to determine the thickness of the adhesive layer.

Erosive/abrasive treatment of the specimens

Specimens underwent an erosive/abrasive treatment daily for 12 days as follows: an erosive challenge was simulated by immersing the specimens in 2.5 ml of hydrochloric acid (HCl, pH 3.0) each under constant agitation (IKA-Vibrax-VXR; IKA®-Werke GmbH & Co. KG, Staufen, Germany). Subsequently, specimens were washed in distilled water and brushed for 5 min in an automated tooth-brushing device at 120 strokes per min and a load of 2.5 N. The toothbrush that was used was a ParoM43 (Esro AG, Thalwil, Switzerland) with a 0.2 mm filament. The slurry that was used for simulated tooth brushing had a standardized abrasiveness (RDA 100). It was prepared according to Imfeld's instructions (IMFELD 2010) from 1 part of Sident 9 (Dr. Storeck, Hanau, Germany) and 5 parts of natrosol-glycerin mixture (wt:wt). The specimens were stored overnight in artificial saliva (KLIMEK ET AL. 1982), and the procedure was repeated the next day (total of 12 times).

Determination of the antierosive effect

The amount of calcium (Ca) that was dissolved during the erosive challenge was determined using atomic absorption spectroscopy (AAS, contrAA®300; Analytik Jena AG, Jena, Germany). In the context of the current study, a low amount of Ca was related to a high antierosive effect. To determine the Ca in the HCl solution that was used to erode the dentine, 2 ml of the HCl was mixed with 2 ml of strontium chloride (SrCl₂) and 2 ml of deionized water. The SrCl₂ was added to this mixture to mask the phosphate that was dissolved in the acid. Measurements were performed at a wavelength of 422 nm (WEGEHAUPT ET AL. 2012A).

Statistics

Data was coded in Excel and analysed using SPSS Version 22. Data distribution was assessed by Kolmogorov-Smirnov test. Data was skewed, and non-parametric methods were thus applied. With respect to descriptive statistics, median and interquartile ranges (IQR) are presented for all data sets from all the experimental time points. For the continuous variables (amount of Ca from day 1 until day 12, cumulated amount of Ca, and material thickness), differences between groups were searched by Kruskal-Wallis test followed by Mann-Whitney U test for individual comparisons. Bonferroni's method was applied to correct for multiple *post hoc* testing. Within groups, Ca release on days 2 to 12 was compared to counterparts measured at day 1 using Wilcoxon test for paired samples. A significant increase in Ca was interpreted as a significant loss in the antierosive

sive effect of the materials under investigation. The alpha-type error was set at 5% ($p < 0.05$).

Results

Material thickness

Layer thickness of the 7 materials under investigation is displayed in Figure 1 (medians, IQR). K-0184 had the highest thickness (median = 48.6 μm , IQR = 25.9 μm ; $p < 0.05$ compared to all other groups). The significantly ($p < 0.05$) least material thickness was jointly observed for Shield Force Plus (median = 13.2 μm , IQR = 6.0 μm), Xeno[®] Select (median = 11.0 μm , IQR = 2.6 μm) and OptiBond[™] All-In-One (Median = 10.9 μm , IQR = 9.3 μm).

Antierosive effect

Calcium release during the erosive treatment on days 1 to 12 as well as cumulative Ca values per material/group are listed in Table II. On all experimental days, the significantly highest Ca release was observed in the positive (unsealed) control group ($p < 0.05$). On all days Ca release differed between Group 2 (K-0184) and OptiBond[™] All-In-One, and from day 3, also between K-0184 and Shield Force Plus ($p < 0.05$).

Shield Force Plus coverage of the dentine (Group 3) resulted in Ca values which, with the exception of days 1 and 2, differed significantly from those observed with K-0184 and Scotchbond[™] Universal, as well as Adhese[®] Universal (except days 2 and 5), and Clearfil[™] SE Bond (days 2, 5 and 6).

A significant ($p < 0.05$) difference was observed between Scotchbond[™] Universal and Shield Force Plus from day 3 onwards. In comparison to OptiBond[™] All-In-One with the exception of days 1 and 5 Scotchbond[™] Universal caused a significantly lower ($p < 0.05$) Ca release. The measured Ca release with Clearfil[™] SE Bond differed significantly from that with Shield Force Plus (exception days 2, 5 and 6) and OptiBond[™] All-In-One (except days 1, 2, 5 and 6).

The cumulated Ca releases between Groups 2, 4, 5 and 8 (K-0184, Xeno[®] Select, Scotchbond[™] Universal and Clearfil[™] SE Bond) did not differ at the 5% level from each other. The respective values measured in Groups 3 (Shield Force Plus) and 7 (OptiBond[™] All-In-One) were significantly higher than the cumulative Ca release in Groups 2, 4, 5, and 8, and did not differ significantly from each other ($p > 0.05$). Again the significantly highest cumulative Ca release was observed in the positive control group (Group 1, $p < 0.05$ compared to all other groups).

In Group 3 (Shield Force Plus) the daily Ca release increased significantly ($p < 0.05$) from experimental day 4 compared to day 1. For OptiBond[™] All-In-One (Group 7) a compared to day 1 significantly higher Ca release was observed from experimental day 10. With the other groups, no significant increase or even a slight decrease was observed when Ca release on days 2 to 12 was compared to that on day 1.

Discussion

The null hypothesis that erosive/abrasive conditions would not alter the protective properties of the materials under investigation was rejected.

In the present study, dentine specimens were prepared from bovine teeth. Several studies have used such specimens to measure loss of dental hard tissues under erosive and abrasive conditions (IMFELD 2001; DE MENEZES ET AL. 2004; MAGALHAES ET AL. 2009; STEINER-OLIVEIRA ET AL. 2010; WEGEHAUPT ET AL. 2013C). This type of specimens offers several advantages, such as ease in collecting the respective teeth and the fact that several specimens can be obtained from one tooth (WEGEHAUPT ET AL. 2008; SHELLIS ET AL. 2011). In addition, bovine teeth can be collected from animals that are held under similar conditions (WEGEHAUPT ET AL. 2012A). These facts result in homogenous specimens and high comparability of results (WIEGAND & ATTIN 2011). Last but not least bovine dentine behaves similarly to human dentine under erosive and erosive/abrasive conditions (WEGEHAUPT ET AL. 2008).

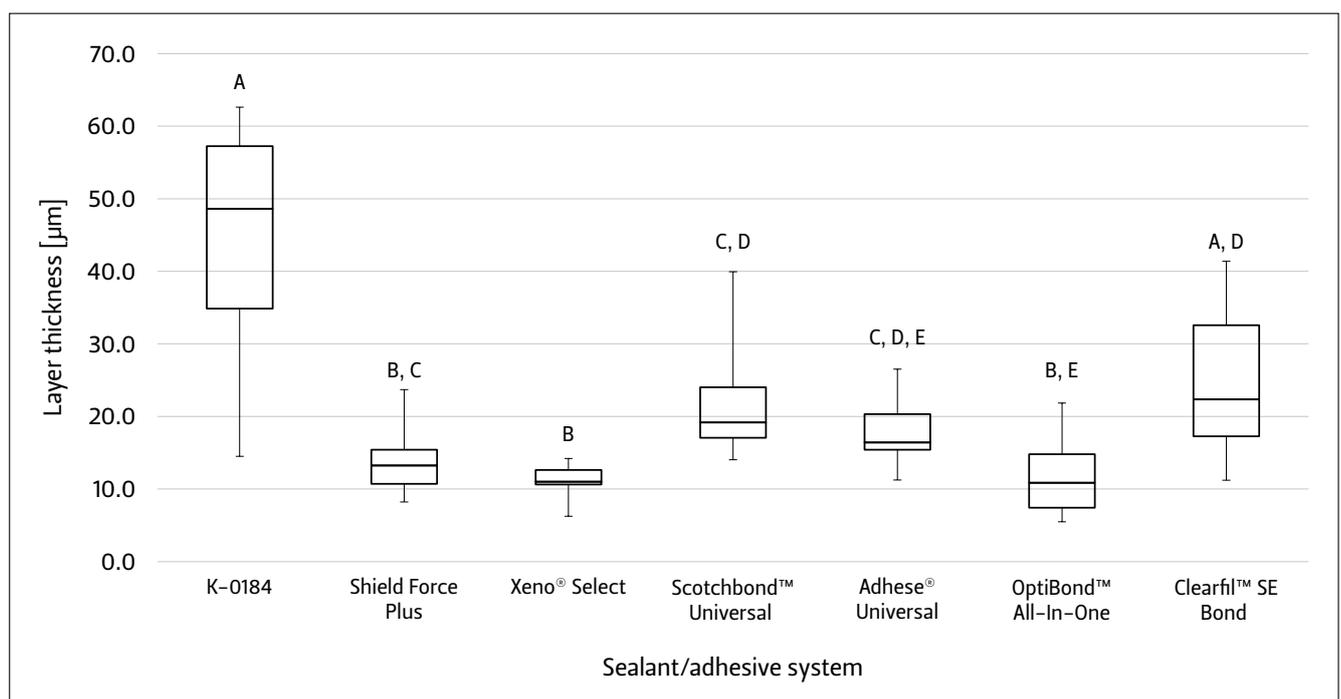


Fig. 1 Layer thickness (median and interquartile range, IQR = whiskers) of the surface sealant and the self-etch adhesives under investigation. Values which did not differ at the 5% level from each other, are designated with the same letter.

Tab. II Calcium concentrations ($\mu\text{g Ca/ml}$, median [interquartile range]) on days 1–12 and cumulated (ctd) of the 8 groups

Days	Group 1: Control	Group 2: K-0184	Group 3: Shield Force Plus	Group 4: Xeno Select	Group 5: Scotchbond Universal	Group 6: Adhese Universal	Group 7: OptiBond All-In-One	Group 8: Clearfil SE Bond
1	1.70 (0.56) A	0.26 (0.21) B, C	0.14 (0.11) C	0.19 (0.09) B, C	0.20 (0.19) B, C	0.23 (0.15) B	0.31 (0.17) B	0.36 (0.24) B
2	0.89 (0.67) A*	0.10 (0.05) B*	0.14 (0.04) B	0.14 (0.03) B, C*	0.13 (0.06) B*	0.14 (0.05) B*	0.23 (0.16) C	0.18 (0.11) B, C*
3	1.37 (0.86) A*	0.06 (0.03) B*	0.18 (0.04) C	0.11 (0.05) C*	0.07 (0.07) B*	0.10 (0.08) B*	0.27 (0.21) C	0.10 (0.08) B*
4	1.63 (0.62) A	0.08 (0.04) B*	0.21 (0.07) C*	0.13 (0.07) B, C, D*	0.08 (0.07) B, D*	0.12 (0.05) D*	0.31 (0.33) C	0.10 (0.08) B, D*
5	1.25 (0.48) A*	0.05 (0.05) B*	0.20 (0.15) C*	0.12 (0.04) B, D*	0.08 (0.07) B, D	0.16 (0.10) B, C	0.29 (0.28) C, D	0.13 (0.19) B, C
6	1.13 (0.30) A*	0.02 (0.04) B*	0.27 (0.14) C*	0.06 (0.03) B*	0.04 (0.08) B*	0.08 (0.05) B, D*	0.38 (0.36) C, D	0.05 (0.19) B, C*
7	1.12 (0.28) A*	0.01 (0.04) B*	0.23 (0.22) C*	0.09 (0.08) B*	0.05 (0.13) B*	0.12 (0.09) B*	0.55 (0.51) C	0.06 (0.07) B*
8	1.15 (0.45) A*	0.00 (0.04) B*	0.24 (0.36) C*	0.04 (0.05) B, D*	0.00 (0.04) B, D*	0.07 (0.05) D, E*	0.44 (0.55) C, E	0.01 (0.09) B, D*
9	1.09 (0.26) A*	0.05 (0.04) B*	0.29 (0.23) C*	0.09 (0.04) B, D*	0.07 (0.10) B, D*	0.13 (0.07) D*	0.58 (0.56) C	0.08 (0.10) B, D*
10	1.18 (0.41) A*	0.06 (0.04) B*	0.42 (0.29) C*	0.11 (0.07) B*	0.07 (0.08) B*	0.16 (0.09) B, D	0.61 (0.55) C, D*	0.11 (0.18) B*
11	1.40 (0.57) A	0.09 (0.19) B	0.53 (0.38) C*	0.11 (0.03) B*	0.12 (0.22) B	0.21 (0.11) B	0.70 (0.66) C*	0.10 (0.25) B*
12	1.32 (0.47) A	0.07 (0.07) B, D*	0.49 (0.51) C*	0.10 (0.04) B*	0.13 (0.16) B, D	0.18 (0.10) D	0.83 (0.70) C*	0.11 (0.23) B, D*
ctd	15.68 (2.95) A	0.90 (0.55) B	3.19 (2.27) C, E	1.30 (0.65) B, D	1.10 (0.80) B, D	1.71 (0.86) D	5.60 (4.80) C	1.76 (1.01) B, D, E

Within each day and cumulatively (horizontal reading), identical letters indicate that there was no statistical difference at the 5% probability level between values. Within a specific group (vertical reading), the Ca values (from day 2 to 12) that did not differ significantly from those measured on day one are marked with an asterisk (*).

The erosive challenge in the current study was simulated using hydrochloric acid (HCl). HCl is the main component of gastric acid and is commonly used to simulate intrinsic dental erosions caused by reflux or eating disorders (SCHLUETER ET AL. 2007; AUSTIN ET AL. 2011; WEGEHAUPT ET AL. 2013C). Next to hydrochloric acid, gastric juice contains proteolytic enzymes such as pepsin or trypsin (HUNT 1951). It is discussed controversially whether proteolytic enzymes contribute to the degradation of the organic dentine matrix and thus promote the progression of erosive lesions (SCHLUETER ET AL. 2007; SCHLUETER ET AL. 2010; BUZALAF ET AL. 2014; GANSS ET AL. 2014). Because of this controversy and also because a lot of comparable studies used pure hydrochloric acid (WIEGAND ET AL. 2007; GANSS ET AL. 2009A; WEGEHAUPT & ATTIN 2010; AUSTIN ET AL. 2011; DE-MELO ET AL. 2011; WEGEHAUPT ET AL. 2012B; WEGEHAUPT ET AL. 2013C), pure HCl was used in the current investigation.

The current in vitro study was performed during 12 days. One day of the in vitro challenge should reflect approximately 30 days (1 month) in vivo. This is based on the assumption by Wiegand and Attin (WIEGAND & ATTIN 2011), who stated that each

tooth receives between 10 and 15 brushing strokes during a normal personal dental care procedure. The majority of people brush their teeth twice a day (GANSS ET AL. 2009B) and consequently, a tooth receives between 600 and 900 brushing strokes per month, which correlates to a daily abrasion of 5 min under current in vitro conditions at 120 brushing strokes/min. However, it needs to be cautioned that the current daily erosive challenges did not reflect the counterparts that may occur in situ. Here, a considerably higher exposure time should have been applied to concur with a real-life situation. However, it was the primary goal of the current study to assess the protection against erosive challenges under abrasive conditions. Hence the erosive challenge was merely used to trigger a Ca release to indirectly measure this protection by the different materials under investigation.

The dentine specimens were treated using a fluoride-free slurry paste with an RDA value of 100 to test the surface sealant and self-etch adhesives under extreme conditions and, concomitantly, to exclude any modifying effects by the fluoride. Fluorides in toothpastes protect against dental hard tissue loss

in vitro as well as under in situ conditions (WIEGAND & ATTIN 2011). It is therefore conceivable that the use of fluoridated toothpaste in the current study would have altered Ca release, which can be seen as a limitation of this investigation.

The current methodology (Ca analysis by means of AAS) is considered to be suitable for the detection of erosive tooth wear (ATTIN & WEGEHAUPT 2014). A proper measurement of dentine loss can be assessed only if the organic dentine matrix is removed prior to the experiment using e.g. collagenases (GANSS ET AL. 2007). This is because the profilometry indenter is disturbed by exposed collagen, which leads to false measurements. Conversely, profilometry is a useful method to determine the layer thickness of applied sealants/adhesives (WEGEHAUPT ET AL. 2013B). In the current study the dentine specimens were dried for 10 min prior to profilometric measurements to prevent alterations in results caused by shrinkage of the dentine (ATTIN & WEGEHAUPT 2014). This is based on observations by Ganss and co-workers (GANSS ET AL. 2007). These authors showed that alterations in dentine can be detected during the first 10 min, and that these alterations are reversed by rewetting (for 30 s) of the dentine. Consequently, it is to be accepted that profilometry is sufficiently precise to measure layer thickness of the materials under investigation (ATTIN ET AL. 2009).

Ca release in the presence of the experimental surface sealant (K-0184) or the self-etch adhesive systems under investigation was significantly lower during each of the 12 experimental days than the corresponding release observed in the unsealed positive control group. Various studies (BRUNTON ET AL. 2000; WEGEHAUPT ET AL. 2012B; WEGEHAUPT ET AL. 2013C; BUZALAF ET AL. 2014) have shown that covering the dentine surface using an adhesive material reduces erosive wear. However, the materials under investigation differed regarding their resistance to erosive and abrasive challenges. Calcium release in the specimens covered with Shield Force Plus (Group 3) and with Opti-Bond™ All-In-One (Group 7) increased over the whole duration of the study, and was significantly higher than the values of the other treatment groups on days 7, 9, 11 und 12. With regard to cumulative Ca release, OptiBond™ All-In-One showed the worst protection, followed by Shield Force Plus.

A study by Walter and co-workers (WALTER ET AL. 2012) assessed adhesion of OptiBond™ All-In-One over the course of 2 years. The loss of adhesion is explained by the hydrolytic degradation of the hybrid layer, i.e. the connection between the dentine and the adhesive. This loss is mainly influenced by the chemical composition of the adhesives rather than by their mode of application (WALTER ET AL. 2012). Self-etch adhesives are hydrophilic (ITOH ET AL. 2010; FELIZARDO ET AL. 2011). The more hydrophilic an adhesive, the higher its water uptake into the resin matrix (ITOH ET AL. 2010). Consequently, even after polymerization, monomers act as a semi-permeable membrane, which can hurt the bond between dentine and adhesive and lead to nanoleakage and phase separation (ITOH ET AL. 2010; SILVA E SOUZA ET AL. 2010). In this context the monomer 2-hydroxyethyl methacrylate (HEMA), which is responsible for the hydrophilicity of some self-etch adhesive systems, is also considered to be involved with their loss of adhesion over time (FELIZARDO ET AL. 2011). This assumption correlated with the observations of the current study. The HEMA-containing Shield Force Plus and OptiBond™ All-In-One showed the lowest protection against Ca loss, whilst the HEMA-free materials K-0184, Xeno® Select und Scotchbond™ Universal protected better against the same. However, to assess the inferior performance

of Shield Force Plus and OptiBond™ All-In-One under current conditions, further studies regarding their chemical composition would be desirable.

There is plenty of literature available regarding the self-etch adhesive system Clearfil™ SE Bond. Multiple studies have used this material as a control because of its well-known resistance (WALTER ET AL. 2012). Accordingly, the current study found good results for Clearfil™ SE Bond. One explanation for its resistance could be its content of certain functional monomers. Because of their mild pH, self-etch adhesives maintain some hydroxyl apatite crystals around the collagen fibres during the demineralization process (SILVA E SOUZA ET AL. 2010). Some studies showed that the functional monomers 10-methacryloxydecyl dihydrogen phosphate (10-MDP), 4-methacryloxyethyl trimellitic acid (4-MET) and 2-methacryloxyethyl phenylhydrogen phosphate (Phenyl-P) form strong bonds with the hydroxyl apatite in dentine, and thus prevent degradation of the hybrid layer (INOUE ET AL. 2005; SILVA E SOUZA ET AL. 2010; WALTER ET AL. 2012). Two of the self-etch adhesive systems tested in the current study, namely Clearfil™ SE Bond and Scotchbond™ Universal, contain 10-MDP. Neither of them showed a significant Ca loss in the course of the experiment. It is conceivable that the chemical interaction between the dentine and the adhesive system is responsible for the bond that is formed, and that this bond is thus a function of the chemical composition of the adhesive.

The groups K-0184, Xeno® Select, Scotchbond™ Universal, Adhese® Universal and Clearfil™ SE Bond showed a higher Ca loss on experimental day 1 compared to days 2 to 12. However, it would be wrong to conclude that protection against Ca loss increased under the erosive/abrasive conditions of the experiment. An increased initial Ca loss was also observed in an earlier study by Wegehaupt and co-workers (WEGEHAUPT ET AL. 2013C). This loss is explained by the inherent properties of the surface sealant and the adhesive systems under investigation. When these materials are applied to the dentine, minerals are dissolved from the inorganic aspect of the dentine, which are then incorporated into the material itself. In addition, an oxygen inhibition zone is created when these resin-based materials are polymerized. This zone contains Ca, which is released during tooth brushing. The highest such difference in Ca release was observed for K-0184 between experimental days 1 and 2. This is in line with the results reported by Wegehaupt and co-workers (WEGEHAUPT ET AL. 2013C). It is to be concluded that the Ca on day 1 stems from the oxygen inhibition zone and the underlying resin layer, and not necessarily from the dentine.

The average layer thickness of the tested adhesive materials was approximately 20 µm. These values are comparable to those published by a different workgroup (BRUNTON ET AL. 2000), who measured 28 µm on average. However, layer thickness varied considerably between groups and also within one of the experimental groups in the current study. The surface sealant K-0184 was, according to the manufacturer guidelines, applied in two increments. It had a total layer thickness that was roughly twice of that observed with the self-etch adhesive systems under investigation. However, OptiBond™ All-In-One and Clearfil™ SE Bond are also applied twice, yet their layer thickness was considerably smaller than that of K-0184. A possible explanation for this could again be the chemical composition of the adhesive systems in comparison to the surface sealant under investigation. In a recent study (SILVA E SOUZA ET AL. 2010) it was shown that the solvent that is contained in an adhesive is a decisive factor for their layer thickness. An adhesive containing tertiary

butanol (T butanol) showed a significantly lower layer thickness compared to other adhesive systems which contained a water-ethanol mixture (SILVA E SOUZA ET AL. 2010). This was corroborated by the current study: the lowest layer thickness was measured for the self-etch adhesive system Xeno® Select, which also contains T butanol.

Furthermore, it is to be noted that layer thickness of a material and its protective effect against erosion/abrasion did not correlate. In spite of their low thickness of less than 20 µm, with the self-etch adhesive systems Xeno® Select, Scotchbond™ Universal and Adhese® Universal Ca release remained low throughout the 12 days of the experiment. Consequently, the mere quantification of layer thickness of a dentine sealant has no relevance in terms of the protective effect of the same material under current conditions. However, the correlation between layer thickness and protective effect appears to increase with increasing treatment cycles. In a recent publication by Zhao and co-workers (ZHAO ET AL. 2016), the resistance of a dentine sealant and a flowable composite was tested during 24 erosion/abrasion cycles (60 min of erosion and 600 BS per cycle). During the whole duration of the study, the flowable composite, which had a layer thickness of 151 µm, prevented dentine loss, whilst the sealant at a layer thickness of 43 µm started to lose its protective effect after 18 cycles. In a clinical study on the erosion/abrasion protection by sealants (SUNDARAM ET AL. 2007) it was also shown that Seal&Protect lost its protective properties after 6 to 9 months, so that a resealing procedure had to be performed.

In view of the results and the limitations of the current study, it may be stated that the surface sealant K-0184 and four of the self-etch adhesive systems under investigation (Xeno® Select, Scotchbond™ Universal, Adhese® Universal, and Clearfil™ SE Bond) protect against erosive dentine loss over a simulated time period of 12 months. The application of this sealant or these self-etch adhesive systems to exposed dentine as a minimally invasive measure against intrinsic acid attacks appears to be a valid method to protect against erosive dentine loss even under abrasive conditions.

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Résumé

L'objet de cette étude était de tester la résistance des produits de scellement et des systèmes adhésifs courants pour la prévention de la perte érosive de dentine sous des conditions abrasives.

96 échantillons de dentine ont été randomisés en huit groupes expérimentaux (1-8; n=12): (1) témoin (pas de revêtement), (2) K-0184, (3) Shield Force Plus, (4) Xeno Select, (5) Scotchbond Universal, (6) Adhese Universal, (7) OptiBond All-In-One et (8) Clearfil SE Bond. Avant et après le scellement de la dentine, une mesure profilométrique a été effectuée afin de déterminer l'épaisseur de couche des produits appliqués. Pendant les douze jours suivants, les échantillons ont été déminéralisés pendant 5 min par jour dans de l'acide chlorhydrique (pH 3) et brossés avec 600 coups de brosses (F = 2,5 N). Dans l'acide chlorhydrique utilisé, la quantité de calcium dissous a été mesurée par spectroscopie d'absorption atomique.

La concentration de calcium dans le groupe témoin (sans revêtement) a été à tous les points de mesure (jour 1-12 et cumulatifs) statistiquement de manière significative la plus élevée. La libération cumulative de calcium la plus faible a été observée pour K-0184, ce qui était significativement différent des groupes 4, 6 et 7. L'épaisseur de couche d'adhésif la plus élevée a été observée de manière significative également pour K-0184.

Le produit de scellement K-0184 et les systèmes adhésifs Xeno Select, Scotchbond Universal, Adhese Universal et Clearfil SE Bond sont en mesure de réduire de manière significative une perte de dentine érosive dans les conditions abrasives choisies sur un total de 7200 coups de brosses.

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