

## The Influence of Some Dentin Primers on Calcium Hydroxide Lining Cement

Alaa El-Araby, BDS, MSc, PhD; Abed Al-Jabab, BDS, MS, PhD



### Abstract

Dentin primer is applied as a routine procedure prior to bonding to improve the sealing properties of direct polymerizing resins. Some primers contain acetone or alcohol that may affect the properties of calcium hydroxide liner which is placed as a direct or indirect pulp cap. If calcium hydroxide is softened or smeared over the cavity walls, the bonding will be impaired. Therefore, if this occurs, the cement must be removed, the walls must be cleansed, and the procedure must be repeated with careful application of dentin primer.

**Objective:** The purpose of this study was to determine the wear and compressive strength of a calcium hydroxide liner after exposure to different kinds of dentin primers for different periods of time.

**Methods:** The calcium hydroxide used in this study was Dycal™. It was mixed according to the manufacturer's instructions and placed in plastic rings of 0.5 mm x 5 mm and allowed to set at 37°C for 15 min under 500 gm load. To determine erosion, the height for each sample before and after application of primers was recorded using a Digital Height Measuring Instrument "Digmar" 817. Compressive strength specimens were also prepared.

**Results:** Calcium hydroxide treated with Optibond (alcohol based) or Syntac (acetone based) for 1 min or 5 min had the highest erosion values and the lowest compressive strength values. Gluma CPs (water based primer) had the least effect on calcium hydroxide values.

**Keywords:** Dentin primers, calcium hydroxide, compressive strength, erosion value

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## Introduction

Calcium hydroxide liners are often placed in deep cavities under restorative materials to protect the pulpal tissues from chemical insults. Calcium hydroxide has the ability to stimulate restorative dentin formation with direct pulp contact. It also serves as a protective barrier for pulp tissue not only by blocking patent dentinal tubules, but also by neutralizing the attack of inorganic acids and leached products from certain cements and restorative materials.<sup>1</sup> Conventional formulations of calcium hydroxide demonstrate low physical properties because it has a low modulus of elasticity.<sup>2</sup>



Although calcium hydroxide has been used for many years as a dental base or pulp capping material, no specific information exists in the literature on the effect of dentin primers or dentin adhesives that may contain acetone, alcohol, or water on the properties of hard-set calcium hydroxide.

The effect of acid solubility on calcium hydroxide is considered an important property of the material because accidental contamination can occur during the acid-etch technique.<sup>3</sup> It is highly desirable that calcium hydroxide over the dentin does not dissolve in the etching solution.<sup>4</sup>

Bruk and Watts<sup>5</sup> found calcium hydroxide lost a significantly greater percentage of its mass following phosphoric acid etching and washing cycles. McComb<sup>6</sup> reported Dycal<sup>TM</sup> exhibited pronounced solubility in water, while other types of calcium hydroxide remained fairly resistant to acid attack.

Dentin primer is applied routinely prior to bonding to improve the sealing properties of direct polymerizing resins. Some dentin bonding agents

or primers contain water, acetone, or alcohol that may affect the properties of calcium hydroxide. If dentin bonding agents, or dentin primers are vigorously applied in the cavity without caution, calcium hydroxide can be softened or smeared over the cavity wall and the bonding to dentin will be impaired. Therefore, the cement must be removed, the wall must be cleansed, and the procedure should be repeated with careful application of a dentin primer.

The aim of this study was to determine the surface erosion and compressive strength of a calcium hydroxide liner after exposure to different types of commercially available dentin primers or dentin adhesives for different periods of time.

## Materials and Methods

The materials used in this study are listed in Table 1.

Calcium hydroxide was supplied in 2 collapsible tubes; one containing the base and the other, catalyst. The material was proportioned according to the manufacturer's directions by dispensing equal lengths of pastes.

## Erosion Test

After mixing, the material was placed in plastic rings with an inner diameter of 5 mm and a 0.5 mm thickness. The filled ring was placed between 2 glass plates with 500 gm applied to produce a smooth surface and to extrude the excess material. The calcium hydroxide was allowed to set in an incubator at 37°C. After 15 minutes, the ring was separated from the glass plates and the excess material was trimmed with a scalpel. After preparation of the test specimens, they were divided into 7 groups (15 specimens each) according to the different primers used, for a total of 105 test specimens. Each group was subdivided into 3 subgroups (5 specimens each) according to the primer application time of 1 min, 5 min, or 60 min. Each test specimen was immersed in 0.5 ml of the required dentin primer in a tight glass container, placed in a dark box for the required period, washed with water spray for 1 min, and then dried with oil free air.<sup>7</sup>

The amount of material lost (surface erosion) was measured in micrometers using a Digital Height Measuring Instrument "Digmar" 817. A measuring

Table 1. Materials used in this study.

Materials	Code	Batch No.	Content	Manufacturer
Dycal™	-	623451	N/A	Dentsply Caulk , Milford,DE,USA
All-Bond® 2 primer	AB	B-2600AM	acetone-based	Bisco Itasco. Inc
Scotchbond® multipurpose	SM	5933MP	water-based	3M Dental Products,St.Paul,USA
Optibond® primer	OB	405058	alcohol-based	Kerr Manufacturing Co.
Singlebond	SB	8004SB	water-based	3M Dental Products, St.Paul,USA
Syntac® single component	SY	546978NN	acetone-based	Vivadent,Schaan, Liechtenstein
Gluma CPS	GCPS	115618	water-based	Heraeus,Kulzer Dormagen, Germany
Prime & Bond® NT®	PB	0105001139	acetone-based	Dentsply. DeTrey. GmbH, Dreieich.

probe with a spherical contact point of 2 mm in diameter was used for inside measurement. The readings before and after application of the different primers were recorded.

### Compressive Strength Test

The specimens were prepared in a split-brass mold with internal dimensions of 12 mm height and 6 mm diameter (American Dental Association - ADA Specification No. 30). The mold was placed on a flat glass plate covered by a thin polyethylene sheet and slightly overfilled with a portion of material within 3 min after commencing the mix. A second flat glass plate and polyethylene sheet were pressed on the top of the mold and held together with a C-clamp. Three minutes after the start of the mix, the mold assembly was transferred to an incubator held at 37°C. One hour later, the ends of the cylinders were ground flat with 240-grit silicon carbide metallographic paper. The specimens were removed from the molds and kept at near 100% relative humidity at 37°C for 24 hours. The control group (without application of any primer) consisted of five specimens. The other test specimens were divided into 7 groups (15 specimens each) according to the different primers used. Each group was subdivided into 3 subgroups (5 specimens each) according to the application time of 1 min, 5 min, or 60 min. Each test specimen was immersed in 0.5 ml of the required dentin primer in a tight glass container and placed in a dark box for the required period. Specimens were then washed with water spray for 1 min and dried with oil free air.<sup>3</sup>

The specimens were loaded in compression at a cross head speed of 0.05 in/min on a universal testing machine Instron 8500 (Instron Corp, Canton, MA) using a 1,000-lb load cell. The value for compressive strength was reported as the average of five specimens.

### Results

#### Erosion Test

The data were subjected to a two way analysis of variance (ANOVA) with the post-hoc Tukey test to compare the means and to locate the significant differences. The mean erosion values after application of different primers for the seven test groups can be seen in Figure 1 and Table 2.

The ANOVA test indicated there were significant differences in the erosion values obtained after application of different primers to calcium hydroxide ( $p < 0.0001$ ). In addition a significant interaction effect was noted between different primers and different periods.

There was significant variation among different time periods ( $p < 0.0001$ ). The amount of erosion was increased when increasing the application time of different primers.

The post-hoc Tukey test for multiple comparisons of data indicated the test group treated with Optibond for 1 min or 5 min had the highest mean erosion value (0.04 or 0.036  $\mu\text{m}$ , respectively) and was significantly different from other test groups ( $p < 0.05$ ). There was no significant difference

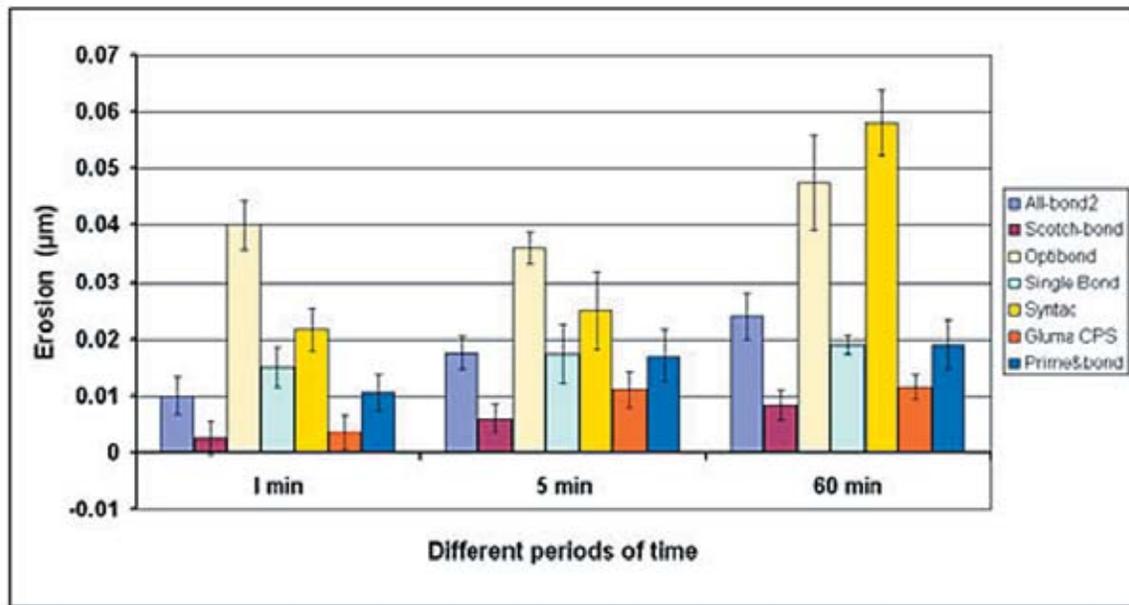


Figure 1: Mean erosion values of Dycal™ after application of different primers for different periods.

Table 2. Mean erosion values of Dycal™ after application of different primers for different periods.

Primers	Mean ± S.D. (µm)		
	1 min	5min	60min
All-Bond 2	0.01 ± 0.0032 *	0.0176 ± 0.0029 *	0.024 ± 0.004 *
Scotch-bond	0.0026 ± 0.0031 *	0.006 ± 0.0025 *	0.0083 ± 0.0026 *
Optibond	0.04 ± 0.0043	0.036 ± 0.0028	0.0435 ± 0.0085
Singlebond	0.015 ± 0.0035 *	0.0174 ± 0.0053 *	0.019 ± 0.0017 *
Syntac	0.0217 ± 0.0038	0.025 ± 0.0068 *	0.058 ± 0.0058
Gluma CPS	0.0036 ± 0.0031 *	0.0111 ± 0.0032 *	0.0115 ± 0.0021 *
Prime & Bond NT	0.0105 ± 0.0031 *	0.017 ± 0.0046 *	0.019 ± 0.0044 *

Stars (\*) denote no significant difference within each column.

between other test specimens treated with different primers for 1 min or 5 min as marked with stars (\*) in Table 2 ( $p > 0.05$ ). There was no significant difference between the erosion values obtained from the test groups treated with Optibond or Syntac for 1 min ( $p = 0.055$ ). On the other hand, the erosion values of test specimens treated with Scotch-bond was significantly lower than those treated with Optibond and Syntac for 1 min ( $p < 0.0001$ ). Optibond and Syntac had the highest erosion values after 1 min and 5 min.

The test group treated with Syntac for 60 min had the highest mean erosion value ( $0.058 \mu\text{m}$ ) and was significantly different from other test groups ( $p < 0.05$ ). There was no significant difference between the erosion values obtained from the test

groups treated with Optibond or Syntac for 60 min ( $p = 0.914$ ). There was no significant difference between other test groups as marked with stars (\*) in Table 2 ( $p > 0.05$ ). On the other hand, the groups treated with Scotch-bond or Gluma CPS were significantly different from those treated with Optibond or Syntac for 60 min ( $p < 0.05$ ). It was noted Scotch-bond and Gluma CPS had the least effect on calcium hydroxide.

#### Compressive Strength Test

The compressive strength of the control group was  $12.15 \pm 1.3$ . The results of the compressive strength test for calcium hydroxide after treatment with different primers at 1 min, 5 min, and 60 min periods are shown in Figure 2 and Table 3.

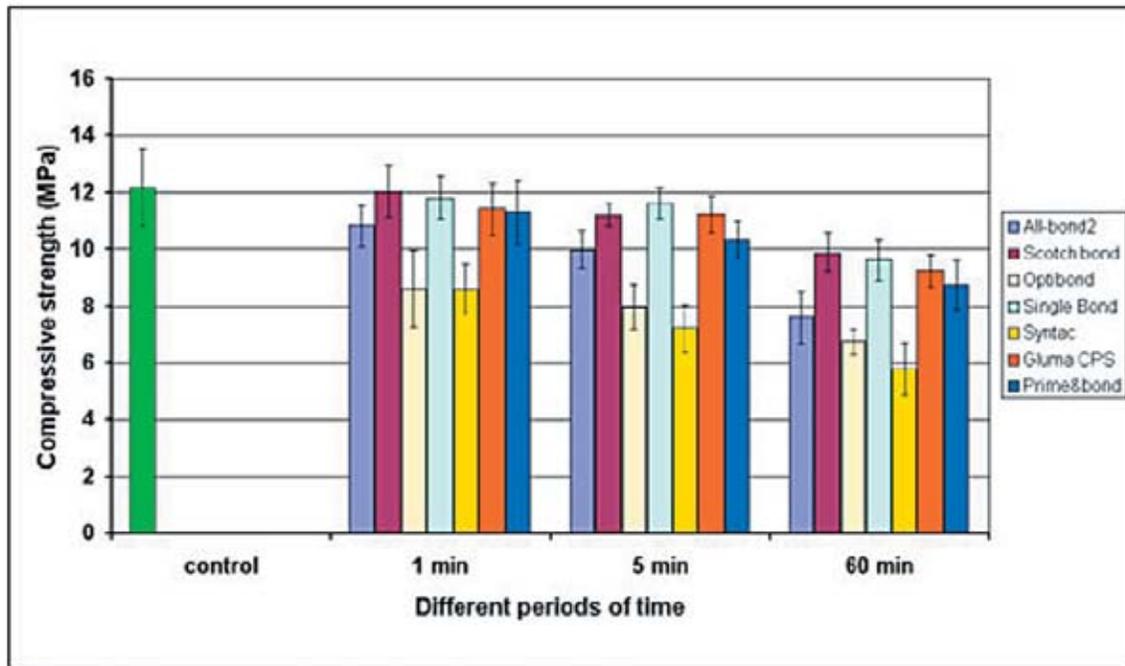


Figure 2: Mean compressive strength values of different test groups.

Table 3. Mean compressive strength values of Dycal™ after application of different primers for different periods.

Primers	Mean ± S.D. (MPa)		
	1 min	5 min	60 min
All-Bond 2	10.81 ± 0.73 *	9.95 ± 0.66 *	7.58 ± 0.91 A
Scotch-bond	12.01 ± 0.91 *	11.18 ± 0.41 *	9.86 ± 0.68 *
Optibond	8.61 ± 1.35	7.95 ± 0.8	6.75 ± 0.43 A
Singlebond	11.81 ± 0.77 *	11.6 ± 0.55 *	9.61 ± 0.72 *
Syntac	8.57 ± 0.88	7.21 ± 0.81	5.78 ± 0.91
Gluma CPS	11.4 ± 0.92 *	11.21 ± 0.66 *	9.23 ± 0.59 *
Prime & Bond NT	11.3 ± 1.13 *	10.3 ± 0.66 *	8.73 ± 0.85 *

Stars (\*) denote no significant difference within each column. Means of same letters were not significantly different.

ANOVA showed a significant difference among different test groups and a significant difference in the mean compressive strength values at different periods of time ( $p < 0.0001$ ). The compressive strength of calcium hydroxide decreased with increasing application time of different primers. The post hoc-Tukey test was performed for multiple comparisons.

At 1 min, the means of compressive strengths of different test groups treated with different primers were not significantly different as marked with stars (\*) in Table 3 ( $p > 0.05$ ). The groups of calcium hydroxide treated with Optibond or

Syntac were significantly lower than the control group and those groups treated with different primers. There was no significant difference in the mean compressive strength between the groups treated with Optibond and Syntac ( $p = 1$ ). The mean compressive strength values were 8.6 MPa and 8.5 Mpa, respectively. There was no significant difference in the mean compressive strength values between the control group and those groups treated with Scotch-bond or Gluma CPS.

At 5 min, there was no significant difference in compressive strength values between test groups

treated with Optibond and Syntac ( $p=0.52$ ). The mean compressive strength values were 7.9 MPa and 7.2 MPa, respectively. There was a highly significant difference in compressive strength values between the test groups treated with different primers and the groups treated with Optibond or Syntac ( $p<0.05$ ).

At one hour, the mean compressive strength of calcium hydroxide treated with Syntac was the lowest, and it was significantly lower than other test groups. Although, there was no significant difference between groups treated with All-Bond 2 and Optibond ( $p=1$ ), they were significantly lower than other test groups.

### Discussion

The solubility of calcium hydroxide in phosphoric acid has been studied by many investigators.<sup>3,5,7</sup> The acid solubility of calcium hydroxide is considered an important property because of accidental contamination during the acid-etch technique.<sup>7</sup> The high solubility of calcium hydroxide may result in contamination of the bonding agent and increased marginal leakage.<sup>8</sup>

In this study surface erosion of calcium hydroxide was evaluated after application of different dentin primers.

The calcium hydroxide used in this study was Dycal™, which contains calcium 1-methyl trimethylene disalicylate as an ester and a mixture of ortho and para N-ethyl toluene sulphonamide as a plasticizer.

Posser et al.<sup>9</sup> found calcium hydroxide was hydrolytically unstable, releasing calcium and hydroxide ions when in contact with water. These allow free passage of water, which then attacks vulnerable cement structure, leading to disintegration of these cements, and this exerts a considerable influence on the physical properties. The rate of erosion is controlled by plasticizer. Like all other dental cements, calcium hydroxide is set by an acid-base reaction as defined by Wilson.<sup>10</sup>

An infra-red spectroscopy study by Posser et al.<sup>11</sup> showed Dycal is set by an acid-base

reaction between alkyl salicylate and calcium hydroxide. During the course of cement formation, phenolic protons were replaced by calcium ions to form a chelate structure of calcium phenolate. The weakness of and friability of the cement suggested chelates are bound together only by secondary attractions.

Barnes<sup>12</sup> observed the loss of calcium hydroxide under amalgam restoration in four clinical cases. Phillips<sup>13</sup> suggested calcium hydroxide bases become soft when using a water coolant during removal of amalgam from a cavity preparation and indicated calcium hydroxide was sensitive to the base-catalyst ratio.

Chong<sup>14</sup> reported there was no difference in the compressive strength of calcium hydroxide after seven min and 24 hours. This fact is important because the restorations would be placed within seven min after starting the mix. Shazad et al.<sup>15</sup> found calcium hydroxide had sufficient compressive strength 3 min after mixing to withstand the force of condensation of a restoration. In this study compressive strength was measured after one, five, or sixty min.

The results of this study showed calcium hydroxide responded differently upon the attack of different primers. It is interesting to note primers are not similar in composition. They are classified according to their contents into water-based, acetone based, and alcohol-based.

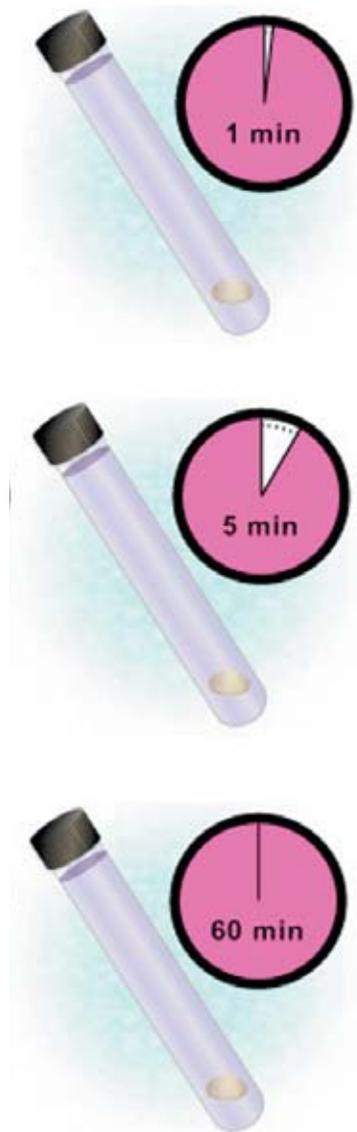
Calcium hydroxide treated with Syntac or Optibond showed higher erosion values and lower compressive strength in comparison to the other specimens treated with other primers. Syntac is a self etching primer which contains a high concentration of acetone (74%) and maleic acid (10%). This accounts for high erosion values and low compressive strength as acetone and maleic acid can penetrate through the cement, attack the chelate structure, dissolve the plasticizer, and cause disintegration of the cement. This is also the case with All-Bond 2 which contains acetone (60%) and alcohol (10%). It was noted acetone was more aggressive than alcohol in disintegration of the cement after one hour, and calcium hydroxide was susceptible to the attack of acetone or alcohol contained in some primers.

Other test specimens treated with Scotchbond, Singlebond, and Gluma CPS showed lower erosion values and higher compressive strength values than that obtained after application of All-Bond 2, Optibond, or Syntac. These results could be related to the composition of different primers used. It has been known Scotchbond and 3M Singlebond are water-based containing an aqueous solution of hydroxyethyl methacrylate (HEMA), Gluma CPS is water-based containing HEMA and glutaraldehyde. For this reason, Scotchbond, Singlebond, and Gluma CPS have the least effect on the properties of calcium hydroxide. Washing of test specimens with water spray might also cause dissolution of the superficial layer of the test specimens.

### Conclusion

The amount of surface erosion of calcium hydroxide was increased with increasing the application time for different dentin primers. Scotchbond primer or Gluma CPS primers had the least effect on calcium hydroxide. The calcium hydroxide treated with Optibond or Syntac for 1 min or 5 min had the highest erosion values and the lowest compressive strength values. At 60 min, the highest amount of erosion was recorded for test specimens exposed to Syntac.

The results of this study point to the important role of dentin primer on the erosion of calcium hydroxide cement bases and the efficacy of sealing the restoration. Dentin primers or dentin adhesives must be applied very carefully over calcium hydroxide.



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## About the Authors

**Alaa El-Araby, BDS, MSc, PhD**



Dr. El-Araby is an Assistant Professor in the Division of Dental Biomaterials Science, Department of Restorative Dental Sciences of the College of Dentistry at King Saud University in Riyadh, Saudi Arabia. He is a member of the Japanese Society of Dental Materials and Devices, the Saudi Dental Society, and the Academy of Dental Materials. His research interests include the field of metallurgy as well as the fields of adhesive dentistry and polymers.

**Abed Al-Jabab, BDS, MS, PhD**



Dr. Al-Jabab is an Assistant Professor and Head of the Division of Dental Biomaterials Science, Department of Restorative Dental Sciences of the College of Dentistry at King Saud University in Riyadh, Saudi Arabia. He also serves as the Director of the Research Center at that institution. Dr. Al-Jabab is a member of the Editorial Board of the Saudi Dental Journal, the International Association for Dental Research, the Saudi Dental Society, and the Minerals, Metals and Materials Society. His research interests include the study of titanium alloys, the corrosion of alloys, dentin bonding agents, and dental composites.