

Tensile bond strength of hydroxyethyl methacrylate (HEMA) bonding agent to bovine dentine surface at various humidity

Adioro Soetojo

Department of Conservative Dentistry
Faculty of Dentistry Airlangga University
Surabaya - Indonesia

ABSTRACT

One factor that make bonding agent adhere to dentine surface maximally is the humidity condition around the dentine surface. The best bond strength between bonding agent with dentine surface is depending on the moist surface. It mean that the dentine surface should neither too dry or wet. The objective of this research is to know the tensile bond strength of hydroxyethyl methacrylate (HEMA) bonding agent to bovine dentine surface at various humidity. The bovine dentine was grounded to give flat surface, which was then etched with 37% phosphoric acid for 15 seconds. Dentine was washed with 20 ml water and dried with blot dry technique. The dentine, except the control group, was placed in a desiccator for one hour at difference humidity. Dentin was removed from desiccator, then covered with bonding agent and put into tensile tool plunger. Self-cured acrylic resin was applied on this bonding agent layer, which was placed on opposite-plunger. After 24 hours, tensile bond strength was measured with Autograph instrument. Data was statistically analyzed with One-Way ANOVA at 95% confidence level, continued with LSD test. Results of this study showed that 60%–90% humidity gave produce the lower of tensile bond strength of bonding agent to dentine surface ($p \leq 0.05$). In conclusion, the treatment in 60% humidity gave the greatest tensile bond strength.

Key words: humidity, dentine bonding agent, bovine dentine, tensile bond strength

Correspondence: Adioro Soetojo, c/o: Bagian Ilmu Konservasi Gigi, Fakultas Kedokteran Gigi Universitas Airlangga. Jln. Mayjend. Prof. Dr. Moestopo No. 47 Surabaya 60132, Indonesia.

INTRODUCTION

In operative dentistry field, drying is one step of the application of dentine bonding based on hydroxyethyl methacrylate (HEMA) to dentine surface. This step is done based on total etched technique. The purpose of the procedure is to blot up excess water that were used for washing dentine surface after etching process.¹⁻³ The purpose of washing is to removed the salt that formed as reaction of acid etched materials and dentine mineral. Some researchers said that the dentine surface should neither too dry nor wet.⁴ If the surface is too dry, the dentine collagen will collapse so that the bonding between resin and dentine collagen will be difficult. On the contrary, if the dentine surface is too wet, there are too many water molecules around collagen, so hydrogen bond between water and amino collagen will inhibit bonding agent to bond with collagen. Therefore an optimal condition of dentin surface is needed to obtain a maximal bonding between dentine bonding agent and dentine collagen.

A good moisture characteristic of dentin bonding materials is referring to the low level of viscosity so that can improve surface energy. HEMA is a humectants hydrophilic agent that has ability to wet dentin surface.⁵

Generally, the bond efficacy of resin bonding materials to dentin collagen depend on some factors that are: the lowness of monomer viscosity, concentration and type of monomer, acid application as conditioner, humidity and temperature around fibril kolagen.⁴⁻⁷ Research concerning the influence of humidity to tensile bond strength of dentin bonding materials at dentin surface was done by in vitro experiment. In this research, they used 33%, 50%, 75%, and 100% humidity. Humidity of oral cavity depends on the usage of rubber dam. If we use rubber dam, the humidity is 50% at 23 °C that influenced by the humidity of the dental practice room. But, if we do not use rubber dam, the humidity is about 80–94%.⁸

In this research, we used humidity that ranging from 60–90% with temperature 25 °C. This 60% humidity is minimum humidity according to the research previously. At 50 % and 65 humidity, the tensile bond strength of bonding materials at dentine is higher than 80% and 90% humidity.⁹ The purpose of this research is to know the tensile bond strength between HEMA dentine bonding agent to bovine dentine surface at various humidity.

This research will be explain the mechanism of tensile bond strength of HEMA dentine bonding agent to bovine dentine surface at various humidity, so clinically will be

achieve maximum tensile bond strength of HEMA dentine bonding agent to dentine surface.

MATERIALS AND METHODS

Materials that were used: bovines incisive, acid etch (Ivoclar Vivadent, Schaan/Liechtenstein), HEMA dentine bonding (Voco, Germany).

Appliances that were used: diamond disk, diamond drill, emery number 400 and 1000 (Fuji Star, Japan), desiccator with vacuum faucet, hygrometer and air thermometer (Haar. Synth. Hygro, Germany); compressor/air suction tool (Schuco, USA), Autograph AG-10 TE, (Shimadzu, Japan).

Tooth sample was cleaned carefully by removed dirt at tooth surface using brush, while for hard or soft tissue using sharp scalpel. During cleaning, tooth was always in wet condition. Tooth was cut with diamond disk and planted in hard gypsum cylinder log. The dentin part was facing upward. Preparation was done until dentin surface using fissure diamonds bur. the surface of dentin was attenuated with silicon emery number 400 and continued with number 1000. After that, dentin was covered with adhesive tape which 3 mm diameter and attached precisely in the middle of the dentin surface.¹⁰ The preparation above as according to procedure that was done previously.¹¹ Next, dentin specimen was coated with 37% phosphoric acid etch with cotton pellets for 15 second, then washed with 20 ml aquades using injection sput and dried smoothly using cotton pellets. This drying techniques is call blot dry technique.¹²

Samples were placed into desiccator for 1 hour at 60% humidity for group I; 70% for group II; 80% for group III; 90 % for group IV, and 65% for group of V (control group) with 8 samples each.¹⁰ Immediately, after released from desiccator, primary condensation and bonding were mixed and then applied to dentine surface using disposable brush for 30 second, after that were exposed with light curing unit for 20 second (as according to manufacturer's guide). Next, the cylinder log was inserted into plunger. The opponent plunger was filled with self cured acrylic as filling materials over dentine bonding and then united with the other plunger and fixed by installing lock dowel. Samples were kept at room temperature ($\pm 28^\circ\text{C}$) for 24 hours. After

24 hours, the tensile bond strength of the samples was measured with Autograph (cross head speed = 10 mm/minute, range: 5, capacities of load cell: 5 kN/500 kgf). The result which shown at screen have set of kgf (1 kgf = 9.81 N. 1MPa = N/mm²). Wide of dentine surface sample = $\text{Ar}^2 = 7.1 \text{ mm}^2$. Data was statistically analyzed with One-Way ANOVA at 95% confidence level and was continued with LSD test.

RESULTS

The tensile bond strength, mean, and standard deviation of HEMA dentine bonding agent to bovine dentine surface can be seen in table 1. The differences of all samples in this experiment were analyzed with One-Way ANOVA at 95% confidence level. The result showed that tensile bond strength of HEMA dentine bonding agent to bovine dentine

Table 1. Mean and standard deviation of tensile bond strength between dentine bonding agent based on HEMA with bovine dentine surface (MPa)

Humidity	n	\bar{X}	SD
60%	8	16.74	1.8
70%	8	12.74	2.5
80%	8	11.59	2.4
90%	8	10.02	2.1
Control 65%	8	15.50	1.8

Description:

n = Sample amount, \bar{X} = Mean of tensile bond strength, SD = Standard Deviation

surface at 60%–90% humidity level significantly different ($p < 0.05$). LSD test was used to determine the difference of each sample (Table 2).

The data distribution of tensile bond strength was normal that analyzed with Kolmogorov-Smirnov test. Sample group at 60–90% humidity have p value more than 0.05, it mean the variable data has normal distribution. Lavene test was used to prove that the tensile bond strength sample was homogenous. From the calculation, it obtained that sample group of tensile bond strength at 60–90% humidity was homogeneous ($p > 0.05$).

Table 2. LSD of the tensile bond strength between dentine bonding agent based on HEMA with bovine dentine surface

humidity	60%	70%	80%	90%	Control (65%)
60%	XXX	+	+	+	--
70%	+	XXX	--	+	+
80%	+	--	XXX	--	+
90%	+	+	--	XXX	+
Control (65%)	--	+	+	+	XXX

Description:

+: significance difference, —: not significance

The effect of 60–90% humidity to the tensile bond strength between resin bonding materials and dentin surface showed the significance in each sample group ($p < 0.05$). To know the significance of each sample group the data was analyzed using LSD test (Table 2). The result showed that sample group at 60% humidity have p value smaller than 0.05 compared to sample group at 70%, 80%, and 90% humidity. It mean the tensile bond strength between resin bonding and dentin at 60%, 70%, 80%, and 90% humidity was significantly different. The tensile bond strength at 60% humidity compared to sample group at 65% humidity was not significantly different ($p > 0.05$).

Tensile bond strength of resin bonding to dentin was significantly different between at 70% and at 60%, 90% and 65% (control group) humidity ($p < 0.05$), but at 70% humidity and 80% have no significance ($p > 0.05$). If we compared, sample group at 80% humidity and 90% humidity had p value more than 0.05. This matter means there was no significance of the tensile bond strength between sample group at 80% and 90% humidity. The tensile bond strength of sample group between at 80% humidity and to control sample group show the significance. If compared to control group, tensile bond strength of sample group at 90% humidity was significantly different ($p < 0.05$).

DISCUSSION

Tensile bond strength between resin bonding and dentine surface is caused by chemical interaction and mechanical retention. Chemical bond happened because of the interaction between carbonyl ester of HEMA with amino of collagen dentine. In the other hand, mechanical bonding obtained by penetration of HEMA into inter-fibril cavity (nano-space) which later polymerized. Microscopically, there are no collagens at whole dentine surface, while mechanical bonding entangle all areas of dentin surface. Therefore, mechanical bond always more dominant than chemical bond.

To prove that chemical bond also influence in the process of mechanical bond between dentine bonding resin and dentine collagen, a research that caused collagen collapse was done.¹⁰ In this experiment the mean value of tensile bond strength between dentine bonding resin and dentine collagen decrease become 11.88 ± 2.91 MPa (equal to $\pm 29\%$ degradation).

The higher tensile bond strength happened because the efficacy of monomer infiltration is totally at demineralized dentine layer. If monomer infiltration just at the top, it will leave matrix of demineralized dentin unprotected. This matter caused collagen hydrolyzed so that the tensile strength is low.⁴

In this research, the influence of humidity to tensile bond strength of resin bonding agent to dentine can be seen in table 1. The highest value of tensile bond strength is obtained at 60% humidity, then decreased significantly at

70%, 80% and 90% humidity. High humidity will decrease tensile bond strength. This result is same as Besnault and Attal research's.⁹ So 60% humidity is the best humidity where the amount of water molecules is ideal enough to re-expansion collagen fibril so that resin will easy to penetrating into cavity between fibril. Chemically, interaction between resin and collagen is strong. Dentine bonding agent based on HEMA contains acetone as a solvent. Acetone is a solution that evaporate easily, can slight resin liquid so decrease the viscosity. When this liquid applied at dentine surface, it will penetrate into micro cavity between collagen fibril, and then chasing water molecule and then evaporated. Thereby, it will leave dentine bonding resin bind with collagen fibril.¹³

Acetone concentration influence the thickness of resin bonding layer and it's tensile bond strength, but the thickness of bonding resin have no relation with it's tensile bond strength. Resin material leakage is caused by acetone evaporation, bad polymerization, and the low strain strength of dentine bonding resin (because the acetones amount is too much). Another characteristic of acetone are: water chasing effect, able to increase vapor pressure of water, especially water around collagen. The optimum concentration of acetone is 37% weight. In this concentration the thick of resin bonding layer is 30.2μ and the tensile bond strength is 63.5 MPa.

In this research, the tensile bond strength at 60% humidity was higher than at 70% humidity, hence at 70% humidity the amount of water molecules is higher than at 60%, so the acetone ability to chase water also low at that humidity. At 60% humidity the acetone ability to chase water is higher, and in this condition bonding resin will immediately penetrate to collagen.

In conclusion, maximum tensile bond strength value between HEMA dentine bonding agent and bovine dentine surface obtained at 60% humidity. As suggestion, further research is needed to examine the tensile bond strength of HEMA dentine bonding agent with dentine collagen with various pH of HEMA, HEMA concentration, acetone concentration, and temperature in connection with humidity of dentine surface.

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