THE GLUMA SYSTEM FOR BONDING TO DENTIN: EFFECT OF ACIDIC PRETREATMENT AND SURFACE WETNESS ON BOND STRENGTH

SISTEMA GLUMA DE ADESÃO DENTINÁRIA: EFEITO DO PRÉ-TRATAMENTO ÁCIDO E DA UMIDADE DA DENTINA.

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The aim of this study was to investigate the bond to dentin of a resin composite promoted by the Gluma bonding system applied to dry or wet dentin surfaces. The bonding system was modified as regards the conditioner and the composition of the primer. Various acidic conditioners were used, and the primer was modified by addition of water and acetone. Bond strengths between 12 and 22 MPa were recorded. It was concluded that bonds of good strength can be obtained with 20% phosphoric acid as pretreatment. It was further concluded that "moist" dentin does not interfere with bonding, but that "wet" dentin may reduce the bond strength about 25%.

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Uniterms: Bond strength; Dentin bonding agent; Restorative dentistry.

INTRODUCTION

Etching of dentin with phosphoric acid was introduced by Fusayama and coworkers in 1977 and described in an early study on dentin bonding (FUSAYAMA et al.[6], 1979). More recently, numerous studies have reported clinical success with adhesive resins applied to dentin eched with phosphoric acid (FUSAYAMA[7], 1980; BERTOLOTTI[3], 1990, KANCA[10], 1990). The so-called all-etch techniqe, according to which both enamel and dentin are etched with phosphoric acid, represents a significant simplification of the restorative procedures involving resin composites.

Many manufacturers of dentin-bonding systems indicate that dryness of dentin is critical for optimal performance of their product (KANCA[12], 1992). But vital dentin is inherently wet and thorough dessication is very difficult to achieve clinically (NORDENVALL; BRÄNNSTRÖM[15], 1980). On the other hand, many studies have demonstrated that some adhesive systems provide stronger bonds to both enamel and dentin when the tooth surface is not excessively dried but rather in a moist state. (KANCA[11,12], 1992; PRATI; PASHLEY[16], 1991; GWINNETT[8], 1992; SWIFT; TRIOLO[17], 1992; CHARLTON; BEATTY[4], 1994).

It has been found that dentin-bonding agents based on acetone are particularly effective in bonding to wet dentin. The explanation has been reported to be associated with

the water-displacing effect of the acetone (KANCA[12], 1992; GWINNETT[8], 1992).

It was the purpose of the present work to investigate the denting-bonding agent Gluma when applied to dry, moist, or wet dentin surfaces. In an attempt to increase the effectiveness of the system, the traditional pretreatment by EDTA was replaced by phosphoric acid, and the Gluma primer was modified by means of water and acetone.

MATERIAL AND METHODS

The materials used in the investigation are presented in Tables 1 and 2. The Gluma system involves the consecutive use of 1) a conditioner, 2) a primer, and 3) a bonding resin before application of the resin composite. The conditioners used in the study are presented in Table 3 (together with the results), and were based on either EDTA or on H_3PO_4. The EDTA-solution was the Gluma 2 Cleanser, and the H_3PO_4 was applied either as a liquid (l) or as a gel (g). The composition of the primers are presented in Table 2, and were produced by mixing HEMA, glutaraldehyde, water, and, in some groups, acetone. The primer d-1 has the same composition as the Gluma 3 Primer.

The bond strength to dentin was measured by use of extracted human teeth having been stored at room temperature in a solution of 1% chloramine T. The teeth were embedded in epoxy resin, and a flat dentin surface was produced by wet grinding on carborundum paper # 1000 at the final stage. The dentin surfaces were washed, dried by a stream of air and then pretreated with one of the conditioners shown in Table 3. After a washing of the dentin surfaces for 15 seconds with water, the dentin was left in either a dry, a moist, or a wet state, to be specified below. Then the surfaces were primed with either Gluma 3 Primer or one of the primers shown in Table 2. After 30 seconds, excess primer was removed by drying with compressed air. A split teflon mold with a cylindrical hole (diameter = 3.6 mm; height = 5 mm) was then clamped to the prepared dentin surface. Next, a drop of Gluma 4 Sealer was placed inside the mold and excess was removed with a blast of air, leaving a film of resin on the dentin surface. The mold was then filled with Pekafill to a height of 2-3 mm, and curing was carried out for 60 seconds by means of the Visilux 2 lamp (3M,USA). Further details in the preparation of the specimens have been given before (Munksgaard et al.[14], 1985).

After polymerization of the resin composite, the mold was removed, and the specimens stored in water at 37°C for 24 hours. The bonds were then broken in shear in an Instron Universal Testing Machine (Instron, England) at a crosshead speed of 0.5 mm/min.

Three different series of experiments were carried out, each comprising several groups. The number of

TABLE 1 - List of materials used in the investigation

MATERIAL	BATCH NO	MANUFACTURER
Pekafill Composite Resin	1800-K	Bayer, Germany
Gluma 2 Cleanser (EDTA)	5610-E	Bayer, Germany
Gluma 3 Primer	025120	Bayer, Germany
Gluma 4 Sealer	015022	Bayer, Germany
37% H_3PO_4 liquid (l)	P920626	3M, USA
20% H_3PO_4 gel (g)	Experimental	Bayer, Germany

TABLE 2 - Experimental primers. Composition in weight%

PRIMER	HEMA	GLUTERALDEHYDE	WATER	ACETONE
a-1	64	9	27	0
a-2	32	4.5	13.5	50
b-1	50	7	43	0
b-2	25	3.5	21.5	50
c-1	42	6	52	0
c-2	21	3	26	50
d-1*	35	5	60	0
d-2	17.5	2.5	30	50

* Primer d-1 corresponds to the Gluma 3 Primer

specimens in each group was eight. A main difference between the three series was the degree of dryness or wetness of the dentin surfaces.

Series A - The conditioners are specified in Table 3. After rinsing of the dentin surface, the dentin was dried for 10 seconds with a blast of air. This state of the dentin will be referred to as a "dry" dentin surface. Gluma 3 Primer was used as primer.

Series B - The pretreatment in this series was always 20% H_3PO_4 gel, applied for 15 seconds. After rinsing of the dentin surface, water was removed from the surface by gently pressing two times with paper tissue (Medical Wipes, Clark Corporation, USA). This state of the dentin will be referred to as a "moist" dentin surface. The composition of the primers a-1 to d-2 are specified in Table 2.

Series C - The pretreatments in this series were either 20% H_3PO_4 gel or the EDTA solution, applied for 15 or 30 seconds, respectively. After rinsing of the dentin surface, water was gently removed from the surface by means of a damp cotton pellet. This state of the dentin will be referred to as a "wet" dentin surface. The composition of the primers d-1 and d-2 are specified in Table 2.

RESULTS

The results of the bonding experiments are shown in Tables 3, 4, and 5, together with the the variables defining the groups in each series. The data were treated statistically by analysis of variance.

Table 3 gives the results of series A, in which primer was applied to dry dentin. Analysis of variance reveals a difference between the five mean values, and that the bond strength obtained in experiment no. 2 is significantly lower than the other values ($p < 0.005$). The remaining values of the series are not different from each other ($p > 0.05$)

TABLE 3 - Series A of experiments. The primer was Gluma 3 Primer, applied for 30 sec

Experiment Nº.	Conditioner	Time of application(sec)	State of dentin	Bond Strength (MPa ± SD)
1	EDTA	30	dry	17 ± 3.8
2	37% H_3PO_4 (l)	60	dry	12 ± 2.8
3	20% H_3PO_4 (l)*	15	dry	16 ± 2.9
4	20% H_3PO_4 (g)	30	dry	18 ± 1.9
5	20% H_3PO_4 (g)	15	dry	21 ± 5.5

* Prepared by dilution of 37% H_3PO_4 (l) with water

TABLE 4 - Series B of experiments. The conditioner was 20% H_3PO_4 gel, applied for 15 seconds

EXPERIMENT Nº.	PRIMER	STATE OF DENTIN	BOND STRENGTH (MPA+SD)
6	a-1	moist	17 ± 1.5
7	a-2	moist	18 ± 1.3
8	b-1	moist	22 ± 3.6
9	b-2	moist	18 ± 1.8
10	c-1	moist	20 ± 6.8
11	c-2	moist	16 ± 2.9
12	d-1	moist	17 ± 5.0
13	d-2	moist	15 ± 2.4

Table 4 gives the results of series B, in which the primers were applied to moist dentin. Analysis of variance showed no difference between the eight mean values ($p>0.025$).

Table 5 gives the results of series C, in which the primers were applied to wet dentin. Analysis of variance showed no difference

between the three groups.

A comparison of series A excluding experiment no. 2 with Series B showed no difference in bond strength between the two series ($p > 0.05$).

A comparison of series B with series C showed a statistically significant difference between the two series ($p < 0.001$).

DISCUSSION

The all-etch technique for restoration of teeth with resin composite is gaining widespread use. The technique involves a conditioning of the tooth by which both enamel and dentin are etched by phosphoric acid. At the time when the Gluma system was developed, etching of dentin was considered hazardous to the pulp, and instead of phosporic acid, an EDTA solution was used as conditioner of the dentin (Munksgaard and Asmussen[13], 1984). However, recent work has demonstrated that use of phosphoric acid in combination with effective dentin-bonding agents is not harmful to the pulp (Cox and Suzuki[5], 1994). On the contrary, the all-etch technique is beneficial by saving a step in the bonding procedure, thus reducing the risk of inadvertent errors by the dentist. The results of series A show that 60 seconds etching of the dentin with strong acid (37% H_3PO_4) gives significantly lower bond strength with the Gluma system. This is in agreement with earlier work, showing reduced bond strength comparing 37% H_3PO_4 with EDTA (Asmussen and Bowen[2], 1987). On the other hand, the present results show acceptable strength after etching for shorter times with lower concentrations of the acid.

Since a thorough drying of dentin is neither desirable nor possible in a clinical situation, it is of prime importance that bonding systems are effective on dentin in various states of wetness. The results of Series B show that a good bonding can be obtained with the Gluma system applied to moist dentin. In previous studies it has been pointed out that acetone may be an effective agent in securing bonding to wet dentin (Kanca[12], 1992). The acetone is reported to displace the water in the demineralized upper layer of dentin after the etching, and thus to facilitate the penetration of the primer into the dentin surface to form the hybrid layer. However, in the present study no extra effect of the acetone could be demonstrated by comparing a-1 with a-2, b-1 with b-2, etc. It seems that the mixtures with only water, HEMA and glutaraldehyde are sufficiently hydrophilic to be able to produce the hybrid zone.

In an earlier study, in which a simplified Gluma system was investigated, the balance between the amounts of HEMA, Glutaraldehyde, water, and solvent was found to exert a significant influence on bond strength (Araujo and Asmussen[1], 1989). In that work a so-called Gluma Resin was developed, combining the primer and the ensuing resin into a single step. In the present work the results of Table 4 show no variation in bond strength with the composition of the primer. One explanation may be related to the fact that the ratio between HEMA and glutaraldehyde is close to seven in all experimental primers of Table 2. Further, the experimental situation is different in that in the present work the primer is modified, whereas in our earlier work it was the Gluma Resin that was subjected to experimental changes.

In the series C of experiments, the condition of the dentin was described as "wet". It has been a matter of controversy what is meant by "wet" dentin (Gwinnett[9], 1994), and how to quantify wetness. In this series the dentin was dabbed with a damp cotton pellet, and it was our subjective impression that the dentin in this series was more "wet" than in series B, where the dentin was best described as "moist". The increased degree of wetness is probably the reason why the values are lower in series C as compared to series A (excepting experiment no. 2) and B. As in series B, it made no difference to bond strength whether the primer contained acetone or not (d-1 versus d-2). As stated above, this may be do to the hydrophilic nature of the primer even without

TABLE 5 - Series C of experiments

Experiment Nº.	Pretreatment	Time of application (sec)	Primer	State of dentin	Bond Strenght (MPa ± SD)
14	20% H_3PO_4 (g)	15	d-1	wet	15 ± 5.0
15	20% H_3PO_4 (g)	15	d-2	wet	12 ± 5.6
16	EDTA	30	d-1	wet	14 ± 7.1

acetone. As in series A, the acidic conditioners gave as good bonding as did the EDTA-solution.

Although the bond is lower on wet dentin than on dry or moist dentin, bond strengths in the range of 12 to 15 MPa may still be argued to be bonds of a size useful in a clinical situatuion. This results contrasts with that of another study, reporting significantly lower bond strengths when applying the Gluma bonding system to wet dentin surfaces (Gwinnett[8], 1992). The reason for this discrepancy is not quite clear. One possibility is the degree of wetness, which may have differed between the studies. As stated above, defining the wetness of the dentin surface is not a straightforward job.

CONCLUSIONS

1. Good bonding may be obtained with the Gluma system after an appropriate acidic conditioning of the dentin.

2. Good bonding may be obtained with the Gluma system applied to moist surfaces, but a moderate reduction in bond strength may be noted with wet dentin.

RESUMO

Investigou-se a adesão de uma resina composta à dentina, promovida pelo sistema Gluma de adesão, quando aplicado a superfícies de dentina secas ou úmidas e ainda introduzindo-se algumas alterações na fórmula original do "primer" do sistema, assim como utilizando-se vários condicionadores ácidos no pré-tratamento da dentina.

Valores de resistência ao cisalhamento entre 12 e 22 MPa foram registrados. Foi verificado que um discreto umedecimento da dentina melhora o desempenho do adesivo Gluma mas que um excesso de umidade, pode diminuir os valores de resistência ao cisalhamento em até 25%.

UNITERMOS: Resistência adesiva; Adesivos dentinários; Dentística restauradora.

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