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Subject: Manuscript Submission ID#PBOCI-2020-0116 - Pesqui Bras Odontopediatria
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PBOCI
PESQUISA BRASILEIRA EM
ODONTOPEDIATRIA E CLÍNICA INTEGRADA

November, 10th 2020

Dear authors,

**Nanik Zubaidah, Ruslan Effendy, Adioro Soetojo, Tri Estiyaningsih, Muhammad Iqbal Tanzil,
Khusnul Khotimah**

It is a pleasure to accept your manuscript entitled "**Difference of Chemical Bonds Between UDMA Bonding Agents with Ethanol Solvent and Acetone Solvent on Dentin Collagen**" (PBOCI-2020-0116) in its current form for publication in the Pesquisa Brasileira em Odontopediatria e Clínica Integrada.

Your paper will be published in the next issue of the journal (issue 21 /2021).

We clarify that, due to the pandemic, we have a reduced team in some sectors of the journal, causing the need for a longer period for publication of accepted articles.







For this reason, we expect the manuscript will be published within four months.

Sincerely,

Prof. Ana Maria Gondim Valença

Editorial Office, Pesquisa Brasileira em Odontopediatria e Clínica Integrada

Difference of Chemical Bonds Between UDMA Bonding Agents with Ethanol Solvent and Acetone Solvent on Dentin Collagen

Nanik Zubaidah¹, Ruslan Effendy¹, Adioro Soetojo¹, Tri Estiyaningsih², Muhammad Iqbal Tanzil², Khusnul Khotimah²

¹Department of Conservative Dentistry Faculty of Dental Medicine, Universitas Airlangga, Surabaya, Indonesia.

²Conservative Dentistry Specialist Program, Faculty of Dental Medicine Universitas Airlangga, Surabaya, Indonesia.

Correspondence: Nanik Zubaidah, Department of Conservative Dentistry, Faculty of Dental Medicine, Campus A Universitas Airlangga, Jl. Mayjen Prof. Dr. Moestopo No. 47, Surabaya 60132, Indonesia. **E-mail:** nanik-z@fkg.unair.ac.id

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ABSTRACT

Objective: To investigate the difference of chemical bonds between urethane dimethacrylate (UDMA) bonding agents with ethanol solvent and acetone solvent on dentin collagen. **Material and Methods:** This experimental comparison study used three groups: G1 (Control): UDMA and collagen; G2: UDMA, collagen and ethanol; and G3: UDMA, collagen and acetone. The groups were then pelleted and analysed with FTIR, then the peak value of carbonyl absorption band from each study group was calculated. The result of FTIR analysis and the peak of carbonyl absorption band (P) was calculated using the formula: $P = (BC / AB) \times 100$; AB. BC is measured in centimeters. The study of chemical bond differences between ethanol-solvent UDMA agents compared with acetone-solvent on dentin collagen resulted in a graph of peak of carbonyl absorption bands of UDMA and dentin collagen groups. To determine the chemical bonds of UDMA from the top of the carbonyl ester absorption bands with wavenumber absorption in range 1700-1750 cm⁻¹, the decreasing peak of the carbonyl absorption bands is assumed as more chemical bonds that formed. Data were analysed using Anova one way and Tukey HSD test. **Results:** There were significant differences between the three study groups ($p < 0.05$). **Conclusion:** UDMA bonding agents' chemical bonds with acetone solvent are much higher than the chemical bonds between UDMA bonding agents with ethanol solvent on dentin collagen.

Keywords: Dental Materials; Dental Bonding; Dentin; Dentin-Bonding Agents.

Introduction

Composite resin restoration material is being developed continuously, specifically in terms of aesthetics, usage and manipulation, to improve the bonding of composite resins to the dentin [1]. The attachment of composite restoration to dentin requires a bonding agent that works in two ways, namely forming mechanical bonds through the enamel tags formed by etch and building hybrid layer on dentin to form a chemical bond. A chemical bond is formed between the composite resin and the hydroxyapatite on dentin [1]. Despite the development of adhesive system, the least weak area of the restoration remains the bonded interface, and one of the factors that play a role in composite restoration is dentin bonding [2].

Urethane dimethacrylate (UDMA) is one of the methacrylate derivatives that is frequently used as a bonding agent. UDMA has good properties, such as low viscosity, perfect flexibility and good endurance intraoral due to its ability to inhibit transesterification. UDMA-based composites have low water-absorbing characteristics and low solubility [3,4]. The chemical bonds of the UDMA contained in the factory packaging have been previously investigated [5]. Shin et al. [6] also conducted a survey about the influence of hydrophobic monomers on ethanol solvents.

The solvent contained in the bonding material denotes the key agent in forming dentin and resin interfaces. The solvent plays a role in carrying resin monomer molecules into the dentin tubule. The substances commonly used as a solvent in dentin bonding agents are acetone, ethanol or water. Acetone and ethanol have different physical and chemical characteristics, which naturally affect the bond strength of the dentine bonding material [7-9].

This research is based on the existence of several phenomena, potentially causing composite restoration failure. To date, there has been no specific research about the effect of ethanol and acetone solvent toward the chemical bond strength of bonding agent, pure UDMA. Therefore, we aim to investigate the difference of chemical bonds between UDMA bonding agents with ethanol solvent and acetone solvent on dentin collagen [5,6].

Material and Methods

This study used pure UDMA agents (Sigma Aldrich, St. Louis, Missouri, USA), pure bovine collagen Type 1 (Sigma Aldrich, St. Louis, Missouri, USA), ethanol 98% (Smart Lab Indonesia, Surabaya, Indonesia), acetone 98% (Smart Lab. Indonesia, Surabaya, Indonesia) and KBr powder as materials, and using tools: Jasco FTIR 5300, Mortar and Pestle, KBr die, Presses, Balance Sheet and Micro Pipette.

Experimental Procedure

Group I (Control)

An amount of 2 mg collagen is added with 10 µl pure UDMA and potassium bromide powder (KBr) until the weight reaches 311.1 mg, then ground with mortar and pestle until it becomes a homogenous mixture. The mixture is taken 50 mg and blended into KBr die and then compressed with a 10-ton compressing machine for 5-10 minutes to make clear pellets (thin tablet-shaped). KBr is used as the background matrix because it is IR transparent due to the plasticity of its alkali halides [10]. Then these pellets are observed with the FTIR tool.

Group II (Ethanol)

An amount of 2 mg collagen is added with 10µl pure UDMA (weight 11.1 mg) and potassium bromide powder (KBr) until the weight reaches 311.1 mg, then add 20 µl ethanol. All ingredients are mixed and grinded with mortar and pestle until it becomes a homogeneous mixture. Furthermore, 50 mg of mixture was taken and blended into KBr die and compressed with a 10-ton compressing machine for 5-10 minutes to make clear pellets (thin tablet-shaped). These pellets are then observed with the FTIR tool.

Group III (Acetone)

An amount of 2 mg collagen is added with 10µl pure UDMA (weight 11.1 mg) and potassium bromide powder (KBr) until the weight reaches 311.1 mg, then add 20 µl acetone. All ingredients are mixed and crushed with mortar and pestle until it becomes a homogeneous mixture. Furthermore, 50 mg of mixture was taken and blended into KBr die and compressed with a 10-ton compressing machine for 5-10 minutes to make clear pellets (thin tablet-shaped). These pellets are then observed with the FTIR tool.

The result of FTIR analysis and the peak of carbonyl absorption band (P) [11,12] is calculated using the following formula: $P = (BC / AB) \times 100$; AB, BC is measured in centimeters. This calculation is accurate and reliable if the absorption band's intensity is ranged T: 30–60.

The study of chemical bond differences between ethanol-solvent UDMA agents compared with acetone-solvent on dentin collagen resulted in a graph of peak of carbonyl absorption bands of UDMA and dentin collagen groups (Figure 1). To determine the chemical bonds of UDMA from the top of the carbonyl ester absorption bands with wavenumber absorption in range 1700-1750 cm^{-1} , the decreasing peak of the carbonyl absorption bands is assumed as more chemical bonds that formed.

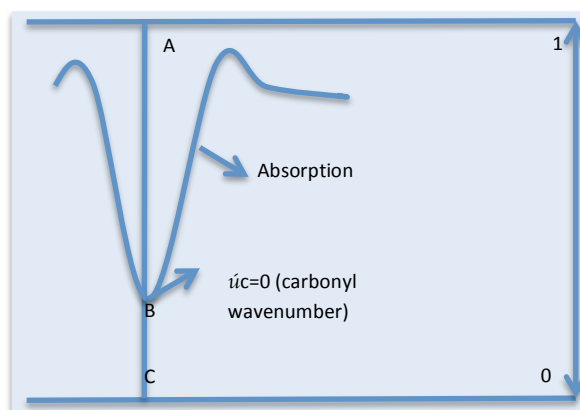


Figure 1. Peak of carbonyl absorption bands of UDMA

Data Analysis

The obtained data is a peak of carbonyl absorption bands (P) that is analysed by normality test by using Kolmogorov-Smirnov and homogeneity test by using Levene test method. Comparative test for data analysis using One Way ANOVA test when data normally distributed and followed by Tukey HSD.

Results

Figure 2 shows the peak of carbonyl ester absorption bands of UDMA at wavelength absorption 1720 cm^{-1} , the peak of carbonyl absorption bands (P) is calculated using the following formula: $AB = 50$; $BC = 50$; $P = BC / AB \times 100 = 50/50 \times 100 = 100$.

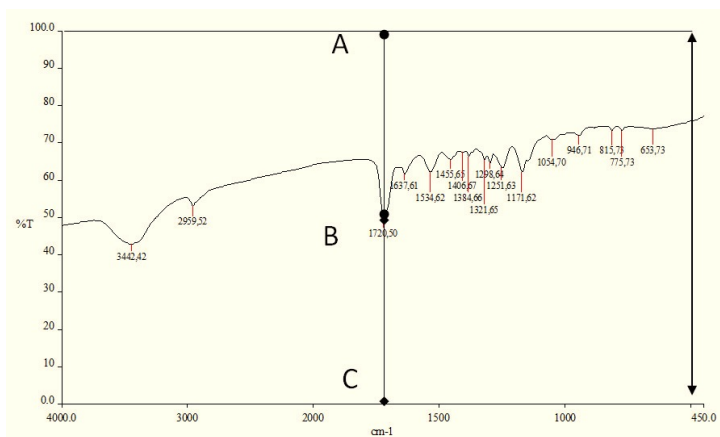


Figure 2. Results of UDMA and collagen.

Figure 3 shows the peak of carbonyl ester absorption bands of UDMA at wavelength absorption 1720 cm^{-1} , the peak of carbonyl absorption bands (P) is calculated using the following formula: $AB = 33$; $BC = 67$; $P = BC / AB \times 100 = 33/67 \times 100 = 49.25$.

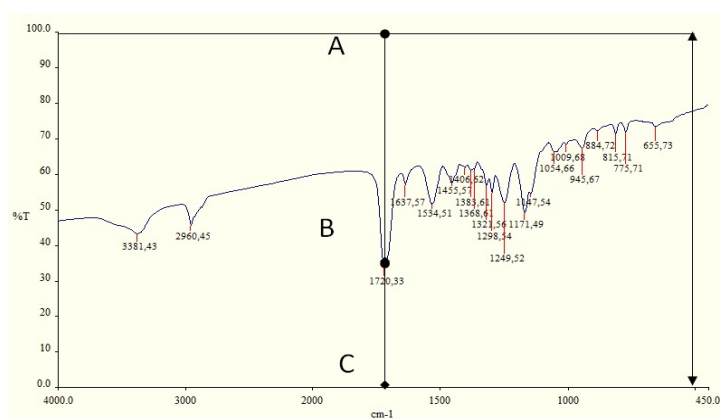


Figure 3. Results of UDMA, ethanol and collagen.

Figure 4 shows the peak of carbonyl ester absorption bands of UDMA at wavenumber absorption 1720 cm^{-1} , the peak of carbonyl absorption bands (P) is calculated using the following formula: $AB = 26$; $BC = 74$; $P = BC / AB \times 100 = 26/74 \times 100 = 35.13$

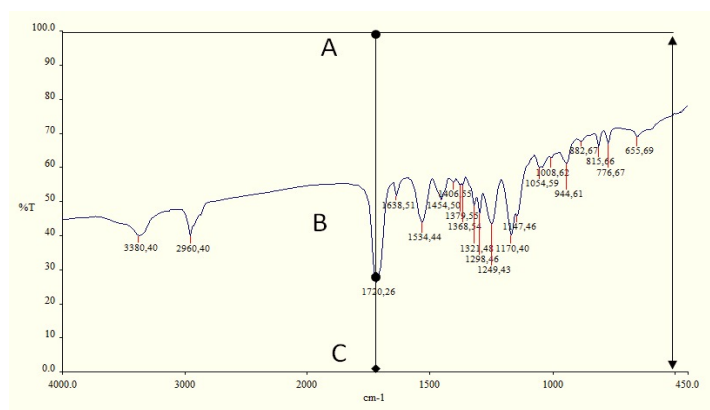


Figure 4. Results of UDMA, acetone and collagen.

There was a significant difference of peak of carbonyl absorption band between the whole group ($p < 0.05$) (Table 1).

Table 1. Mean and standard deviation of peak of the carbonyl absorption bands (P).

Group	N	Mean	SD	p-value*
G1 (UDMA and Collagen)	9	110.6 ^a	9.35	0.000
G2 (UDMA, Collagen and Ethanol)	9	52.38 ^b	8.15	
G3 (UDMA, Collagen and Acetone)	9	25.73 ^c	3.43	

*One Way ANOVA test; Different letter shows a significant difference.

Measurement using Tukey HSD was performed to investigate the difference of carbonyl absorption bands between groups. There was a significant difference between UDMA and collagen group, UDMA with collagen and ethanol, and group of UDMA, collagen, and acetone ($p < 0.05$).

Discussion

The study of solvent contained in dentin bonding suggested that it has an important role as the dentin bonding penetrates the dentin collagen, considering it has physical and chemical properties that help improve dentin bonding into dentin collagen. The addition of solvent affects the chemical bonds between functional groups in monomers and dentin collagen [1,7].

This research used pure UDMA and solved into acetone and ethanol, considering that the commercially available UDMA may contain any additive materials which may cause potential bias [5,7]. UDMA has characteristics of fully aliphatic homopolymer and chemically and physically cross-linked [13]. UDMA can form both chemical (covalent and hydrogen bonds) and mechanical bond to the dentin, specifically in dentinal collagen.

The limit of this research is, we are unable to get the sample with the same level of clearness during sample making because collagen fibril is an elastic tissue, so it is difficult to grind; this can be overcome by checking the sample mixture with dental explorer, if there is still a lump collagen fibers, grinding is continued until the lump completely mixed and becomes homogeneous. Besides, the other difficulty is the physical properties of UDMA agents that are sticky and the solvent evaporates rapidly; this can be solved by mixing UDMA and solvent first so that the mixture becomes more dilute mixing process should be done more quickly.

The data analysis recorded the peak value of carbonyl absorption bands on dentin collagen between the group of UDMA and collagen, and group of UDMA with collagen and ethanol indicated a significant difference ($p < 0.05$); this means chemical bonds in UDMA and ethanol is higher than chemical bonds in UDMA and collagen, with the addition of ethanol solvent, it is capable of restoring the collapsed collagen fibril that occurs due to excessive drying because of having large H-bonding capacity. In addition to that, ethanol is able to control the width of the interfibrillar cavity during the evaporation process so that keeping a good diffusion between monomers.

This study's data showed a significant difference between UDMA with collagen group and UDMA and acetone with collagen ($p = 0.000$). UDMA solved in acetone had a stronger chemical bond. This is probably due to acetone's ability to increase the mixture viscosity, allowing monomer to penetrate into the collagen fibrils.

The study group of UDMA, collagen and ethanol, and UDMA, collagen, and acetone indicated a significant difference ($p < 0.05$). Ethanol is a protic solvent, a protic solvent is a solvent that forms hydrogen bonds with its solute because formed hydrogen bonds will disrupt the new formed covalent bonds between

UDMA and collagen so that the bond becomes unstable. Ethanol has a much lower dielectric constant. It is also an improper solvent for its polar solute. When reacted with a polar compound, the hydrogen bond will not be formed and decrease monomer attachment with collagen fibrils [12].

Aside from the drying technique, the solvents contained in dentin bonding also plays a role in composite restoration bond strength. Some solvents characteristics that have an important role in dentin bond strength are hydrogen bonding capacity, vapour pressure, boiling point, dielectric constant, and dipole moment. A study found that monomers used together with solvents have a higher shear strength than those that do not [14-16].

Solvents play an essential role in the process or penetrating dentin bonding to collagen since it acts as water chasers that displace the water and carry the resin monomers into the remineralized dentine [7,14]. This is due to the acetone and ethanol solvents having strong evaporation power, which evaporates the moisture content of the dentine surface and penetrates the monomers into collagen fibrils [17]. The evaporation pressure of acetone is high and the remaining water on the dentine surface is reduced, causing the monomer material becomes easier to penetrate into collagen. Meanwhile, ethanol's water-chasing ability is weaker than acetone; considerable amounts of water remains on dentine's surface and cause the monomer material becomes difficult to penetrate into collagen fibrils. The less monomers bind to collagen, the weaker chemical bonds that can cause the adhesive strength decreased [18].

Previous authors showed that dentin bonding with ethanol solvent was able to penetrate deeper into dentin intratubular than those with water solvent [6]. This might be due to ethanol evaporates slower than water; thus, it can carry the adhesive material deeper into the intratubular dentin. The same result was also recorded by Guo et al. [19], which stating that the use of ethanol as solvent can distribute the adhesive material deeper and wider into the intratubular collagen, thus increasing the composite resin attachment. However, adding ethanol to the substance with the carboxyl group may inactivate its chemical activity due to the esterification reaction between the carboxyl group from ethanol and UDMA, specifically in an acid environment. This might impair the adhesive ability [20].







The aromatic group contained in UDMA is hydrophobic and able to neutralize the acidity of carboxyl group. Therefore, the dentin bonding with UDMA is expected to function optimally with acetone as solvent [7]. The solvent may affect the adhesive strength due to the chemical reaction with UDMA resin. Acetone solvent is an aprotic solvent, adding acetone to UDMA may establish a more stable bonds. Acetone solvents do not donate H atoms so that acetone does not disturb the covalent bonds formed between UDMA and collagen [11].

On the dentin surface, there is water, so that UDMA dentin bonding is difficult to penetrate the collagen fibrils; ethanol has the ability of water chasing so that the amount of water remaining in the dentin is decreased. The ability of water chasing ethanol is lower than that of acetone, so that the evaporation of ethanol is not as big as acetone. This may cause the remaining water in dentine is decreasing; thus, it will be easier for the monomer to penetrate into dentin [12].

Conclusion

UDMA bonding agents' chemical bonds with acetone solvent are much higher than the chemical bonds between UDMA bonding agents with ethanol solvent on dentin collagen. Further research about the difference in bond strength with different solvents is required.

Authors' Contributions

NZ		https://orcid.org/0000-0002-3205-6071	Conceptualization, Formal Analysis, Data Curation, Writing - Original Draft, Writing - Review and Editing, Supervision and Project Administration.
RE		https://orcid.org/0000-0003-2939-8983	Conceptualization, Methodology, Formal Analysis, Data Curation, Writing - Original Draft, Writing - Review and Editing and Supervision.
AS		https://orcid.org/0000-0002-8632-5492	Conceptualization, Formal Analysis, Investigation, Writing - Original Draft and Writing - Review and Editing.
TE		https://orcid.org/0000-0001-8247-7678	Methodology, Investigation, Writing - Original Draft and Writing - Review and Editing.
MIT		https://orcid.org/0000-0001-8760-7136	Methodology, Investigation, Writing - Original Draft and Writing - Review and Editing.
KK		https://orcid.org/0000-0001-7015-1523	Methodology, Investigation, Writing - Original Draft, Writing - Review and Editing, Visualization and Project Administration.

All authors declare that they contributed to critical review of intellectual content and approval of the final version to be published.

Financial Support

None.

Conflict of Interest

The authors declare no conflicts of interest.

Data Availability

The data used to support the findings of this study can be made available upon request to the corresponding author.

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Alessandro Cavalcanti <alessandrouepb@gmail.com>

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To: nanik-z@fkg.unair.ac.id Cc: ruslan-e@fkg.unair.ac.id, adioro-s@fkg.unair.ac.id

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