The effect of some dentin bonding agents on Dycal lining cement

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يستخدم المحضر العاجي كخطوة وتوريه قبل عملية الألصاق لتحسين خواص الاحكام للحشوات الراتنجيه المبلمرة. تحتوي بعض المحضرات العاجيه على مذيبات مثل الكحول والاسيتون التي قد يكون لها تأثير سلبي يعيق عمليه الصاق مادة الحشو مع العاج السني. لذلك من الضروري أزالة الاسمنت وتنظيف الجدران وإعاده العملية باستعمال المحضر العاجي تحاذر. الغرض من هذا البحث هو تعيين مقاومة الانضغاطات و درجة أهتراء المبطن كالسيوم هيدروكسيد (الدايكال) بعد التعرض لانواع مختلفة من المحضرات العاجية لفترات زمنية مختلفة الوقت. الطرق: تم تحضير خمس عينات من كل مجموعة وتم خلط مادة الكالسيوم هيدروكسيد حسب توصيات المصنع. ثم بعد ذلك تم وضعها في حلقات من البلاستيك ذات قطر ٥مم وارتفاع ٥,٠مم وتركها عند درجة ٣٧ مئوية لمدة ١٥ دقيقة تحت وزن ٥٠٠ جرام. ثم بعد ذلك قياس كل درجة تآكل عينه قبل وبعد المعالجة بالمحضرات السنية وذلك باستخدام جهاز الطول الرقمي لتحديد. كما تم أيضاً تحضير عينات مقاومة الانضغاطات النتائج: أثبتت النتائج أن أعلى قيمة اهتراء كانت للعينات التي تعرضت للمهيئ العاجي الاوبتي بوند و السينتاك . أما بالنسبه للعينات التي تم معالجتها بالمهيئ العاجي حلوماسي بي اس التي تحتوي على الماء كانت لها أقل تأثير على الأسمنت المبطن(الدايكال) يستخدم المحضر العاجي كخطوة وتوريه قبل عملية الألصاق لتحسين خواص الاحكام للحشوات الراتنجيه المبلمرة.

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 capping. If calcium hydroxide is softened or smeared over the cavity walls, the bonding will be impaired. Therefore, the cement must be removed, the walls must be cleansed and the procedure must be repeated with careful application of dentin primer. The purpose of this study was to determine the wear and compressive strength of a calcium hydroxide liner (Dycal) after exposure to different kinds of dentin primers for different periods of time. Dycal 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 minutes under 500 gm load. To determine erosion, the height for each sample before and after application of primers was recorded using Digital Height Measuring Instrument (Digmar 817). Compressive strength specimens were also prepared. Dycal treated with Optibond or Syntac for 1 minute or 5 minutes had the highest erosion values and the lowest compressive strength values. Gluma CPs (water based primer) had the least effect on Dycal.

Introduction

Calcium hydroxide liners are often placed in deep cavities under restorative materials to protect the pulpal tissues from chemical insults. It 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 Testorative materials.[1] Conventional formulations of calcium hydroxide demonstrate low physical properties.[2]

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-setting calcium hydroxide.

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

Bruk and Watts[5] found that Dycal lost a significantly greater percentage of its mass following phosphoric acid etching and washing cycles. McComb[6] reported that Dycal 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 spill over into the cavity, 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 cleansed and the procedure should be repeated with careful application of dentin primer.

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

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Table 1. Materials used In the study

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

Dycal was supplied in 2 coMapsible tubes, one of which is the base and the other is the catalyst. The material was proportioned according to the manufacturer's directions by dispensing equal lengths of pastes.

Materials and Methods

The materials used in the study are listed in Table1.

Erosion Test

After mixing, the material was placed in plastic rings with an inner diameter of 5 mm and 0.5 mm thickness. The filled ring was placed between 2 glass plates with 500 gm applied to produce smooth surfaces 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 of 15 specimens each according to the different primers used with a total of 105 test specimens. Each group was subdivided into 3 subgroups (5 specimens each) according to the primer application time of 1 minute, 5 minutes or 60 minutes. Each test specimen was immersed in 0.5 ml required dentin primer in a tight glass container, placed in a dark box for the required period and then washed with water spray for 1 minute and dried with oil free air.

The amount of material lost (surface erosion) was measured in micrometers using a Digital Height Measuring Instrument "Digmar" 817. A measuring probe with 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 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 three minutes 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 consisted of five specimens which did not receive any primer. The other test specimens were divided into 7 groups according to the different primers used. Each group was subdivided into 3 subgroups (5 specimens each) according to the application time of 1 minute, 5 minutes or 60 minutes. Each test specimen was immersed in 0.5 ml required dentin primer in a tight glass container, placed in a dark box for the required period. Specimens were then washed with water spray for 1 minute and dried with oil free air.[3]

The specimens were loaded in compression at a crosshead speed of 0.05 in/min on a Universal Testing Machine Instron 8500* each 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 two-way analysis of variance with 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 were shown in Figure 1 and Table 2, respectively.

The ANOVA test indicated that there was a significant difference in the erosion values

* Instron Corp; Canton, Mass., USA

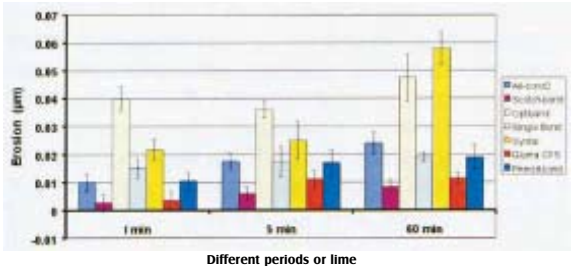

Fig.1. Mean erosion values of Dycal after application of different primers for different periods of time.

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

Primers	Mean ±S.D. (urn)		
	1 min	5 mins	60 mins
All-bond2	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 ± .0058
GlumaCPS	0.0036 ±0.0031*	0.0111 ±0.0032*	0.0115 ±0.0021 *
Prime&Bond	0.0105 ±0.0031*	0.017 ± 0.0046 *	0.019 ± 0.0044 *

Stars * denote no significant difference within each column.

obtained after application of different primers to Dycal (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 the application time of different primers increased.

The post-hoc Tukey test for multiple comparisons of data indicated that the test group treated with Optibond for 1 minute or 5 minutes had the highest mean erosion value (0.04 or 0.036 µm, respectively) and was significantly different from other test groups (P < 0.05). There was no significant difference between other test specimens treated with different primers for 1 minute or 5 minutes as shown 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 minute *(P* = 0.055). On the other hand, the test specimens treated with Scotch-bond was significantly lower than those treated with Optibond and Syntac for 1 minute (P< 0.0001). Optibond and Syntac had the highest erosion values after 1 minute and 5 minutes.

The test group treated with Syntac for 60 minutes had the highest mean erosion value (0.058 M.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 minutes (P= 0.914). There was no significant difference between other test groups as marked with starsf) 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 minutes (P < 0.05). It was noted that Scotch-bond and Gluma CPS had the least effect on Dycal.

Compressive Strength Test

The compressive strength of the control group was 12.15 + 1.3. The results of the compressive strength test for Dycal after treatment with different primers at 1 minute, 5 minutes and 1 hour periods are shown in Figure 2 and Table 3.

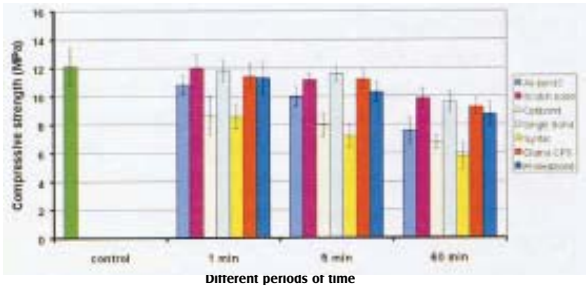

Fig. 2. Mean compressive strength values of different test groups.

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

Primers	Mean ± S.D. (MPa)		
	1 min	5 mins	60 mins
All-bond2	10.8 ± 0.73*	9.95 ± 0.66	7.58 ± 0/91 A
Scotch-bond	12.0 ± 0.91*	11.18 ± 0.41*	9.86 ± 0.68*
Optibond	8.61 ± 1.35	7.95 ± 0.8	6.75 ± 0.43A
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	11.3 ± 1.13*	10.3 ± 0.66*	8.73 ± 0.85 *

Stars * denote no significant difference within each column. A denote means were not significantly different.

Two-way analysis of variance 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 Dycal decreased when the application

time of different primers increased. The post hoc Tukey test was performed for multiple comparisons.

At one minute, 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 Dycal 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 minutes, 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 Dycal 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.[3-5,7] The acid solubility of calcium hydroxide was considered an important property because of accidental contamination during the acid-etch technique.[7] High solubility of calcium hydroxide may result in contamination of bonding agent and increased marginal leakage.[8]

In this study, surface erosion of calcium hydroxide was evaluated after application of different dentin primers. The calcium hydroxide used was Dycal, which contains calcium 1-methyl trimethylene disalicylate as an ester and a mixture of ortho & para N-ethyl toluene sulphonamide as a plasticizer.

Posser *et al.*[9] found that Dycal 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 was controlled by the plasticizer. Like all other dental cements, Dycal set by an acid-base reaction as defined by Wilson.[10]

An infrared spectroscopy study by Posser *et al.*[11] showed that Dycal 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 that chelates are bound together only by secondary attractions.

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

Chong[14] reported that there was no difference in the compressive strength of Dycal after seven minutes and 24 hours, respectively. This fact is important because the restorations would be placed within seven minutes after starting the mix. Shazad *et al.*[15] found that Dycal had sufficient compressive strength 3 minutes after mixing to withstand the force of condensation of a restoration. In this study, compressive strength was measured after 1,5, or 60 minutes.

The results of this study showed that Dycal responded differently upon the attack of different primers. It is interesting to note that primers are not similar in composition. They are classified as water-based, acetone- based, and alcohol-based primers.

Dycal 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 that acetone was more aggressive than alcohol in the disintegration of the cement after one hour and that Dycal was susceptible to the attack of acetone or alcohol that is contained in some primers.

Other test specimens treated with Scotchbond, Single bond, 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. Scotchbond, 3M Single bond are water-based, containing an aquous solution of HEMA. Gluma CPS is water-based containing HEMA and glutraldehyde. For this reason, Scotchbond, Single bond, and Gluma have the least effect on the properties of Dycal. Washing of test specimens with water spray might also cause dissolution of the superficial layer of the test specimens.

Conclusions

1. The amount of surface erosion of Dycal increased when the application time for different dentin primers increased.
2. Scotchbond primer or Gluma CPS primers had the least effect on Dycal.
3. The calciumhydroxide treated with Optibond or Syntac for 1 minute or 5 minutes had the highest erosion values and the lowest compressive strength values.
4. At 60 minutes, the highest amount of erosion was recorded for test specimens that were exposed to Syntac.
5. 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.
6. Dentin primers or dentin adhesives must be applied very carefully over Dycal.

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