Scanning Electron Microscopic Observation of the Effect of Calcium Hydroxide on the Adhesion of a Resin System to Dentin

SUMMARY

Objectives: The aim of the study was to evaluate the effect of calcium hydroxide on the adhesion of a resin system to dentin by scanning electron microscopy (SEM).

Methods: Cavities were prepared on the mesial and distal sides of 10 extracted lower human molars. The mesial cavities were coated with Prime & Bond 2.1 (Caulk, Dentsply) after total etching, and were restored with TPH composite resin (Caulk, Dentsply). The distal cavities were restored with calcium hydroxide (Dycal, Caulk), Prime & Bond 2.1 and TPH Hibrid composite resin, respectively. The teeth were then sectioned and analysed by SEM.

Results: Prime & Bond 2.1 adhered closely to dentin, forming a gap-free attachment with a hybrid layer in most of the specimens. However, in the cavities lined with calcium hydroxide, the polymerisation shrinkage of the resin composite caused the separation of the calcium hydroxide from the dentinal surface, forming 15 to 30 µm wide interfacial gaps.

Conclusion: The interposition of a layer of calcium hydroxide between dentin and adhesive could reduce the dentinal surface available for adhesion of the restoration and disrupt the continuous adhesion of the bonding agent to dentin.

Keywords: Calcium Hydroxide; Adhesive Systems; Adhesion

Introduction

For several decades, the development of adhesive systems has been limited by the belief that acids applied to dentin during restorative procedures cause pulpal inflammation. The use of bases and liners was considered essential to protect the pulp from the toxicity of restorative materials. For many years calcium hydroxide liners were traditionally used in routine restorations for pulp protection because of its antibacterial and therapeutic effect, stimulating dentin bridge formation. However, it has been later reported that microleakage of bacterial components at the cavo-surface margin through the dentinal walls was the primary cause of pulp inflammation rather than the disastrous effects of the material.

Recently, new adhesive systems with higher bond strengths have been introduced to improve the adhesion of restorative materials and to prevent microleakage and eventual pulpal irritation by forming a hybrid layer. These systems include acid etching of not only enamel, but also dentin, with complete removal of smear layer and plugs. Manufacturers have also introduced self-priming adhesive materials, which are claimed to completely infiltrate the etched dentin with resin in one clinical step.

Today, total acid etching of vital dentin and placement of adhesive systems are biologically accepted for routine clinical use. Vital dentin is now etched, and various adhesive systems are placed without utilizing traditional calcium hydroxide bases and liners. Several clinical studies have also reported successful results of direct pulp capping of exposed pulps with various adhesive systems. However, some authors have reported that total etching may increase pulpal irritation because etching procedures demineralise the dentin surface and exposes the collagen fibres. Thus, many clinicians still have some doubts about the toxicity and the sealing capacity of these systems and they prefer to
protect the pulp with intermediate materials, particularly with calcium hydroxide preparations.

The aim of this study was to evaluate the effect of a calcium hydroxide liner on the adhesion of resin system to dentin by scanning electron microscopy (SEM).

**Material and Methods**

10 non-carious, extracted human lower third molars were used in the present study. After the extraction, the teeth were placed in 4% thymol solution for 1 week, and then left in bi-distilled water until ready to use.

Cavities (2 x 2 x 2 mm) were prepared on the mesial and distal sides of the teeth at cementoenamel junctions with an inverted-cone diamond bur, using copious amounts of water spray. Mesial cavities of the teeth were acid etched with 36% phosphoric acid for 20 seconds, then rinsed off and gently dried. The cavities were coated with Prime & Bond 2.1 (Caulk, Dentsply, USA) and allowed to flow for 30 seconds. Then, they were thoroughly air dried for 5 seconds to maintain a uniform layer, and light-cured for 10 seconds. The cavities were filled with TPH composite resin (Caulk, Dentsply, USA) and cured for 40 seconds. The restorations were immediately refined using Soft-Lex disks (3M, St. Paul, Minnesota). The distal cavities of the same teeth were restored with a thin layer of self-hardening calcium hydroxide (Dycal, Caulk, Dentsply, USA) Prime & Bond 2.1 and TPH composite resin, respectively, as described above.

The teeth were embedded in a self curing epoxy resin and sectioned into 4 equal parts mesio-distally and bucco-lingually along the longitudinal axis passing through the centre of the restorations with a #314 bur. Section surfaces were ground flat with wet silicon carbide paper, in decreasing grits (240, 400, and 600). Then, they were cleaned with 37% phosphoric acid for 10 seconds and 2.5% NaOCl for 30 seconds, respectively, to remove the smear layer and visualize resin tags and hybrid layer. After rinsing with distilled water for 60 seconds and drying, they were mounted on metallic stubs. Ethyl alcohol was not used for dehydration to eliminate the risk of debonding of Prime & Bond 2.1. Specimens were dehydrated in a desiccator containing phosphorus pentoxide for 1 week and then coated with gold (Polaron SC 502, Fisons). They were observed with a scanning electron microscope (Jeol, JSM-5200) and images were taken from the representative areas.

**Results**

The adhesion of Prime & Bond 2.1 to dentin, resin composite and enamel was excellent in all specimens, with a hybrid layer of 5 µm in thickness. The residual dentin surface appeared as a clean and homogenous surface with several collagen fibrils forming the scaffold of the dentin surface (Figs. 1 and 2). Adhesion of the bonding agent to cavity floor was superior in comparison with cavity corners and walls (adhesive pool). It was observed that the thickness of the adhesive at the cavity corners and on the walls was not uniform, 0-60 µm and 0-15 µm, respectively (Fig. 3). In 2 specimens, debonding of the adhesive along the dentinal surface was approximately 10-15 µm while the other specimens were gap-free (Fig. 4).
In all cavities lined with calcium hydroxide, polymerisation shrinkage of the resin composite caused detachment of the calcium hydroxide from the dentinal surface. The thickness of calcium hydroxide was 150-300 µm and had a granular structure. In addition, the thickness of the gaps was 15-30 µm and patent dentin tubules were observed (Fig. 5). The relationship between the resin composite and the dentinal surface in the outermost areas where calcium hydroxide was not applied were also examined. Excellent adhesion of the composite resin-adhesive complex to the dentin, mediated by a hybrid layer was evident.

Discussion

Good marginal adaptation of restorative materials is very important and the reason of pulpal irritation after the placement of a dentin-bonding agent is due to separation or debonding of the resin from dentin, followed by the passage of bacteria and their toxins to the pulp tissue. In the present study, it was demonstrated that the polymerisation shrinkage of the resin composite caused detachment of the calcium hydroxide, leading to the formation of an interfacial gap between calcium hydroxide and dentin in all of the specimens treated with calcium hydroxide. According to Brannström et al, such a microfissure would be quickly colonized by pulpal fluids, and because of inward and outward fluid movement, most of the calcium hydroxide paste would disappear with time. It was also reported that calcium hydroxide did not constitute an adequate long-term protection in the presence of a marginal defect because it did not present an obstacle to the passage of bacteria or their metabolic products to the pulp. In addition, it occupied some of the area that could have been used for dentinal bonding. Therefore, it could be suggested that the application of calcium hydroxide underneath resin composite restorations sometimes may be harmful if it leads to the passage of bacteria and their products. This result was in agreement with other studies, which reported that calcium hydroxide placed under resin composite with a bonding agent tended to be pulled away from the cavity surface, leaving a gap between the lining and the dentin. Furthermore, Reinhardt and Chalkey found that calcium hydroxide did not adhere to the smear layer or to the dentinal tubule complex.

In contrast, in the zones in which the adhesive had been applied in direct contact with the dentin, a well-developed resin-impregnated zone (hybrid layer), ranging from 4 to 6 µm in depth and covered by an uniform adhesive layer (approximately 30 µm), was generally observed in the present study. Thus, a chemical and micromechanical bond was obtained, based on both the penetration of resin tags inside the tubules and the impregnation of the collagen fibres by the adhesive monomer. Nakabayashi et al indicated that this demineralised dentin-resin inter-diffusion zone was acid resistant and had the double function of binding the restoration to the dentin and rendering it caries resistant. It has been also suggested that the hybrid layer provides with a coefficient of elasticity that contributes
to the maintenance of the bond distributing the stress uniformly\textsuperscript{21}.

**Conclusion**

It could be suggested that the interposition of a layer of calcium hydroxide between dentin and adhesive would reduce the dentinal surface available for adhesion of the restoration and would disrupt the continuous adhesion of the adhesive system to dentin. Further clinical studies are needed to determine the effect of calcium underneath the bonding system on the success of resin composite restoration.

**References**


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