

ORIGINAL ARTICLE

Marginal and internal adaptation of composite restorations using a resin liner on deproteinized substrate

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Abstract

Objective. The purpose of this study was to evaluate the use of resin liner on deproteinized substrate on marginal and internal adaptation of composite restorations. **Material and methods.** Twenty-four recently extracted 3rd molars were selected. The crowns were sectioned in a mesio-distal direction, and the specimens were ground to expose a flat enamel area of at least 6 mm in diameter. Cavities were prepared on the central area of flattened surfaces. The specimens were randomly assigned into four groups ($n = 12$): SB – 1. Acid etch, 2. Single bond application (SB); NaOCl/SB – 1. acid etch, 2. 10% sodium hypochlorite solution application for 60 s (NaOCl), 3. SB; SB/PL – 1. acid etch, 2. SB, 3. protect liner F (PL); NaOCl/SB/PL – 1. acid etch, 2. NaOCl, 3. SB, 4. PL. All cavities were restored with Filtek Z250 and polished. The outer and internal margins were stained using Caries Detector[®] (Kuraray), observed under the stereomicroscope, and transferred to a computer measurement program. The length of the gap was expressed as a percentage of the total length of the margins observed. Data (internal adaptation) were submitted to ANOVA and Tukey's test. **Results.** There were no gaps at enamel outer margins in any specimen of any group. There was no statistically significant difference in internal adaptation between SB (18%) and NaOCl/SB (18%). NaOCl/SB/PL showed the worst internal adaptation (21%), with the higher percentage of internal gaps, statistically differing from SB/PL, which showed the best internal adaptation (12%). **Conclusions.** Resin liner can effectively enhance internal adaptation on demineralized dentin. However, on demineralized and deproteinized dentin it affects the internal adaptation of composite restorations negatively.

Key Words: Composites, dental restorations, resin liner, sodium hypochlorite

Introduction

Resin composites were introduced in dentistry in the mid-1960s and have improved in terms of esthetics, wear rate, and handling. However, a major disadvantage of composite restorations is their high polymerization shrinkage [1,2], which can create stress that may disrupt the bond to cavity walls [1]. The magnitude of the stress generated depends on the modulus of elasticity of the composite [1,3,4]. The correlation between stiffness (high elastic modulus materials) and shrinkage stress has been demonstrated [4]. According to Hooke's law, the most rigid material will cause the highest stress.

Unless the surrounding walls to which the composite is bonded supply enough elastic compliance, fractures will occur to compensate for the reduced volume and, consequently, create gaps and subsequent micro-leakage in composite restorations [1,5,6].

The use of intermediate resin liners has been proposed as a means of reducing the potential for marginal gaps [5,6]. The main purpose of resin liners is to act as a flexible stress-absorbing layer, or "elastic buffer", between the shrinking resin composite and the rigid substrate, which may reduce the high stress induced by the rigid composite polymerization shrinkage through a more elastic interface [5]. Estafan et al. [5] have confirmed the efficacy of this technique

Table I. Characteristics and main components of the materials used

Materials	Manufacturer and batch no.	Components
Single Bond	3M Dental Products, St. Paul, Minn., USA Batch: 1105/7BB	Bis-GMA, HEMA, dimethacrylates; water, ethanol, polyalkenoic acid, acid copolymer, photoinitiator
Filtek Z250 (A3)	3M Dental Products, St. Paul, Minn., USA Batch: 1370A3	Bis-GMA; Bis-EMA; UDMA; Inorganic filler—Zirconia/silica (60% volume); photoinitiator
Protect Liner F	Kuraray Co. Ltd, Japan Batch: 0042AY	Bis-GMA; TEGDMA; fluoride-methyl-methacrylate; silanized colloidal silica; prepolymerized organic filler; photoinitiator

in improving the marginal adaptation of composite restorations. In addition, Yazici et al. [7] have shown that a combination of flowable composites and hybrid composites yields the most effective microleakage reduction.

Many bonding systems have been developed to promote good adhesion between resin composite and dental substrates, but they cannot ensure a perfect seal of resin composite restorations, especially at the dentin/resin interface [6,8]. An adequate seal at these margins is critical, as demonstrated in the literature [6,9,10].

It is widely accepted that one method by which resin bond strength to dentin can be improved is through resin impregnation in the exposed collagen network of demineralized superficial dentin. This results in the formation of a hybrid layer after the monomers have been polymerized [11–13]. Although hybrid layer formation is considered the major mechanism involved in effective dentin bonding [9,13–15], some works have shown that this layer may not be necessary for good bond strength [11,15–18].

Dissolution of collagen fibrils after acid etching with a deproteinizing solution such as sodium hypochlorite may result in better monomer diffusion by increasing dentin permeability and changing its composition [17]. The altered dentin substrate may give equivalent or superior bonding results for some adhesive systems [11,16,18,19].

However, the use of sodium hypochlorite is a controversial issue, because, according to Uno & Finger [14] and Frankenberger et al. [15], the hybrid layer acts as an elastic layer decreasing the tension generated by polymerization shrinkage and masticatory impact. Absence of the hybrid coupling zone results in high stress concentrated at the hydroxyapatite surface of the collagen-free bonding interface, which has little flexibility. The risk of debonding is thus increased. Decreased marginal adaptation on deproteinized dentin has been reported [14,15].

The purpose of this study was to evaluate the marginal and internal adaptation of composite restorations using a resin liner on deproteinized substrate. The hypothesis tested was that the use of resin liner on deproteinized dentin might result in better stress transmission through the resin composite to the dental substrate, during the polymerization,

resulting in a better adaptation of the composite restorations.

Material and methods

The materials, manufacturers, batch numbers, and their composition are given in Table I. A 10% sodium hypochlorite solution pH 12.4 was prepared one day before use (Proderma Farmácia de Manipulação LTDA, Piracicaba, SP, Brazil).

Specimen preparation

Twenty-four sound human 3rd molars were selected, cleaned, and stored in a 0.5% Chloramine T solution at 4°C for no more than a week. After removing the roots 1 mm below the cemento-enamel junction, the crowns were sectioned in a mesio-distal direction using an ISOMET 1000 machine (Buehler, UK, Ltd, Lake Bluff, Ill., USA). The sectioned crowns were ground on a water-cooled mechanical polisher (Minimet 1000; Buehler) using 320-, 400-, and 600-grit silicon carbide (SiC) abrasive paper (Carbimet Disc Set, 305178180; Buehler) in order to expose a flat enamel area of at least 6 mm in diameter on the lingual, buccal, or palatine surfaces. The specimens were observed through a stereomicroscope (Zeiss; Manaus, AM, Brazil), at $\times 25$ magnification, to verify if the enamel remained on the surface.

Cavities (5 mm long \times 2 mm wide \times 2 mm deep) were prepared on the central area of the flattened surfaces. They were made using a cylindrical diamond (no. 2143; KG Sorensen Indústria e Comércio Ltda, São Paulo, SP, Brazil) mounted in a high-speed hand piece (Kavo, Joinville, SC, Brazil) under constant air-water cooling. The diamond was replaced after every 10th preparation.

Internal cavity walls had a 90° angle to the enamel surface plan, while the internal cavity angles were rounded with the diamond used.

The sliced tooth sections were randomly assigned into four groups ($n=12$), according to the following treatments:

SB—Control. The Single Bond adhesive system (SB) was applied in accordance with the manufacturer's instructions: the cavity was etched with 35% phosphoric acid (H_3PO_4) gel for 15 s, rinsed for 10 s, and

blot-dried. The adhesive system was applied twice with a 5-s interval in between, dried carefully with air to remove the solvent, approximately 15 s (observing a glossy surface), and light-cured for 10 s using the photocuring unit Elipar Tri-light (ESPE—America Co., Seefeld, Germany) with a power density of 800 mW/cm².

NaOCl/SB. The cavity was etched with 35% phosphoric acid (H₃PO₄) gel for 15 s, rinsed for 10 s and blot-dried. A 10% sodium hypochlorite solution (NaOCl) was applied for 60 s, rinsed for 30 s and blot-dried, leaving a moist surface. The Single Bond adhesive was applied and light cured for 10 s.

SB/PL. The same procedure was followed as for Group SB, except for the application of a resin liner (Protect Liner F—PL) after applying SB.

NaOCl/SB/PL. The same procedure was followed as for Group NaOCl/SB, except for the final application of Protect Liner F after pretreatment with a 10% NaOCl solution and application of SB.

Application of the resin liner Protect Liner F (Groups SB/PL and NaOCl/SB/PL) was standardized: the material was dispensed on a glass slab and then applied with a microbrush in a spiral movement starting from the bottom of the cavity and working towards the top. This procedure allowed the formation of a 0.2 mm thick layer of the liner at the cavity floor. At the lateral wall, the thickness of the liner was less than 0.05 mm. The resin liner was photoactivated for 20 s. Filtek Z250 composite, shade A3, was inserted in a single increment and light-cured for 20 s.

Afterwards, the specimens were stored in distilled water at 37°C for 24 h and then finished and polished in a water-cooled mechanical polisher (Minimet 1000; Buehler), using 600- and 1200-grid SiC sandpaper (Carbimet Disc Set, 305178180; Buehler).

Marginal and internal adaptation evaluation

In order to determine the marginal adaptation at the surface, a 1.0% acid red propylene glycol solution (Caries Detector[®], Kuraray Co., Osaka, Japan) was applied at the restoration margins for 5 s [20]. The specimens were then rinsed in tap water and gently blown dry. This technique stained the gaps so that they could easily be quantified. The cavity margins were evaluated using a stereomicroscope LEICA MZ6 (Leica Microsystems Ltd. Heerbrugg, Switzerland) at ×16 magnification. A digital image of each specimen was obtained at this stage. The length of dye-stained gaps along the cavity margins was measured (µm) from the images using the UTHSCSA Image tool software version 2.0 (alpha 2 – September 1997) developed by the Department of Dental Diagnostic Science at the University of Texas Health Science Center (San Antonio, Tx., USA). The length of the gap formed was calculated as a percentage of the entire margin length.

Table II. Percentage of gaps in internal adaptation test

Resin liner (Protect Liner F—PL)	Treatment of the substrate	
	Without NaOCl	With NaOCl
Without PL	18 (3.9) A a	18 (4) A a
With PL	12 (4.5) B a	21 (4) A b

Statistical differences are expressed in upper case letters in columns and lower case letters in rows ($p < 0.05$). Standard deviation given in parentheses.

In the second part of this study, after evaluation of the marginal adaptation at the surface, the restorations were cut in a cervical – occlusal direction, in slices (1 mm thick), using an ISOMET 1000 machine (Buehler) to obtain 3 to 4 slices of each restoration. Caries Detector[®] was applied on these slices to stain the dentin gaps and the same procedures described previously were accomplished for evaluation of internal adaptation of the composite restorations (Figure 1). The obtained data (internal adaptation) were submitted to ANOVA and Tukey tests at 5% significance.

Results

A perfect enamel marginal seal was observed in all groups. Concerning the internal adaptation, the ANOVA test detected a statistically significant difference among the groups ($p < 0.05$). The results, according to the Tukey test, are given in Table II.

There was no significant difference in the factor treatment of the substrate (with and without NaOCl) between the mean percentage for group SB—control and for group NaOCl/SB. However, when Protect Liner F was used, there was a significant difference between the mean percentage of specimens treated with NaOCl (group NaOCl/SB/PL) and those that were not (group SB/PL). Group NaOCl/SB/PL showed the worst internal adaptation, with the highest percentage of internal gaps, while group SB/PL showed the best.

There was a significant difference between the mean percentage for group SB and that for group SB/PL in the use of a resin liner, with a lower percentage of internal gaps when Protect Liner F was used. When the NaOCl was used, however, there was no significant difference between the specimens in which Protect Liner F was used (group NaOCl/SB/PL) and those in which it was not used (group NaOCl/SB).

Discussion

The presence of marginal gaps contiguous with composite restorations facilitates microleakage [5,6,10,21]. However, in vitro microleakage measurements have not been accepted as predictive of

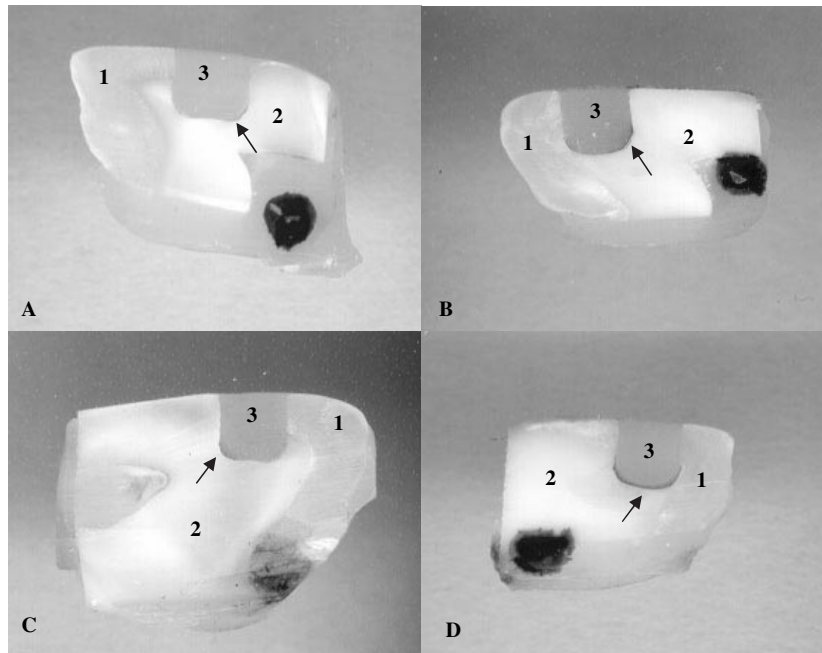


Figure 1. Typical digital image of dyed specimens. A. SB group; B. NaOCl/SB group; C. SB/PL group; D. NaOCl/SB/PL group. (1) Enamel; (2) dentin; (3) composite restoration; the arrows indicate marginal gaps stained by the Caries Detector[®]. Note that the stained gaps are mainly located in the axiopulpal angles of the cavity and also at the pulpal walls, areas subjected to the shrinkage stress. Also, it can be seen that NaOCl groups (B and D) showed more visible and distinct gaps.

restoration failure [21]. The presence of gaps is more reliable once this is considered the first sign of restoration failure. It can be clinically evidenced by marginal discoloration [22]. Identification of early marginal changes could facilitate the prognosis of the longevity/stability of the composite restorations.

The origin of the gaps could be related to three main factors: composite shrinkage stress [1], external stress (mechanical [23] and/or thermal [24]), and flaws during the bonding procedure [25]. In this study, the specimens were not submitted to any kind of external stress (mechanical or thermal), because the objective was to evaluate the gaps formed by the shrinkage stress alone.

All groups in this study showed a perfect enamel marginal seal, as also shown by Haak et al. [26]. This was because all the restoration margins were located in enamel, which has a highly mineralized structure and permits a more reliable bond in its use of an etch and rinse technique than that achieved by dentin [26].

Achieving a strong, durable seal to dentin has proved a great challenge to clinicians and researchers [6,9,10,25] because dentin has more complex structural components and properties compared to enamel [9,25], rendering the bonding procedure more susceptible to flaws in this substrate. Evaluation of the internal adaptation showed the presence of gaps in most of the cavities. The internal margins were mainly located in dentin and the gaps were specially situated at the pulpal wall and axiopulpal angles, which are stress concentration regions, and a problematic zone for the composite application (Figure 1).

The presence of gaps at the pulpal wall and axiopulpal angles causes fluid flow in the dentin tubules, resulting in typical postoperative sensitivity. It is important to enhance the adhesion between dentin and resin composite because such improved bond strength not only prevents postoperative sensitivity but also leads to better retention of the restorations [6,21].

Some studies have shown leakage at the hybrid layer [27,28]. This phenomenon might be due to hydrolytic degradation of the exposed collagen network not well infiltrated by the monomers creating a weak zone. This zone is vulnerable to degradation after long-term exposure to water [27,28].

One alternative method for obtaining dentin bonding is to remove collagen with a NaOCl solution after acid-etching and rinsing, since this alters the composition of the dentin surface, which could result in a more predictable substrate for bonding [16,29]. Pioch et al. [29] stated that removal of the collagen layer using sodium hypochlorite prevents the formation of nanoleakage.

Collagen removal can be performed using NaOCl gel or NaOCl solution. For this study, a 10% NaOCl solution was selected because according to Arias et al. [30] the NaOCl solution is more effective in completely removing the collagen fibrils than NaOCl gel, which seems more unstable. Thus, in this study the deproteinized groups did not present a hybrid layer [19,30].

Deproteinization improves bond strength, especially in the case of acetone-based adhesive systems [11,16,18], but this is a controversial issue, because

this improvement is not always observed, particularly when the ethanol/water-based adhesive systems are used [16,19,30].

Saboia et al. [31] showed that collagen removal may be important in reducing microleakage when an acetone-based adhesive system is used and has no influence on microleakage for ethanol/water-based adhesive systems. This concurs with Montes et al. [3], who found a significant increase in the percentage of excellent margins of restorations in cavities treated with NaOCl and restored using an acetone-based adhesive system.

However, enhanced adaptation to dentin cavity walls was not observed in this study. The results showed no significant difference between group SB (18%) and group NaOCl/SB (18%). This could have been because of the lower diffusion of the ethanol/water based adhesive system (SB) than that observed for the acetone-based adhesive systems. The SB was applied twice with a 5-s interval in between, after, lived for 15 s, observing a glossy surface. Perhaps this amount of time was insufficient to permit full diffusion of the monomer into the substrate in order to improve the bonding [32]. These results concur with those of Toledano et al. [33], who showed a similar gap formation in dentin margins regardless of whether a hybrid layer was present or not.

Alterations in the substrate due to dissolved and destabilized collagen fibrils could have brought about changes in the surface energy [34] and in the redox potential of the substrate [35], and may have inhibited better marginal adaptation on deproteinized specimens in this study. According to Daumer et al. [36], sodium hypochlorite disrupts the pyridinoline cross links that occur in collagen types I and II, with formation of chloramines and protein-derived radical intermediates. These reactive residual free radicals on deproteinized dentin may compete with the propagating vinyl-free radicals generated during light activation of the adhesive, resulting in premature chain termination and incomplete polymerization [35].

The type of adhesive system (solvent and monomer composition) and the competence of the operator can influence the results of different studies, because demineralization and deproteinization of the dentin are technique-sensitive procedures. Munksgaard et al. [8] reported that the low technique sensitivity of an adhesive might be linked to its ability to wet and adhere to collapsed collagen fibers and to the surface of the underlying mineralized tissue. They observed that all adhesives tested had higher or unaltered bond strength to hypochlorite-treated dentin as compared with normal etched dentin. These results are related to the type of solvent of each adhesive system. Acetone-based systems better facilitate the infiltration of the adhesive components and thus mediate a stronger bond to dentin, especially on deproteinized substrate.

It should be considered that the available adhesive systems are not optimal for adhesion to NaOCl-treated

dentin surfaces. The parameters of treatment as well as the constitution of primer materials should be optimized prior to clinical use of this technique [29].

Despite problems related to long-term durability of the hybrid layer, according to Uno & Finger [14] it functions as an inherent elastic buffering layer capable of absorbing the resin composite shrinkage stress. The hybrid layer has a lower modulus of elasticity than the restorative resin composite, consequently allowing better distribution of the stress [14].

Another way to manage polymerization shrinkage stress is to use "an elastic cavity wall", as proposed by Kemp-Scholte & Davidson [37]. The use of a liner has been proposed to improve the marginal adaptation of composite restorations [5,26].

The results of this study confirm the effectiveness of the use of a resin liner (Protect Liner F) showing the best internal adaptation in the group SB/PL (12%). This is because polymeric materials with lower modulus of elasticity exhibit a viscous flow when submitted to stress, showing plastic deformation [38]. So, when this "elastic" liner is applied on the cavity walls before the insertion of the restorative composite, the polymerization shrinkage stress might be partially absorbed by the liner. This can also reduce the stress that is applied to the tooth structure, allowing gap reduction and better adaptation to the cavity walls. However, the 16.25% reduction of the volume of restorative composite applied to the cavity cannot be ignored—it causes a reduction on polymerization shrinkage, provoking some decrease in contraction stress and also allowing better marginal adaptation.

On the other hand, when the resin liner was applied after the collagen was removed from the acid-etched dentin with NaOCl, the worst internal adaptation (group NaOCl/SB/PL -21%) could be seen.

Protect Liner F has a greater monomer content than restorative composites, presenting lower modulus of elasticity, but higher shrinkage level [2]. The higher polymerization shrinkage could generate higher stress at the resin liner-dentin interface. On demineralized substrate (Group SB/PL), this stress could be partially absorbed by the hybrid layer. However, on deproteinized substrate, without hybrid layer [19,30] (Group NaOCl/SB/PL), polymerization stress of the resin liner could not be entirely absorbed by this rigid substrate, producing more gaps in this group. Besides, the debonding area could be between adhesive system and deproteinized dentin.

According to Salim et al. [39], the debonding area was located at the adhesive system-dentin interface. In a SEM analysis, they observed a wide gap between the substrate and the adhesive system, Single Bond, in all the specimens that were deproteinized with a 10% NaOCl solution, indicating an adhesive failure.

In conclusion, the hypothesis tested in this work must be rejected. Use of a resin liner on deproteinized dentin does not improve internal adaptation.

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