

## Research Report

## Tensile bond strength of hydroxyethyl methacrylate dentin bonding agent on dentin surface at various drying techniques

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### ABSTRACT

**Background:** There are several dentin surface drying techniques to provide a perfect resin penetration on dentin. There are two techniques which will be compared in this study. The first technique was by rubbing dentin surface gently using cotton pellet twice, this technique is called blot dry technique. The second technique is by air blowing dentin surface for one second and continued by rubbing dentin surface gently using moist cotton. **Purpose:** This experiment was aimed to examine the best dentin surface drying techniques after 37% phosphoric acid etching to obtain the optimum tensile bond strength between hydroxyethyl methacrylate (HEMA) and dentin surface. **Method:** Bovine teeth was prepared flat to obtain the dentin surface and then was etched using 37% phosphoric acid for 15 seconds. After etching the dentin was cleaned using 20 cc plain water and dried with blot dry techniques (group I), or dried with air blow for one second (group II), or dried with air blow for one second, and continued with rubbing gently using moist cotton pellet (group III), and without any drying as control group (group IV). After these drying, the dentin surfaces were applied with resin dentin bonding agent and put into plunger facing the composite mould. The antagonist plunger was filled with composite resin. After 24 hours, therefore bond strength was measured using Autograph. **Result:** Data obtained was analyzed using One-Way ANOVA with 95% confidence level and continued with LSD test on  $p \leq 0.05$ . The result showed that the highest tensile bond strength was on group I, while the lowest on group IV. Group II and IV, III and IV, II and III did not show significant difference ( $p > 0.05$ ). **Conclusion:** Dentin surface drying techniques through gentle rubbing using cotton pellet twice (blot dry technique) gave the greatest tensile bond strength.

**Key words:** Tensile bond strength, hydroxyethyl methacrylate dentin bonding agent, dentin surface drying technique

### ABSTRAK

**Latar belakang masalah:** Teknik pengeringan permukaan dentin agar resin dapat penetrasi dengan sempurna adalah dengan cara pengusapan secara halus sebanyak dua kali menggunakan bulatan kapas, yang disebut blot dry technique, dengan semprotan udara selama 1 detik atau dengan semprotan udara selama 1 detik yang dilanjutkan dengan pengusapan secara halus menggunakan bulatan kapas basah yang diperas dan kelebihan air pada kapas diserap dengan kertas hisap. **Tujuan:** Penelitian ini bertujuan untuk mengetahui teknik pengeringan permukaan dentin yang terbaik setelah etsa dengan asam fosfat 37% untuk mendapatkan kekuatan perlekatan tarik yang optimum antara HEMA dentine bonding agent dan permukaan dentin dengan menggunakan alat ukur Autograph. **Metode:** Permukaan dentin gigi sapi diasah rata, kemudian di etsa dengan asam fosfat 37% selama 15 detik. Permukaan dentin dicuci dengan 20 cc air dan kemudian dikeringkan dengan cara blot dry technique (kel. I), dengan semprotan udara selama 1 detik (kel. II) atau semprotan udara selama 1 detik dan dilanjutkan dengan pengusapan secara halus menggunakan bulatan kapas basah yang diperas dan kelebihan air pada kapas diserap dengan kertas hisap (kel. III) dan tanpa pengeringan permukaan dentin sebagai kontrol (kel. IV). Selanjutnya permukaan dentin diulasi dengan resin bonding dan diletakkan kedalam plunger dengan permukaan menghadap permukaan komposit. Setelah 24 jam dilakukan pengukuran kekuatan tarik dengan menggunakan alat ukur Autograph. **Hasil:** Analisis data menggunakan uji Anova satu arah dengan derajat kepercayaan 95% dan dilanjutkan dengan test LSD pada  $p \leq 0,05$ . Dari hasil penelitian didapatkan kekuatan perlekatan tarik yang paling tinggi pada kelompok I, sedangkan yang paling rendah pada kelompok IV. Didapatkan perbedaan bermakna pada kelompok I, II, III, dan IV. Pada kelompok II dan IV, III, dan IV, II, dan III tidak

didapatkan perbedaan bermakna ( $p > 0,05$ ). **Kesimpulan:** Teknik pengeringan permukaan dentin dengan cara pengusapan secara halus menggunakan bulatan kapas sebanyak 2 kali menghasilkan kekuatan perlekatan tarik terbesar.

**Kata kunci:** Kekuatan perlekatan tarik, HEMA dentine bonding agent, teknik pengeringan permukaan dentin.

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## INTRODUCTION

One of the phase of teeth restoration using composite resin is the application of dentin bonding agent on the surface of dentin that functions to join composite restoration with tooth tissues so that the dentin bonding strength increase. The application of dentin bonding using total etching system is by acid etching on enamel and dentin surfaces previously followed by washing and drying and then application of resin dentin bonding agent. The purpose of acid etching is to open the dentin collagen fibril tissues so that resin agent could bind physically and chemically with fibril collagen. Other researchers said that the purpose of washing is to remove salts which are formed by the reaction between acid etching agent and enamel and dentin minerals. After the washing, drying up is done to remove which is meant to remove the left over of washing water after acid etching.<sup>1</sup> Other researchers stated that the procedure of dentin surface drying is different from the one of the enamel surface, for that reason the surface of dentin must not too dry or too wet. If the surface is too dry, the hydrogen bond will be cut off so that the dentin collagen may collapse, shrink, and the collagen amino cluster will be covered among the structure of secondary collagen. This condition will results in difficulty for the resin agent to penetrate into the remaining cavity and it will cause the absence of mechanical retention between resin bonding agent and dentin collagen. If the surface of the dentin is too wet, there will be a lot of water molecules surrounding the dentin collagen so that a bond between hydrogen and water with carbonyl resin bonding cluster may be developed resulting in the ability to bond with dentin collagen amino cluster.<sup>2</sup>

The success of the fusing of dentin bonding agent to the dentin surface depends on monomer viscosity, types and concentration of monomer, acid etching application as conditioner, temperature and humidity around the fibril collagen.<sup>2,3,4</sup> The studies found out that in 33%, 50%, 75% and 100% humidity, the tensile strength between dentin bonding agent and dentin teeth gets increased following the decrease of humidity.<sup>5</sup> In vitro study on dentin humidity of 30%, 50%, 65%, 80% and 95% showed that tensile strength of bonding agent on dentin will increase following the decrease of the humidity around dentin.<sup>6</sup> Whereas, the study in humidity of 50%, 80%, and 95% proves that tensile strength dentin bonding will decrease with regard to the increase of humidity.<sup>7</sup> Other research study, proved that 70% humidity is the best one, because it develops the maximum bond between the carbonyl HEMA cluster and dentin collagen whereas 60% humidity gives the maximum

tensile strength.<sup>8</sup> The best condition of the dentin surface is moist in order to obtain the maximum bond between resin bonding agent and dentin collagen. Dentin moist means the condition related with water content contained in the dentin surface. The humidity developed around the dentin collagen will influence the physical and chemical nature so that it will affect the collagen bond towards resin agent. Water content in dentin can be measured from the ratio between water weight in dentin with the total of dentin weight and water content in it (%).

To reach maximal bonding between dentin bonding agent on dentin surface, it is important that collagen fibril should be in permeable condition/active.<sup>1,2</sup> Permeable collagen fibril is strongly influenced by the moist surrounding dentin surface. The dentin surface drying techniques to enable a perfect resin penetration, some studies reported that dentin surface drying by rubbing gently using a cotton pellet for two times (blot dry technique), or with air blow for 1 second or with air blow for 1 second, and then followed by gently rubbing it with a cotton pellet which has been wetted by water, the excess of water was removed by squeezing and put on blotting paper.

The purpose of this study was to examine the best dentin surface drying techniques after acid etching with 37% phosphoric acid in 15 second to obtain optimum tensile strength of hydroxyethyl methacrylate (HEMA) dentin bonding agent to bovine dentin surface.

## MATERIAL AND METHOD

Bovine's incisors were taken from slaughter house of Pegirian, Surabaya. In this experiment, a newly pulled out incisor from the bovine tooth which was vital, caries free, unscratched, with no chipped part, no abrasion that had been checked under a light microscope is used. The tooth was then cleaned, soaked in physiologic solution and kept in a refrigerator in 4° C. After that the tooth was cut under flowing water using a diamond disk and then with a fissure diamond drill so that the dentin was open. The dentin surface was smoothed by number 400 and number 1000 silicon scrub papers, cleaned under 20 cc water sprayer, the dentin surface part was covered with an adhesive tape which has been given 3 mm hole and was stocked right to the middle of the dentin surface, and then it was planted into a hard gypsum cylinder block which was then etched with 37% phosphoric acid using a cotton pellet in 15 seconds and at last it was washed with 20 cc aquadest from an injection syringe.

The experiment samples were made more than 40 samples and then 40 samples which comply with the criteria were randomly taken, 10 samples for each experiment group. Group I: the dentin surface was dried with blot dry technique,<sup>9</sup> group II: dentin surface was dried by air blow for 1 second, group III: the dentin surface was dried by air blow for 1 second, and then followed by gently rubbing it with a cotton pellet which had been wetted by water, then the excess water removed by squeezing and put on blotting paper, and group IV: the control group, with no dentin surface drying.

After the dentin surface drying process had been done, the dentin surface was covered with total etched dentin bonding agents, prime and bond. It was left that way for 30 seconds, sprayed using air blower, then was lighted up with light cured for 20 seconds (as instructed by the factory). After having been given treatment the cylinder block was put into a plunger, with its surface was made facing to the composite mold.

The opponent plunger was filled with resin composite on the dentin bonding and top of it was given a celluloid strip and a thin glass, next a weight of 1000 gram was put on it for one minute, and then the left over was removed using a scalpel, and after that it was light cured for 40 seconds with a light curing unit (factory direction is followed). After the composite application had been finished, the specimen was taken out from the composite placing equipment aid and the molding ring was released. Then the sample that had been ready was placed into a plastic tube containing aquadest for 24 hours. After 24 hours the sample tensile strength was tested using an Autograph measuring equipment (Shimadzu, Japan) with the cross head speed of 10 mm/minute, range: 5, load cell capacity 5 kN/500 kgf. The result was a tensile strength in kgf where 1 kgf = 9.81 N. 1 MPa = N/mm<sup>2</sup> width of experimental dentin surface =  $\pi r^2 = 7.1 \text{ mm}^2$ . The data was analyzed with One-Way ANOVA at 95% confidence level continued with LSD test on  $p \leq 0.05$ .

## RESULT

The tensile bond strength, mean, and standard deviation of HEMA dentin bonding agent on bovine dentin surface at various dentin surface drying techniques can be seen in Table 1.

In order to find out if the distribution of the tensile strength data was normal and homogenous, the Kolmogorov-Smirnov test was administered, resulting  $p > 0.05$  which means that the data has a normal distribution. To examine if the data was homogenous, the Levene test is administered.

The difference 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 dentin bonding agent to bovine dentin collagen with various dentin surface drying techniques significantly different ( $p < 0.05$ ). To determine the difference of each samples continued with LSD test on  $p \leq 0.05$  (Table 2). The result showed that

experiment group I was compared with experiment group II, III and IV, it will have  $p < 0.05$ , meaning that there was a significant difference. The tensile bond strength of resin bonding to dentin at group II and IV, III and IV, II and III was not significantly different ( $p > 0.05$ ).<sup>10</sup>

**Table 1.** Mean and standard deviation of tensile bond strength between HEMA dentin bonding agent and bovine dentin surface at various dentin surface drying techniques (Mpa)

Drying technique	n	$\bar{X}$	SD
Group I	10	17.9021	2.6853
Group II	10	9.5432	1.4314
Group III	10	11.6121	1.7418
Group IV (control)	10	7.3214	1.0982

Description: X : Mean of tensile bond strength, n : The number of samples, SD : Standard Deviation

- Group I : The dentin surface was dried with blot dry technique  
 Group II : The dentin surface was dried with air blow for one second.  
 Group III : The dentin surface was dried with air blow for one second, followed by rubbing it softly using a cotton pellet which has been wetted with water, the excess water was removed by squeezed and put on blot paper.  
 Group IV : No drying was done on dentin surface (control group).

**Table 2.** LSD of the tensile bond strength between HEMA dentin bonding agent on dentin surface at various dentin surface drying techniques

Drying technique	Group I	Group II	Group III	Group IV (control)
Group I	xxx	+	+	+
Group II	+	xxx	-	-
Group III	+	-	xxx	-
Group IV	+	-	-	xxx

Description: +: Significant difference, -: not significant

## DISCUSSION

The tensile bond strength between HEMA dentin bonding agent on dentin surface is derived because there are chemical bonding and mechanical retention. Mechanically, because there is a penetration of HEMA into inter-fibril nano space, dentin tubules, dentin peritubules, dentin intertubules which later polymerized. In addition, physical bonding occurs because of Van Der Waals tensile bond between both agents. Whereas this chemically happens because of the interactions between carbonyl ester of HEMA from the

bonding agent with amino of collagen dentin which produce amide groups [C(O)NH]. This bonding is strong due to its covalent characteristics called as inter-atomic primer bond. Microscopically, collagen is not found in the entire dentin surface, where as mechanical affixing involves the whole dentin surface so that the mechanical bonding is more dominant than the chemical bonding.<sup>11</sup>

Dentin is living tissue which contain approximately 60% inorganic components (hydroxyapatite), 30% organic component and 10% water. Those organic component are 90% collagen and 10% non collagen. Most of those collagens are type I and few of them are type V. The tensile strength in this experiment is the adhering strength between resin bonding agent (HEMA) and bovine dentin surface which was flat. In this research the dentin bovine was used because it contains type I collagen, type V collagen, non collagueus protein and proteoglicane and others. The tensile strength of resin bonding on the human dentin surface is a little bit different from the surface of bovine teeth. Other researcher stated that resin bonding strength on human dentin is higher than on bovine teeth, this is due to the amount of bovine dentin apatite mineral is less than the one of human teeth.<sup>2</sup>

In this research, the influence of various dentin surface drying techniques to tensile bond strength of resin bonding agent to dentin can be seen in table 1. Group I showed the highest tensile bond strength, which is 17.9 Mpa. The result of this study goes along well with other researchers which states that the highest value of tensile strength is earned in 60% moisture. Because of dentin surface drying technique was the same with what was done by other research, the acquired dentin surface moisture which was probably  $\pm 60\%$ .<sup>8</sup> The result of this research was also appropriate with the research of other researchers, which stated that 60% moisture is the best moisture since the number of water molecules is ideal enough to re-expand fibril collagen so that the resin will penetrate easily into nano space among fibril and chemically there will be strong interactions between resin and collagen. Resin agent contains acetone which easily evaporates so that the resin dentin bonding that reacts with fibril collagen would form hybrid dentin layer which was believed to be the main strength of resin attachment with dentin.<sup>12,13</sup>

The higher tensile bond strength happend because the efficacy of monomer infiltration is totally at demineralized dentin layer. On dentin surface drying techniques that was applied on group II, group III, and without drying process on the dentin surface on group IV, it was found that this drying process might probably result in bigger number of water molecules than that of blot dry technique as in group I. As the number of water molecules was higher than those in blot dry technique, the capability of acetone to drive away water and then evaporate around the dentin collagen was also low, so that the resin bonding conjugation with fibril collagen will decrease too.<sup>3,12,14</sup> At blot dry technique, the acetone ability to chase water was higher, and in this

condition bonding resin will immediately penetrate to collagen. Acetone concentration influences the thickness of resin bonding layer and tensile bond strength.<sup>12</sup>

Higher moisture will reduce the decrease of HEMA concentration in nano space among fibrils, it means there was a decrease in the mechanism retention between HEMA resin and dentin collagen and there was a decrease in the chemical bond between HEMA and collagen, as when there are a lot of water molecules around dentin collagen, the hydrogen bond between water and collagen amino may happen and this will prevent the bonding agent from conjugate with collagen.<sup>2</sup> While the drying using air blow may cause trauma on the soft fibril collagen so that the fibril collagen will collapse. Due to the collapse of fibril collagen, so it was difficult for resin bonding to bind with dentin collagen, resulting in the decrease of its tensile strength.<sup>11</sup>

It can be concluded the drying technique of the dentin surface by rubbing it gently using a dry cotton pellet twice (blot dry technique) will result in the highest tensile strength. As suggestion, further research is needed to examine the tensile bond strength of HEMA dentin bonding agent with dentin surface at various pH of HEMA, HEMA concentration, and acetone concentration.

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