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Microtensile adhesion of sealants to intact enamel

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Summary. Contamination of etched enamel with saliva has been shown to result in sealant failure. Hydrophilic adhesives improve retention of sealants when enamel is contaminated.

Objective. The objective of the present study was to characterize the adhesion of two sealants to unprepared enamel etched with phosphoric acid or conditioned with the all-in-one, self-etch adhesive Adper Prompt L-Pop.

Methods. The two proximal enamel surfaces of 16 sound molars were assigned to four conditioning regimens: (1) 35% phosphoric acid for 15 s; (2) Adper Prompt L-Pop, one layer light-cured prior to sealant placement; (3) Adper Prompt L-Pop, two layers light-cured prior to sealant placement; and (4) Adper Prompt L-Pop, one layer co-cured with sealant. One of two sealants, i.e. Clinpro or Delton DDS, was applied, light-cured, followed by a composite build-up to provide support for microtensile bond testing.

Results. The highest mean bond strengths were obtained when Adper Prompt L-Pop was applied in two layers and light-cured prior to the insertion of Clinpro Sealant. Co-cure of Adper Prompt L-Pop with either sealant resulted in bond strengths which were not significantly different from those of the phosphoric acid control. The use of Adper Prompt L-Pop in one layer cured prior to sealant placement resulted in statistically lower bond strengths than any other application technique regardless of the sealant used. Clinpro Sealant resulted in statistically higher bond strengths than Delton DDS Sealant.

Conclusions. Application of one layer of Adper Prompt L-Pop co-cured with the sealant resulted in bond strengths similar to those obtained with phosphoric acid etching.

Introduction

The first report of the use of the acid-etch technique to seal fissures dates from 1967 [1]. Several studies have since demonstrated that sealants are highly effective in caries reduction, especially if the sealant remains intact [2–6].

One of the factors responsible for sealant failure is saliva contamination of the etched enamel surface [7,8]. It has been shown that a very short period of contact between saliva and etched enamel can prevent penetration of fluid resins into the enamel microporosities [9]. The use of hydrophilic dentin adhesives as an intermediate layer between etched enamel and sealant was first reported in 1992 [10]. Hydrophilic dentin adhesives improve sealant retention [11] and decrease microleakage when enamel is contaminated with saliva [8]. The use of a filled hydrophilic dentin adhesive in lieu of the sealant may also reduce microleakage around saliva-contaminated enamel and improve retention clinically [12,13].

The recent introduction of 'all-in-one' self-etching adhesives (SEAs) shortened and simplified the bonding technique. Self-etching adhesives do not require a separate acid-etch step since they condition and prime enamel and dentin simultaneously, without rinsing, relying on their ability to partially dissolve

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hydroxyapatite to yield a resin-infiltrated zone with incorporated minerals [14]. Therefore, the use of a nonrinsing hydrophilic adhesive for sealant placement has an advantage for the clinician. Self-etching adhesives have been classified into three categories: mild, moderate and aggressive [15]. Self-etching materials are composed of aqueous mixtures of acidic functional monomers, generally phosphoric acid esters, with a pH somewhat higher (less acidic) than phosphoric acid etching gels [16].

The objective of the present study was to characterize the adhesion of two sealants to unprepared enamel etched with phosphoric acid or conditioned with the all-in-one SEA Adper Prompt L-Pop (3M ESPE, St Paul, MN, USA). The null hypothesis tested was that neither the type of sealant nor the type of enamel conditioning used in this study affected microtensile enamel bond strengths.

Methods

Sixteen third molars which had been freshly extracted for surgical reasons were stored in 0·5% chloramine solution until they were used in the present study. The enamel was evaluated under a stereomicroscope to check for the presence of possible enamel defects. If any defects were detected, the teeth were discarded. Care was taken to choose teeth with similar proximal flat surfaces in order to eliminate intervening variables. Each tooth was cleaned with pumice with a prophy cup at low speed for 10 s.

The selected teeth were attached to a phenolic ring (Buehler Ltd, Lake Bluff, IL, USA) with sticky wax and sectioned in a precision low-speed diamond saw (Isomet 1000, Buehler Ltd) to obtain two proximal enamel specimens of $8{\cdot}0 \times 4{\cdot}0$ mm [2] from each tooth (a total of 32 proximal surfaces). Four enamel specimens were then randomly assigned to each of eight experimental groups (Table 1). The materials and respective batch numbers are described in Table 2.

Table 1. Experimental groups.

Group	Etch	Adper Prompt L-Pop	Sealant
1	Yes*	No	Clinpro
2	No	One layer, light-cured	Clinpro
3	No	Two layers, light-cured	Clinpro
4	No	One layer, co-cured with sealant	Clinpro
5	Yes*	No	Delton DDS
6	No	One layer, light-cured	Delton DDS
7	No	Two layers, light-cured	Delton DDS
8	No	One layer, co-cured with sealant	Delton DDS

*Using 35% H_3PO_4.

In groups 1 and 5, the enamel was etched with 35% phosphoric acid gel (Scotchbond Etchant, 3M ESPE) for 15 s, rinsed for 15 s, and air-dried with oil-free compressed air until the enamel displayed a white, frosty appearance. A layer of sealant (Group 1 – Clinpro Sealant, 3M ESPE; Group 5 – Delton DDS Sealant, Dentsply Caulk, Milford, DE, USA) was applied to a thickness of $0{\cdot}75 \pm 0{\cdot}1$ mm and light-cured for 20 s with a halogen light-curing device (Curing Light 2500, 3M ESPE). The intensity of the curing light was monitored daily and exceeded 500 mW cm^{-1} [2]. To provide a gripping surface for the microtensile test, a universal hybrid composite resin (Filtek Z250, Shade A3, 3M ESPE) was applied and polymerized in four increments on

Table 2. Materials used.

Material	Manufacturer	Batch number	Composition
Clinpro Sealant	3M ESPE, St Paul, MN, USA	20021121	Light-cured, bisGMA/TEGDMA-based sealant, unfilled. Colour change chemistry to aid in placement procedure
Delton DDS Light-cured Sealant	Caulk Dentsply, Milford, DE, USA	020222	Resin-based, light-cured sealant
Adper Prompt L-Pop Adhesive System	3M ESPE	132149	One-step self-etch system adhesive system in patented L-Pop delivery system. HEMA phosphates provide the acidic component, with HEMA, bisGMA and a modified polyalkenoic acid providing the resin components. Water-based, pH ≈ 1
Filtek Z250	3M ESPE	2YE	Visible-light-activated hybrid restorative composite. Resin consists of bisEMA, bisGMA and UDMA. Filled to 60% by volume with zirconia silica filler, average particle size = 0·6 μm
Scotchbond Phosphoric Acid Etchant	3M ESPE	2YX	35% phosphoric acid with silica thickening agent. pH ≈ 0·6

the surface of the cured sealant to obtain an 8·0-mm-high composite build-up.

In groups 2 and 6, Adper Prompt L-Pop was activated, following the manufacturer's instructions, and brushed onto the enamel surface with a rubbing movement and pressure for 15 s. The excess was blown with an air stream until the surface appeared shiny with a thin adhesive film. The adhesive was light cured for 10 s. A layer of sealant (Group 2 – Clinpro Sealant; Group 6 – Delton DDS Sealant) was applied to a thickness of $0{\cdot}75 \pm 0{\cdot}1$ mm and light-cured for 20 s. To provide a gripping surface for the microtensile test, Filtek Z250 was applied in four increments in the same manner as described for group 1.

In groups 3 and 7, Adper Prompt L-Pop was activated and a first layer was applied onto the entire enamel surface with a rubbing motion for 15 s, and this was thoroughly air-dried to remove the aqueous solvent. Then a second layer of Adper Prompt L-Pop was applied and rubbed for 15 s, thoroughly air-dried, and light-cured for 10 s. A $0{\cdot}75 \pm 0{\cdot}1$-mm-thick layer of sealant (Group 3 – Clinpro Sealant; Group 7 – Delton DDS Sealant) was immediately applied and cured for 20 s. The composite build-up was inserted as described previously.

In groups 4 and 8, Adper Prompt L-Pop was activated and one layer was applied onto the enamel surface with rubbing motion for 15 s, and this was thoroughly air-dried without light curing. The sealant (Group 4 – Clinpro Sealant; Group 8 – Delton DDS Sealant) was immediately applied over the nonpolymerized adhesive and they were co-cured for 20 s. The composite build-up was inserted as described for the other groups.

The specimens were mounted on a phenolic ring and sectioned parallel to the adhesive interface to obtain $0{\cdot}7 \pm 0{\cdot}1$-mm-thick slabs. These slabs were individually identified by applying different colours to the composite resin. Each slab was attached to a phenolic ring and a second set of $0{\cdot}7 \pm 0{\cdot}1$-mm-thick sections was carried out perpendicularly to the adhesive interface. All sticks were individually identified by painting coloured dots on the dentin portion. For some specimens, the bonded interface close to the margin of the enamel was not exactly perpendicular to the long axis of the stick, and therefore, these sticks were discarded.

The specimens were tested individually by attaching them to a Geraldeli jig [17] using ZapIt cyanoacrylate glue (ZapIt, DVA, Corona, CA, USA). The sticks were then submitted to a tension load using an Instron 4204 testing machine (Instron Co., Canton, MA USA) at 1 mm min^{-1} cross-head speed. A Mitutoyo absolute digital caliper (Mitutoyo Corp., Kanogawa, Japan) with an accuracy of 0·001 mm was used to measure the sides of the bonding interface and to calculate the bonding area in millimetres [2]. The load (in kilograms) and the bonding surface area of the specimen were registered on a work sheet, and microtensile bond strengths were calculated in MPa.

Statistical analysis was carried out with the SPSS 10 (SPSS Inc., Chicago, IL, USA) computer software package. A two-way analysis of variance was computed, followed by a Tukey post-hoc test at $P \leq 0{\cdot}05$.

Results

The results are shown in Tables 3 and 4. The highest mean bond strengths for Clinpro Sealant were obtained when Adper Prompt L-Pop was applied in two layers and light-cured prior to the insertion the

Table 3. Mean microtensile bond strengths (MPa) and standard errors.

Enamel treatment	Sealant	Mean*	Standard error	Number of sticks†
(1) Phosphoric acid	Clinpro	15·69[b]	1·24	35
	Delton DDS	15·45[b]	1·16	40
(2) Adper Prompt L-Pop, one layer, light-cured prior to sealant placement	Clinpro	9·77[c]	1·19	38
	Delton DDS	9·22[c]	1·24	35
(3) Adper Prompt L-Pop, two layers, light-cured prior to sealant placement	Clinpro	22·78[a]	0·99	54
	Delton DDS	15·16[b]	1·06	48
(4) Adper Prompt L-Pop, one layer, co-cured with sealant	Clinpro	16·60[b]	1·03	50
	Delton DDS	18·03[a,b]	1·02	51

*Means followed by the same superscript letter are not statistically different at $P < 0{\cdot}05$.
†Since the group sizes are unequal, the post-hoc test uses the harmonic mean of the group sizes.

Table 4. Two-way analysis of variance.

Source	Sum of squares	d.f.	Mean square	*F*-value	Significance
Enamel conditioner	4154·036	3	1384·679	25·745	$P < 0·000$
Type of sealant	260·497	1	260·497	4·843	$P < 0·028$
Conditioner versus sealant	1196·361	3	398·787	7·414	$P < 0·000$
Error	18448·281	343	53·785		
Total	112639·059	351			

sealant. The use of Adper Prompt L-Pop in one layer cured prior to sealant placement (treatment 2) resulted in statistically lower bond strengths than any other application technique, regardless of the sealant used. The co-cure technique, where adhesive and sealant were cured with a single light-cure exposure, resulted in bond values which were not statistically different from those of the phosphoric acid control.

When means were pooled for the enamel-conditioning technique, Clinpro Sealant resulted in statistically higher bond strengths than Delton DDS Sealant at $P < 0·028$ (Table 4). All failures were of an adhesive nature.

Discussion

Etching enamel with phosphoric acid forms porosities on the enamel surface [18]. Low-viscosity resins composed of unfilled bisGMA flow promptly into the microdepressions created by the acid [18], allowing high enamel bond strengths [19]. Water participates in the chemical reaction associated with the interaction of SEAs with enamel. This water, present in the composition of SEAs, is needed for acidic monomers to ionize and trigger demineralization of hard dental tissues. This makes SEAs less susceptible to variations in the degree of substrate moisture, which is a characteristic of SEAs.

The enamel-etching capability of SEAs has been studied abundantly [20–22]. One of the shortfalls of SEAs is that they may not etch enamel to the same depth achieved with phosphoric acid [14,21]. However, other studies have demonstrated that enamel bonding with SEAs is as effective as enamel bonding after phosphoric acid etching [23,24].

The previous version of Prompt L-Pop, which had a pH similar to the current Adper version used in the present study, resulted in an etching effect similar to that of phosphoric acid both for intact [15] and for prepared enamel [25]. One study reported that Prompt L-Pop resulted in enamel bond strengths which were significantly higher than other simplified adhesives [26]. When compared to total-etch adhesives, it has been shown that the bond strengths of Prompt L-Pop are similar [23] or lower [15,27]. A different study reported that the enamel bond strengths obtained with Adper Prompt L-Pop were comparable to those obtained upon etching enamel with phosphoric acid [28]. Additionally, the enamel etching pattern in aprismatic enamel after the application of Adper Prompt L-Pop was very similar to the etching pattern of phosphoric acid [29]. When combined with the composite Z250, as used in the present study, Adper Prompt L-Pop resulted in mean enamel bond strengths of 33·0 MPa. The bond strengths obtained with the total-etch adhesive Single Bond were statistically similar (32·2 MPa) [30]. In another study [31], Adper Prompt L-Pop (experimental version EXL #547) was used with Transbond XT (3M Unitek, Monrovia, CA, USA) to bond brackets in shear mode. The corresponding enamel bond strengths (9·7 MPa) were statistically similar to those of phosphoric-acid-etched enamel (10·4 MPa).

The magnitude of enamel bond strengths obtained in the present study is somewhat lower than the typical enamel bond strengths cited in the literature for enamel specimens bonded with adhesive systems and composite resins [14,25]. Two reasons may have accounted for this difference. First, the enamel used in the present study was not roughened; therefore, the composition of the bonding substrate may have included aprismatic enamel. Changes occur in the outermost enamel layer after eruption [32] and a prismless enamel layer may be present [33] that prevents the penetration of adhesives. Secondly, enamel bond strengths obtained with the microtensile method have been described as being generally lower than those obtained with shear or tensile methods [34].

Isolation of teeth prior to sealant placement is difficult, especially in partially erupted molars. This limitation is expressed in the rate of sealant replacement, which is higher in molars that were in an early

stage of eruption stage as a result of saliva contamination of etched enamel [35]. Complete penetration of the etchant into the fissures is an essential step in the retention of pit and fissure sealants [36]. However, there have been reports of insufficient penetration of the phosphoric acid etchant into the fissure system [37,38]. Since the use of a hydrophilic adhesive prior to the insertion of the sealant improves retention of the sealant and decreases microleakage [11,39], the use of Adper Prompt L-Pop may be a valid alternative to acid etching with phosphoric acid.

What this paper adds

- Self-etching adhesives result in bond strengths similar to those obtained with phosphoric acid etching when used with sealants.
- The highest mean bond strengths for Clinpro Sealant was obtained when Adper Prompt L-Prop was applied in two layers and light-cured prior to the insertion of sealant.

Why is this paper relevant to paediatric dentists?

- The use of self-etching adhesives do not require a separate acid-etch step since they condition and prime enamel and dentin simultaneously.

Conclusions

Within the limitations of this study, the following conclusions can be drawn:

1 For Clinpro Sealant, two coats of Adper Prompt L-Pop cured prior to sealant placement resulted in higher enamel bond strengths than any other enamel surface treatment used in the present study.

2 For either Clinpro Sealant or Delton DDS Sealant, one layer of Adper Prompt L-Pop co-cured with the sealant resulted in bond values which were not statistically different from those of the phosphoric acid control.

3 Clinpro Sealant resulted in higher enamel bond strengths than Delton DDS Sealant.

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Résumé. Il a été montré que la contamination de l'émail mordancé par la salive conduit à un échec des scellements de sillon. Les adhésifs hydrophiles augmentent la rétention des scellants quand l'émail est contaminé.

Purpose. L'objectif de cette étude a été de caractériser l'adhérence de deux scellants à de l'émail non préparé mordancé à l'acide phoshorique ou conditionné avec l'adhésif «tout-en-un» Adper Prompt L Pop.

Méthodes. Les deux surfaces amélaires proximales de 16 molaires saines ont été sélectionnées pour quatre types de conditionnement: (A) acide phosphorique 35% pendant 15 sec; (B) Adper Prompt L-Pop, une couche photopolymérisée avant le placement du scellant; (C) Adper Prompt L-Pop, deux couches photopolymérisées avant le placement du scellant; (D) Adper Prompt L-Pop, une couche photopolymérisée en même temps que le scellant. Un des deux scellants (Clinpro or Delton DDS) a été appliqué, photopolymérisé, suivi par un renforcement composite pour fournir un support au test de micro-traction du collage.

Résultats. Les forces moyennes d'adhérence les plus élevées ont été obtenues quand Adper Prompt L Pop a été appliqué en deux couches et photopolymérisé avant l'insertion de Clinpro Sealant. Le Adper Prompt L Pop co-polymérisé avec chaque scellant a résulté en des forces de liaison non significativement différentes du témoin avec acide phosphorique. L'utilisation de Adper Prompt L Pop en une couche polymérisée avant placement a donné des forces de collage statistiquement plus faibles que les autres techniques quelque soit le scellant utilisé. Clinpro a donné des forces de collage statistiquement plus élevées que Delton DDS.

Conclusions. L'application de Adper Prompt L-Pop co-polymérisé avec le scellant a donné des force de d'adhérence similaires avec celles obtenues avec mordançage à l'acide phosphorique.

Zusammenfassung. Die Kontamination von geätztem Schmelz mit Speichel wurde als mögliche Ursache für Retentionsverlust von Versiegelungsmaterial herausgestellt. Hydrophile Adhäsive verbessern die Retention von Versiegelungen an kontaminiertem Schmelz.

Ziel. Das Ziel dieser Studie war es, die Adhäsion von zwei Versiegelungsmaterialien an unpräpariertem Schmelz zu untersuchen einmal nach Ätzung mit Phosphorsäure und zweitens nach Konditionieren mit dem selbstätzenden Primer Adper Prompt L-Pop.

Methoden. Die proximalen Flächen von 16 kariesfreien Molaren wurden vier verschiedenen Vorbehandlungsmethoden zugeordnet:

A: 35%ige Phosphorsäure für 15 s; B: eine Lage Prompt L-Pop, gehärtet vor Auftragen der Versiegelungsmaterials; C: zwei Schichten Prompt L-Pop, Lichthärtung vor Versiegelerauftrag; D: Prompt L-Pop, eine Schicht, gehärtet zusammen mit dem Versiegelungsmaterial. Eines der beiden Versiegelungsmaterialien (Clinpro oder Delton) wurde appliziert und mit einem Kompositaufbau versehen um einen Abreißversuch vorzubereiten.
Ergebnisse. Die höchsten Mittelwerte wurden erzielt mit zweischichtiger L-Pop Applikation und Lichthärtung vor Auftrag von Clinpro. Gleichzeitiges Härten von L-Pop und Versiegelungsmaterial führte zu Werten, die nicht signifikant verschieden waren von den Werten der Säureätztechnik. Die einschichtige L-Pop-Applikation zeigte (unabhängig vom Versiegeler) niedrigere Werte. Clinpro erreichte höhere haftfestigkeitswerte als Delton.
Schlussfolgerungen. Die einschichtige Applikation von L-Pop und gleichzeitige Polymerisation von Versiegelungsmaterial und Primer führte zu ähnlichen Haftfestigkeitswerten wie die Säure-Ätztechnik.

Resumen. La contaminación con saliva del esmalte grabado se ha señalado que produce un fallo en el sellador. Los adhesivos hidrofílicos mejoran la retención de los selladores cuando el esmalte está contaminado.
Objetivo. El objetivo de este estudio fue describir la adhesión de dos selladores al esmalte sin preparar, grabado con ácido fosfórico o acondicionado con el adhesivo de autograbado todo en uno, Adper Prompt L-Pop.
Métodos. Las dos superficies de esmalte proximales de 16 molares sanos se asignaron a cuatro sistemas de acondicionamiento: (A) ácido fosfórico al 35% durante 15 segundos; (B) una capa fotopolimerizada de Adper Prompt L-Pop, antes de colocar el sellador; (C) dos capas fotopolimerizadas de Adper Prompt L-Pop antes de colocar el sellador; (D) una capa de Adper Prompt L-Pop fotopolimerizada junto al sellador. Tras aplicar uno de los dos selladores (Clinpro o Delton DDS) y fotopolimerizar, se continuó con una construcción de composite para dar soporte al test de adhesión por microtracción.
Resultados. Las medias de adhesión más altas se obtuvieron cuando se aplicó Adper Prompt L-Pop en dos capas y se fotopolimerizó antes de la inserción del sellador Clinpro. La polimerización conjunta de Adper Prompt L-Pop con cualquiera de los selladores produjo fuerzas de adhesión que no eran significativamente diferentes de las de control con ácido fosfórico. La utilización de Adper Prompt L-Pop en una capa, fraguada antes de la colocación del sellador, produjo fuerzas de adhesión estadísticamente inferiores que ninguna otra técnica de aplicación independientemente del sellador usado. El sellador Clinpro produjo fuerzas de adhesión estadísticamente más altas que el sellador Delton DDS.
Conclusiones. La aplicación de una capa de Adper Prompt L-Pop co-polimerizada con el sellador produjo fuerzas de adhesión similares a las obtenidas con el grabado de ácido fosfórico.

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