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Wear Resistance and Surface Roughness of a Newly Devised Adhesive Patch for Sealing Smooth Enamel Surfaces

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Clinical Relevance

The adhesive patch under investigation showed good wear resistance and clinically tolerable surface roughness values following chemomechanical exposure; therefore, it appeared suitable as a smooth enamel sealant.

SUMMARY

A laboratory study assessed the wear resistance and surface roughness after chemical and mechanical wear of a newly devised adhesive patch when used as a smooth surface sealant.

Forty-eight enamel discs were prepared from bovine lower central incisors. Sixteen specimens were treated with one of two sealing options: the prototype of an adhesive patch or a flowable resin. Unsealed enamel served as the positive control. Wear and surface roughness was measured at baseline and after all the samples were immersed in saliva or lactic acid (n=8 per treatment group) for up to 21 days, during which the experimental and control enamel surfaces were exposed to 10 double-stroke toothbrush cycles per day.

In saliva and lactic acid, the sealed specimens showed no significant wear during the observation period (p=0.1841). Only untreated specimens exposed to lactic acid showed a significant substance loss after 14 and 21 days (p=0.0186). The patch and flowable resin showed no differences in surface roughness values at respective times (p=0.385); whereas the surface roughness of the unsealed specimens in lactic acid was significantly higher (p<0.0001).

It was concluded that the adhesive patch under investigation merits further study to assess its potential as a sealant for smooth enamel surfaces.

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INTRODUCTION

Pit and fissure sealing is considered an effective procedure for caries prevention (Horowitz, Heifetz & Poulsen, 1977). Based on good clinical results with fissure sealing, approaches have been taken that extend this preventive concept to smooth enamel surfaces (Hicks & Silverstone, 1982; Schmidlin & Besek, 2003). However, abrasive wear resistance and protection against organic acids of smooth surface sealants that are to be used clinically need to be addressed first in an *in vitro* environment. A smooth surface sealant should provide adequate resistance against chemomechanical challenges to enable optimal enamel protection. Apparently, unfilled resins do not perfectly fulfill these demands, while filled flowable resin composites do (Schmidlin & others, 2002). Enamel specimens sealed either with a single or double application of an unfilled resin showed high leakage in lactic acid. Using scanning electron microscopy (SEM), these specimens revealed clearly visible localized disintegration of sealed areas following acid attack. Attempts to seal dentin with unfilled resins to prevent erosion and abrasion have shown some protection of the teeth in a reciprocating erosion/abrasion machine compared to unprotected dentin, but it was also evident that the resin layer broke down partially as specimens were subjected to an acidic environment and toothbrushing (Azzopardi & others, 2001). These results elucidate the need for establishing a wear-resistant sealant to enable a predictably resistant and stable sealant layer. Furthermore, a smooth surface sealant should provide surface roughness below the threshold surface roughness for bacterial retention after oral exposure (Bollen, Lambrechts & Quirynen, 1997).

An adhesive patch specifically designed to seal smooth enamel surfaces has recently been developed (Schmidlin & others, 2005). As revealed by a radiochemical liquid scintillation method in the latter study, this patch protects enamel significantly better than a double layer of unfilled resin from chemomechanical challenges. Based on these promising initial results, this study aimed to assess the wear resistance and surface roughness of this patch when used as a smooth surface sealant after chemical (exposure to saliva or lactic acid) and mechanical (toothbrushing) exposure.

METHODS AND MATERIALS

Specimen Preparation

Forty-eight freshly extracted permanent central lower bovine incisors were selected for this study. Discs, approximately 7 mm in diameter, were cut from the midlabial aspect of each tooth using a trephine. The discs were then thinned, with retention of the natural enamel surface kept to approximately 2 mm in depth, and centrally inserted in stainless steel specimen carriers (PPK, Zurich, Switzerland) using a light-curing flowable composite (Tetric Flow, Ivoclar Vivadent, Schaan, Liechtenstein). The custom-made steel carriers ensured an exact repositioning of the specimens in a 3D-measuring device for wear measurements. The enamel was flattened and polished under water cooling with a rotating sandpaper device (Pedemax-2, Struers A/S, Copenhagen, Denmark), using waterproof silicon carbide paper (Struers A/S) for 15 seconds, each with 1,200 and 2,400 grit, then for 1 minute with 4,000 grit at 150 rpm.

The 48 enamel specimens were then randomly divided to 3 groups of 16 each, which received the following treatment: Group A) remained untreated (positive control), Group B) sealed with a fine hybrid composite (Tetric Flow, Vivadent) and Group C) sealed using an adhesive patch bonded to the enamel surface with a bonding system (Syntac Classic, Ivoclar Vivadent). The adhesive patch used in this study is a methacrylic groups-containing, elastic, crosslinked, urethane-based polymer material approximately 100-µm thick, according to the manufacturer. Upon light exposure in the wavelength ranging from 400 to 500 nm (blue light), full polymerization of the methacrylic groups occurred, rendering the patch hard and solid, enabling it to copolymerize with other resin-based dental materials (Schmidlin & others, 2005). For the sealed groups (B and C), enamel specimens were etched using 35% phosphoric acid for 60 seconds (Ultraetch, Ultradent Products Inc, South Jordan, Utah, USA), rinsed with 40 mL of distilled water and air dried. Additionally, acetone was applied in order to ensure a perfect drying of the etched enamel. Subsequently, enamel bond was applied. After a 20-second penetration time, the bonding agent was blown to a thin layer. The filled fine hybrid composite material in Group B was adapted to a thin layer and light cured against a transparent polyester strip (Hawe, Bioggio, Switzerland). In Group C, the patch (Prototype, Ivoclar Vivadent) was firmly adapted and light cured for 60 seconds (Optilux 500, 700 mW/cm^2, Demetron Inc, Danbury, CT, USA).

After sealing, the specimens were stored in a 50%-humidity chamber and randomly allocated to 2 groups of 24 specimens. One half was designated for immersion in saliva and the other half in lactic acid (n=8 per group).

Chemical and Mechanical Exposure

Specimens were randomly divided into two groups and immersed at 37°C under constant motion for 21 days in 5 mL of artificial saliva or 5 mL of lactic acid (15 µmol/L, pH 4). The artificial saliva used in this study had a pH of 7.6. It consisted of hydrogen carbonate (22.1 mmol/L), potassium (16.1 mmol/L), sodium (14.5 mmol/L), hydrogen phosphate (2.6 mmol/L) plus boric acid, calcium thiocyanate and magnesium in concentra-

tions of less than 1 mmol/L. After immersion in either saliva or lactic acid for 1, 2, 4, 7, 14 and 21 days, the liquid immersion media were discarded and the specimens rinsed with distilled water. At these intervals, the specimens received an equivalent of 10 double-stroke brush cycles per day in an automated 8-place brushing machine using a standard manual toothbrush (Paro M29, ESRO AG, Thalwil, Switzerland). The frequency of brushing was 60 cycles/minute. The head load was set at 250 g. For brushing, 60 g of abrasive slurry were placed into the chamber of each specimen. This standard abrasive slurry consisted of 10 g calcium pyrophosphate suspended in a 50 g solution of carboxymethyl cellulose (0.5%), glycerol (10%), saliva substitute and 50 µL of a silicon antifoaming agent. After brushing, the teeth were rinsed with 20 mL of distilled water and re-immersed in saliva or lactic acid, respectively.

Wear and Mean Surface Roughness Analyses

Parameters were collected at baseline and following immersion and brushing after 1, 2, 4, 7, 14 and 21 days.

Quantitative analysis of wear of untreated enamel and sealants was carried out before and after treatment phases using a surface analyzer (3DS, PPK, Zurich, Switzerland). The custom-made surface analyzer consisted of a computer, connected with a surveyor's table that moved in 1 µm steps in an x, y and z axis by three stepper motors controlled by a touch-switch caliper-needle. Before sealing, the center of each specimen was marked. The coordinates of reference-points of the surveyor's table, specimen carriers and center points were saved and allowed exact repositioning of the specimens after each cycle. The size of the measuring field was set to 9 mm^2. To prevent the specimens from drying, an adhesive tape (Tesa, Beiersdorf, Hamburg, Germany) was luted circularly to the carriers and filled with tap water. Substance loss was calculated by overlying the scanning data with congruent points and subtracting initial measurements (after embedding and polishing) from subsequent measurements (after sealing and at evaluation time). Wear was calculated as the mean substance loss over all points measured.

At baseline and after each treatment period, an impression was taken using an addition-type polvinylsiloxane of low viscosity (President light body, Coltène AG, Altstätten, Switzerland) and replicas (Stycast, Emerson & Cuming, Westerlo, Belgium) of the surfaces were cast. The average surface roughness (Ra) was quantified in µm using a computerized profilometer (Form Talysurf 50, Rank Taylor Hobson). The average readings of 10 measurements per specimen were compared. After evaluation, the replicas were sputtered with gold (Sputter SCD 030, Balzers Union, Balzers, Liechtenstein) and qualitatively analyzed by SEM at 500x magnification (Amray 1810/T, Amray Inc, Bedford, MA, USA).

Data Analysis and Presentation

Statistical analysis was performed with StatView (Version 5, Abacus Concepts Inc, Berkley, CA, USA). SPSS (Version 10, SPSS Inc, Chicago, IL, USA) was used for repeated measures analysis only. Results with *p*-values less than 5% were considered to be statistically significant.

First, univariate statistics for wear and surface roughness at every time point and for every treatment/immersion group were computed. As data distribution was fairly symmetrical, parametric statistics were applied. Second, the influence of chemical and mechanical stress was investigated. Differences between the observed values for wear and surface roughness at days 2, 4, 7, 14 and 21, and the values measured at day 1 (W_{day}-W_{day1}, Ra_{day}-Ra_{day1}) were computed. Repeated measures analyses, together with the Bonferroni post-hoc correction, were applied to disclose differences in the influence of the chemical and mechanical stress among the six treatment/immersion groups. Moreover, linear regression equations for each treatment/immersion group were estimated separately. These regression equations provide a more precise description of the influence of the chemical and mechanical stress on wear and surface roughness over time. The estimated slope, together with the corresponding p- and R-Square values, were reported. In order to visualize the changes over time, a bivariate scattergram, together with the estimated regression lines and corresponding 95% CIs, is presented. Moreover, for the differences W_{day14}-W_{day1} and W_{day21}-W_{day1}, a one sample *t*-test was applied.

RESULTS

Sealant Layer Thickness and Wear Rate

The mean layer thickness at baseline was 245.1 µm (95% CI=202.8 µm, 287.4 µm) in the composite treatment group and 100.6 µm (94.5 µm, 106.8 µm) in the patch group. The results of vertical dimensions and their changes after immersion (mean ± SD) in either artificial saliva or lactic acid are presented in Table 1. In artificial saliva, no differences in wear rates over time were found between treated and untreated specimens (p=0.562). These findings were confirmed by regression analysis (Table 2 and Figure 1). The 95% CIs of the regression lines for all treatment/immersion groups were overlapping and thus confirmed the findings of the repeated measures analysis.

On the other hand, untreated specimens in lactic acid showed significant substance loss using the repeated measures analysis, which was evident after 14 and 21 days (p=0.019).

Table 1: *Vertical dimensions (in µm) of specimens measured at baseline and after immersion in either artificial saliva or lactic acid (for 1 up to 21 days) and consecutive brushing (10 strokes per day). Figures in parentheses represent standard deviations.*

				Saliva			
Treatment	Baseline/ 0 strokes	day 1/ 10 strokes	day 2/ 20 strokes	day 4/ 40 strokes	day 7/ 70 strokes	day 14/ 140 strokes	day 21/ 210 strokes
Composite	259.6 (104.6)	260.9 (102.5)	261.2 (102.5)	260.3 (101.7)	257.0 (103.4)	259.2 (102.2)	255.7 (104.0)
Patch	98.0 (10.6)	102.9 (13.2)	102.1 (10.0)	101.6 (10.8)	98.0 (8.7)	100.9 (10.1)	99.1 (11.1)
Untreated	0.6 (1.9)	2.4 (3.7)	4.4 (5.9)	4.7 (7.1)	1.7 (5.4)	2.3 (6.8)	5.1 (5.5)
				Lactic Acid			
Treatment	Baseline/ 0 strokes	day 1/ 10 strokes	day 2/ 20 strokes	day 4/ 40 strokes	day 7/ 70 strokes	day 14/ 140 strokes	day 21/ 210 strokes
Composite	230.5 (45.6)	230.2 (46.1)	229.6 (47.4)	230.2 (46.9)	229.8 (46.6)	227.2 (45.7)	229.6 (46.9)
Patch	103.4 (12.5)	104.1 (14.8)	105.9 (13.7)	105.4 (14.4)	103.1 (13.7)	105.0 (13.7)	107.1 (14.7)
Untreated	1.1 (2.5)	3.7 (3.7)	1.8 (3.5)	0.2 (2.7)	-2.8 (3.4)	-5.9 (5.3)	-6.0 (3.1)

Table 2: *Linear regression of wear for every treatment/ immersion group separately, represented as slopes together with the corresponding p- and R-Square values.*

	Saliva		
	Slope	*p*-value	R-squared
Composite	0.224	0.277	0.006
Patch	0.106	0.604	0.007
Untreated	-0.018	0.903	0.000
	Lactic Acid		
	Slope	*p*-value	R-squared
Composite	0.059	0.675	0.005
Patch	-0.081	0.576	0.008
Untreated	0.413	0.007	0.175

Mean Surface Roughness Ra and Micro-morphologic Assessment

The mean surface roughness of specimens was 0.03 ± 0.01 µm at baseline. The results of surface roughness after immersion are presented in Table 3. Specimens sealed with flowable resin or the adhesive patch showed comparable mean surface roughness characteristics during the period of investigation and did not differ significantly from one other. The influence on surface roughness change over time caused by chemical and mechanical stress was most pronounced in the untreated, lactic acid group. As calculated using the repeated measures analysis, untreated specimens differed significantly from the other groups ($p<0.0001$). On the other hand, with specimens immersed in artificial saliva, samples treated with the patch under investigation showed higher surface roughness values than their untreated counterparts ($p=0.044$). No other pairwise differences in the influence of chemical and mechanical stress over time on the change of surface roughness between groups could be identified ($p=0.385$).

The visual impression given in Figure 2 confirmed the findings of repeated measure analysis. Results of the calculated linear regressions are summarized in Table 4. The most pronounced influence of chemomechanical stress on surface roughness values over time was evident in the untreated, lactic acid group (slope=0.007, $p=0.0023$, R-Squared=0.219).

SEM evaluation revealed clearly visible surface alterations after the 21-day observation period (Figure 3). A smooth enamel surface could only be seen on untreated enamel in the saliva group. All other treatments resulted in clearly visible surface striations caused by the brushing procedure. In some sealed specimens, partial loss of fillers in the superficial zone of the resin material caused some pit-like defects. These were most pronounced in the patch group after exposure to lactic acid. Untreated enamel in the lactic acid group showed additional erosive changes.

DISCUSSION

The pre-cured patch tested in this study was shown to be as wear-resistant as the flowable resin material under investigation. No significant loss of bonding material could be detected over the observation period. In contrast, enamel exposed to lactic acid showed significant substance loss compared to all other groups. The sealing of smooth tooth surfaces may be an effec-

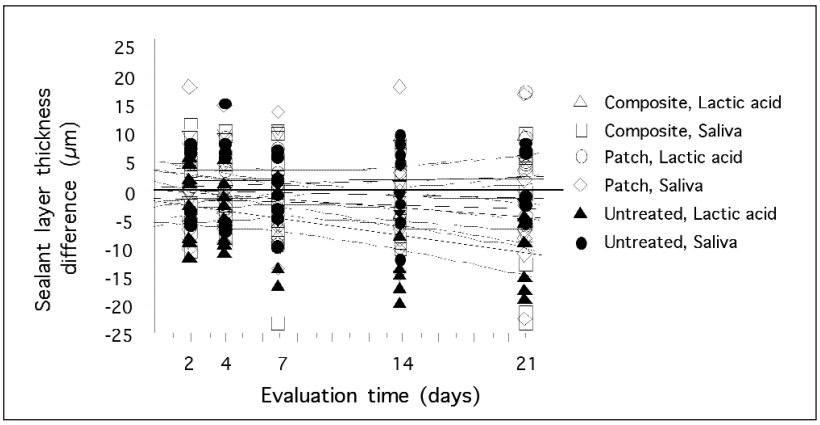

Figure 1. *Graphic depiction of the sealant layer thickness difference as bivariate scattergram with regression lines and 95% confidence bands.*

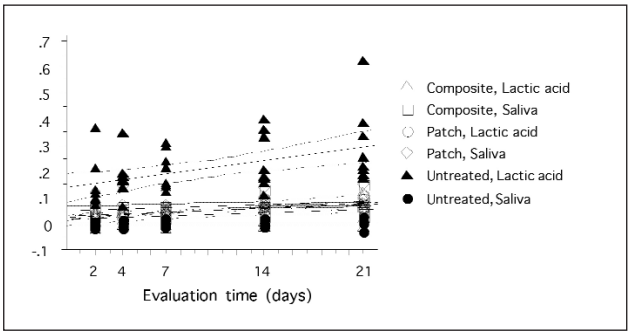

Figure 2. *Graphic depiction of the mean surface roughness difference as bivariate scattergram with regression lines and 95% confidence bands. Note that if the 95% CIs are not overlapping, there is strong statistical evidence of the differences between the regression lines.*

tive preventive strategy in dentistry. Several possible fields of application using adhesive bonding techniques have arisen, such as sealing areas that are at higher risk for caries development—interproximal surfaces (Schmidlin & Besek, 2003), the sealing of enamel white-spot lesions to eliminate caries progression (Robinson & others, 2001; Tantbirojn & others, 2000), the sealing enamel or dentin in patients with uncontrolled dental erosion (Azzopardi & others, 2001) or the sealing of sensitive root dentin (Momoi & others, 2003).

It should be acknowledged that the study conditions described in this report differed from the *in vivo* situation where there was no protective salivary pellicle (Zahradnik, Moreno & Burke, 1976) and in enamel surfaces that were in continuous contact with the erosive challenge in the lactic acid group. Possible deleterious effects of sealant expansion and contraction by thermal cycling were also not tested. Nevertheless, the current experimental conditions appeared suitable to test the initial behavior and stability of a bonding material for an *in-vitro* screening.

A previous laboratory study demonstrated that even the twofold application of an unfilled resin on smooth enamel surfaces resulted in leakage and partial loss of the bonding agent (Schmidlin & others, 2002). In that study, the authors used a similar protocol to this investigation, but without applying additional wear induced by tooth-slurry/toothbrush abrasion after chemical stressing during a 14-day observation period. Despite

Table 3: *Mean surface roughness (Ra in µm) of specimens measured after immersion in either artificial saliva or lactic acid (for 1 up to 21 days) and consecutive brushing (10 strokes per day). Figures in brackets represent Standard Deviations.*

Saliva						
Treatment	day 1/ 10 strokes	day 2/ 20 strokes	day 4/ 40 strokes	day 7/ 70 strokes	day 14/ 140 strokes	day 21/ 210 strokes
Composite	0.03 (0.01)	0.07 (0.01)	0.07 (0.01)	0.07 (0.01)	0.09 (0.03)	0.10 (0.03)
Patch	0.04 (0.01)	0.10 (0.03)	0.09 (0.04)	0.10 (0.03)	0.11 (0.03)	0.13 0.03
Untreated	0.04 (0.01)	0.04 (0.01)	0.04 (0.01)	0.04 (0.04)	0.04 (0.01)	0.04 (0.01)
Lactic Acid						
Treatment	day 1/ 10 strokes	day 2/ 20 strokes	day 4/ 40 strokes	day 7/ 70 strokes	day 14/ 140 strokes	day 21/ 210 strokes
Composite	0.05 (0.03)	0.06 (0.01)	0.07 (0.01)	0.07 (0.01)	0.09 (0.01)	0.10 (0.02)
Patch	0.03 (0.01)	0.06 (0.02)	0.07 (0.01)	0.08 (0.02)	0.10 (0.02)	0.11 (0.03)
Untreated	0.04 (0.01)	0.19 (0.1)	0.21 (0.08)	0.24 (0.07)	0.27 (0.11)	0.33 (0.15)

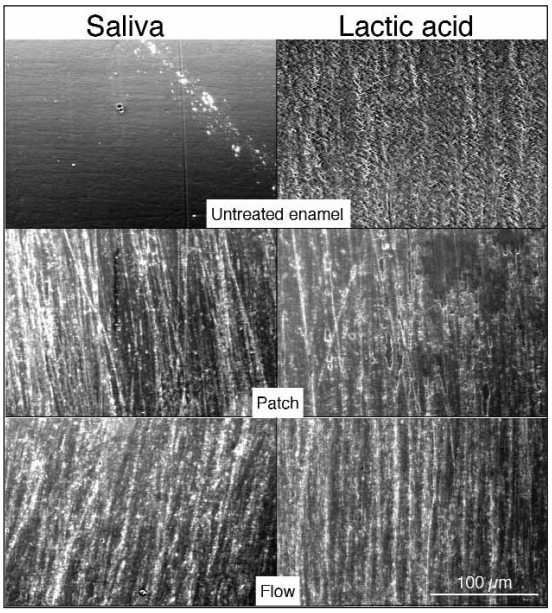

Figure 3. *SEM pictures at 500x magnification. Surface alterations after immersion in lactic acid and artificial saliva. Consecutive brushing are visible in the form of striations.*

this shorter, less aggressive setup, the unfilled resins tested clearly lacked the ability to withstand chemical degradation. These results were supported by the findings of another study that tested the effectiveness of two bonding agents applied on dentin when exposed to an erosive/abrasive challenge. The use of a nanofilled bonding agent (mean filler particle size 7 nm) was less wear-resistant compared to a microfilled (filler particle size 7 µm) bonding agent (Azzopardi & others, 2001). Partial breakdown, as specimens were subjected to acid attack and toothbrushing, however, was shown for both materials tested. In this context, clinical experience and laboratory experiments support the assumption that enamel and dentin continues to be protected from erosive attack when the sealant appears to be lost clinically (Handelman, Jensen & Pameijer, 1978; Davidson & Bekke-Hoekstra, 1980). This can be explained by the presence of tags. The remaining adhesive may still cover micro crevices of the etch pattern, and a hybrid layer may have been established. Nevertheless, a mechanically stable, wear- and acid-resistant sealant seems to be a prerequisite for predictable, long-term success and stability.

When enamel specimens were immersed in lactic acid and consecutively brushed under the conditions of this study, surface roughness values increased significantly and exceeded the suggested threshold surface roughness for bacterial retention, representing an Ra value of more than 0.2 µm (Bollen & others, 1997; Carlen & others, 2001). Both the flowable material, when light-cured against a matrix, and the adhesive patch showed smooth surfaces at the beginning and only a small Ra increase after chemomechanical stressing, that did not exceed 0.17 µm.

Table 4: *Linear regression of the surface roughness measurements for every treatment/immersion group separately, represented as slopes together with the corresponding p- and R-Square values.*

	Saliva		
	Slope	*p*-value	R-squared
Composite	0.002	0.001	0.255
Patch	0.002	0.013	0.153
Untreated	0.000	0.552	0.009
	Lactic Acid		
	Slope	*p*-value	R-squared
Composite	0.002	0.004	0.195
Patch	0.003	<0.0001	0.490
Untreated	0.007	0.002	0.219

Within the limitations of this study, the protective potential of the adhesive patch was confirmed. The patch showed a similar laboratory performance to a filled resin composite and may therefore successfully protect enamel. The method presented may provide valuable information regarding the stability and resulting surface roughness of this newly devised material under standardized conditions. Clinical trials are now warranted to assess the clinical effectiveness of this material.

CONCLUSIONS

This study investigated the wear resistance and resulting mean surface roughness of a newly devised adhesive patch after chemomechanical exposure. Findings confirmed the results of an earlier investigation that mainly evaluated the caries protective value of this material. It was found that the patch had good wear resistance and a clinically acceptable surface roughness. Both parameters were comparable to the corresponding values obtained using a flowable resin composite that served as the negative control. It was concluded that this patch is of considerable interest in the ongoing search for a controllable application technique of sealants to smooth enamel surfaces and, therefore, merits further clinical investigations.

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