

# Chemical Bond Strength Difference between 4-Meta Bonding Agents with Ethanol and Acetone Solvent on Type I Collagen

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## Chemical Bond Strength Difference between 4-Meta Bonding Agents with Ethanol and Acetone Solvent on Type I Collagen

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### Abstract

Bonding material is a coupling agent required in performing composite restoration on caries and dentine erosion. 4-META is one of the materials often used as bonding material. Bonding material contains solvent agent which promotes monomer infiltration to collagen. The most commonly used solvents are acetone and ethanol.

The aim of this studies was to determine the chemical bond strength difference between 4-META with ethanol and with acetone on type I collagen. Materials and methods: The sample of this study was divided into three groups: mixture of 4-META and collagen as control group (C); mixture of 4-META, ethanol and collagen (T1); and mixture of 4-META, acetone, and collagen (T2) as treatment groups. The samples from all groups pelleted using KBr before being analyzed by means of FTIR. One Way ANOVA and Tukey HSD test were used to analyze the results data ( $p < 0.05$ ). Results: Carbonyl absorbance bands peak (P) value of C group was 75.15, while T1 was 47.91 and T2 was 28,18. The smaller P value, the greater the bonding strength of the chemical.

The chemical bond strength of the 4-META and acetone bonding material is greater than soluble ethanol on type I collagen.

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### Introduction

An adequate restoration is required to treat tooth erosion and caries cases. Composite resin is widely known as a restorative material with a satisfaction result. In enamel erosion and caries tooth cases, a wide area of enamel removal is unnecessary as acid etched and bevel structure around the cavity are already enough as the retention of restoration because surface produces higher bond strength due to the larger surface area and additional mechanics.<sup>1,2</sup> Etch substance will dissolve hydroxyapatit enamel to produce porosity on tooth, which will be filled by the composite resin as multiple resin tags to provide the attachment on tooth surface.<sup>3</sup> In contrast, for dentine erosion and caries tooth cases, a

preparation of bevel structure alone could not provide enough attachment. Surface produces. The application of bonding agent prior to composite restoration is necessary to increase retention between composite and tooth surface.<sup>4</sup>

On the crown area, dentine is located under the enamel, while on the root area it was under cementum. Dentine become roof of pulp chamber which contains blood vessels and free-end nerves.<sup>5</sup> Dentine is anatomically related to pulp tissue through small channel called dentine tubuli. Organic compound in dentine is 30% greater than organic compound in enamel. Macroscopically, dentine tubuli is channel-shaped like which contained odontoblast processes and dentine tubuli fluid.<sup>6</sup>

Current bonding agent development is focusing on bonding agent for dentine to achieve bonding attachment to dentine and cementum.<sup>6</sup> Common bonding agent usually contains specific functional monomer ester as the result of bivalent alcohol reaction with metacrylate acid and phosphor or carboxyl acid-derivat. Among all functional monomer as an adhesive agent, 4-

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META (4-Methacryloxyethyl trimellitic anhydride) is the most common used as an adhesive agent.<sup>7</sup> 4-META has been used widely because its high biocompatibility and excellent attachment resistance on dentine and cementum surface. This material is also both hydrophilic and hydrophobic.<sup>3</sup>

Solvent is added to monomer to facilitate the monomer to infiltrate into the collagen. The function of solvent agent is to solve or dilute bonding agent into less thick.<sup>8</sup> Acetone and collagen are the most used as a solvent agent.<sup>9,10,11</sup>

Recent study about dentine bonding discovered that specific functional monomer can interact chemically with tooth surface. The benefit from chemical interaction on dentine bonding showed there is contribution in the work system of bonding agent which prolonged adhesive bonding.<sup>12</sup> Chemical bond between 4-META with collagen is covalent bond or primary bond between atom. On chemical reaction, covalent bond is formed between nitrogen atom from collagen and carbon atom resin aldehyde.<sup>4</sup>

Detection of chemical bond between adhesive agent and collagen has become controversial issue in many literature, therefore some techniques are used to determine the chemical bond on adhesive agent like using XPS (X-ray Photoelectron Spectroscopy), micromechanical test, and specific tool using infrared spectrum system, Fourier Transform Infra-Red (FTIR) spectroscopy, which has special characteristic to detect penetration and interaction of specific adhesive agent into biologic tissue.<sup>13</sup>

Based on the description above this study was done to determine the chemical bond difference between 4-META with ethanol and 4-META with acetone bonding agent on type I collagen by means of FTIR spectroscopy.

## Methodology

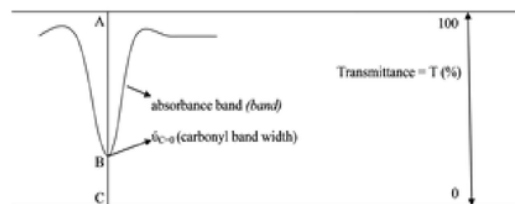
### Procedures

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This study is experimental laboratory with post-test only control group design. It used type-1 collagen (bovine) as a sample. The total sample of this study was 18 and were divided into 3 different groups: mixture of 4-META (Chemlin, Nanjing, China) and collagen (Sigma-Aldrich, Missouri, USA) as control group (C); mixture of 4-META, ethanol (Merck, Darmstadt, Germany)

and collagen (T1); and mixture of 4-META, acetone (Smart Lab Indonesia, South Tangerang, Indonesia), and collagen (T2) as treatment groups.

For the control group, 4 mg collagen was mixed with 20  $\mu$ l 4-META, while in T1 group, 200  $\mu$ l ethanol was added to the mixture, and for T2 group, 200  $\mu$ l acetone was added. KBr (Merck, Darmstadt, Germany) was added until the total volume of 672.4mg was reached in each group mixture before being mixed until the consistency went homogenic. 50 mg of each mixture was inserted into KBr die to be vacuum pressed with 10-ton pressure to produce transparent pellets. Pellets from each group were observed using FTIR spectroscopy (Perkin Elmer, Massachusetts, USA). From FTIR spectroscopy result, Peak (P) of carboxyl group (C=O) was measured using formula  $P = BC/AB \times 100$  as shown in figure 1.



**Figure 1.** Peak (P) of carboxyl group (C=O) was measured using formula  $P = BC/AB \times 100$ .

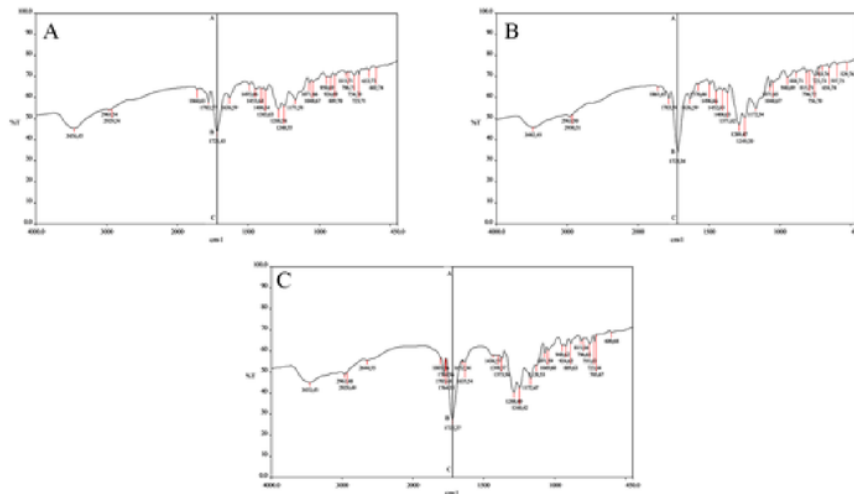
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### Statistical analysis

Data were analyzed with SPSS ver. 17 (IBM Company, New York, USA) using One Way ANOVA and Tukey HSD test. Tests were performed with a 5% level of significance.

## Results

The result observed from three groups by FTIR spectroscopy was shown on figure 2. Table 1 showed there is a significant different between C, T1 and T2 groups with  $p=0,000$  ( $p < 0,05$ ). The mean of P value in T2 groups was the lowest.



**Figure 2.** The result observed from three groups by FTIR spectroscopy.

Groups	Mean±SD
4-META +collagen (C)	75.15±7.13 <sup>a</sup>
4-META +ethanol + collagen (T1)	47.91±9.13 <sup>b</sup>
4-META +acetone + collagen (T2)	28.18±6.2 <sup>c</sup>

**Table 1.** The mean of P Value C, T1 and T2 groups.

a,b,c different superscripts show a significant difference (p<0,05).

### Discussion

Dentine are made up from 60% inorganic material in hydroxyapatit  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$  form; 30% organic material and 10% water. 90% of organic material are consisted of collagen, and the rest 10% are from non-collagen material.<sup>13</sup>

Attachment of dentine bonding to dentine collagen can be both mechanically or chemically. Mechanical attachment of dentine bonding to collagen can be occurred when dentine bonding penetrating into nano interfibriler space and hardened or polymerized into mechanical anchorage. Some theory considered chemical attachment of dentine bonding and collagen as

the result of chemical interaction between functional group of amino collagens with resin carbonyl group, which formed into peptide bond.<sup>3</sup>

Chemical bond of resin 4-META with collagen which was observed using Saturation Transfer Difference NMR showed that the strenght of chemical bond is determined by the ability of monomer functional group to bind molecules from collagen.<sup>14</sup> There is hydrophobic interaction between hydrophobic functional group from 4-META with hydrophilic collagen surface.

Based on hybrid layer formation concept, to achieve a strong and everlasting bonding, dentine demineralization followed by an adequate resin infiltration and polymerization are required. Monomer infiltration to dentine tubules could be enhanced by adding solvent to bonding agent.<sup>15</sup>

In this study, 4-META with acetone and ethanol solvent was used to determine the chemical bond of the substance with type I collagen. Result showed that T1 and T2 groups showed a low carbonyl absorbance band peak value than C groups. The lower P value, the more molecule from bonding agent were bonded to collagen, therefore the chemical bonding was getting stronger. It is in an accordance with previous study which stated that solvent in bonding agent will give optimal bonding between the monomer in bonding agent and collagen in dentine.<sup>9</sup>



Solvent have important role in dentine bonding agent penetration into collagen. Solvent have chemical and physical properties which affect dentine bonding agent penetration, including viscosity, surface tension, solubility parameter and H-bonding capacity, which able to increase both functional group-monomer and functional group-collagen chemical bonding. The most common solvents in bonding agent are ethanol and acetone due to their good chemical and physical properties.<sup>9</sup>

The use of ethanol and acetone also affects the shear strength of monomer. Monomer with solvent has higher shear strength compared to monomer without solvent. High evaporation rate of those solvents caused water evaporation from dentine surface and increased monomer penetration into collagen fibrils.<sup>16,17</sup>

The lower P value in T2 group compared to T1 and C groups was caused by carboxylate group in 4-META dentine bonding agent which belongs to hydrophilic polar compounds bind to aromatic group which belongs to hydrophobic non-polar compounds, which well soluble in acetone compare to ethanol. Based on these results, then it can be concluded that the most optimum solvent to 4-META was acetone.<sup>18</sup>

Acetone solvent has high dipole moment combined with low dielectric constant which allow polar and non-polar compounds to solve, therefore acetone became appropriate choice for dentine bonding agent with hydrophilic and hydrophobic properties. Acetone has lower hydrogen bond formation with ketone (C = O) than alcohol (-OH). However, acetone has good water-removing capacity (so-called water chaser). Therefore, if acetone dissolves 4-META there will be high evaporation, the decrease of residual water and finally allows the monomer to concentrate into the dentine.<sup>18</sup>

Acetone possesses low H-bonding capacity, therefore acetone solvent cannot be used in dry-bonding technique, since it will not be able to recover collagen fibrils after collapsing due to demineralization. Acetone can only be used in moist areas.<sup>7</sup> This theory also supported that optimal humidity to achieve good attachment between dentine bonding agent and collagen fibril 70% because in that condition, water around fibril is very optimal for chemical interaction of dentine bonding monomer with collagen fibril.<sup>3</sup>

Ethanol solvent it is less optimal when used as a solvent in a carboxylic acid group such

as 4-META due to esterification reaction of carboxylic group with hydroxyl group, in particular under acidic conditions which can result in the inactivation of the carboxylic group for the demineralization process and the adhesion ability.<sup>18</sup>

Ethanol is a protic solvent which forms the hydrogen bond with the solute. But, it has lower dielectric constant which prevent the formation of hydrogen bond and result in reduced monomer attachment with collagen fibrils. The water chasing capability possessed by ethanol is lower than those of acetone so that the evaporation of ethanol is not as high as acetone, resulting in the remaining water on the surface of the dentine is still abundant and causing difficult to penetrate into collagen fibrils.<sup>18</sup>

### Conclusion

From this study, it can be concluded that the chemical bond strength of the 4-META bonding agents with acetone solvent is greater than with ethanol solvent on type I collagen.

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