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Research Report

# The difference between residual monomer dentin bonding HEMA and UDMA with acetone and ethanol solvent after binding to type I collagen

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***ABSTRACT***

***Background:*** *In caries and non-caries lesions involving dentine, it is necessary to provide dentine-bonding material to help improve retention between the composite resin and the tooth surface. Composite resin attachment to dentine is influenced by bonding polymerization reactions. In several studies, researchers found that polymerized monomers will experience volume shrinkage because not all will fully polymerize but, rather, become residual monomers that can cause post-operative pain.* ***Purpose:*** *This study aimed to identify the difference in the amount of residual monomers between HEMA- and UDMA-based dentin bonding materials with acetone and ethanol solvents after binding to type I collagen.* ***Methods:*** *Four groups featured in this study: HEMA with acetone solvent and type I collagen , HEMA with ethanol solvent and type I collagen , UDMA with acetone solvent and type I collagen and UDMA with ethanol solvent and type I collagen . All groups were checked by high performance liquid chromatography (HPLC) to quantify the remaining amount of monomers.* ***Results:*** *The percentage of residual monomers of dentine bonding HEMA with acetone solvent and type I collagen was 10.69%, HEMA with ethanol solvent and type I collagen was 13.93%, UDMA with acetone solvent and type I collagen was 2.89% and UDMA with ethanol solvent and type I collagen was 7.48%.* ***Conclusion:*** *HEMA with ethanol solvent has the highest number of residual monomers, while UDMA with acetone solvent has the lowest.*

***Keywords:*** *acetone; ethanol; HEMA; residual monomer; UDMA*

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## INTRODUCTION

Dentin is a perpetually wet hard tissue because it contains dentinal tubular fluid that renders composite resin with hydrophobic properties incapable of attaching to dentine. Therefore, a bonding material is required to glue dentin to composite resin. In widespread cervical lesions extending as far as the dentine and/or near the cementum dentin bonding is necessary to help increase retention between the composite resin and the tooth surface.[1]

The adhesiveness of dentin bonding to dentine collagen fibrils also constitutes an important interaction. Dentin bonding can penetrate the nano interfibrillar cavities before polymerizing to mechanically form anchorage. Polymerized monomers will experience volume reduction because not all monomers undergo complete polymerization becoming residual monomers, namely ones which do not react after polymerization is in process.[2]

Common bonding is generally based on 2-hydroxyethyl-methacrylate (HEMA), but recently many non-HEMA based bonding materials have been developed. Generally, HEMA substitutes present in non-HEMA based bonding materials are monomer dimethacrylates such as urethane dimethacrylate (UDMA).[3] In order to promote deeper monomers penetration of the dentine, the bonding material contains solvents which play a role in transporting the monomers to the tooth, promoting dissolution of the thick monomers and facilitating penetration of the demineralized dentin. Acetone is known to have a very high vapor pressure, while that of ethanol is lower.[4]

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Determination of the number of residual monomers eluted from the dental material is usually carried out with high-performance liquid chromatography (HPLC) which constitutes an extremely strong separation method.[5] In several studies, HPLC has been used to measure residual monomers in resin materials because they are capable of detecting soluble and non-volatile reactive compounds such as bisphenol-a glycidyl methacrylate (Bis-GMA), urethane dimethacrylate (UDMA) and triethylene glycol dimethacrylate (TEGDMA).[6] The purpose of this study was to prove the existence of monomer differences in residual HEMA and UDMA dentin bonding with acetone and ethanol solvent after binding to type I collagen.

## MATERIALS AND METHODS

The research sample consisted of acetone-coated HEMA dentin bonding material (Solobond M, Voco, Germany), ethanol-based HEMA (Tetric N-bond, Ivoclar vivadent, Liechtenstein), UDMA acetone (Spectrum bond, Dentsply, Germany), ethanol-coated UDMA (Tetric N-bond, Ivoclar vivadent, Liechtenstein) and collagen type I (Sigma chemical, St. Louis, USA).

For the purposes of this study, the subjects were divided into four groups, group 1: HEMA dentin bonding with acetone + type I collagen, group 2: HEMA dentin bonding with ethanol + type I collagen, group 3: UDMA dentin bonding with acetone + type I collagen and group 4: UDMA dentin bonding with ethanol + type I collagen . In order to conduct the research, 100 µl standard solution was produced from pure HEMA (Sigma Chemical, St. Louis, USA) and UDMA (Sigma Chemical, St. Louis, USA) and added to 900 µl methanol (methyl alcohol, Mallinckrodt Chemical, USA) because pure HEMA and UDMA can only dissolve in methanol. This standard solution is only a standard reference for monomers that will be detected on inspection by high performance liquid chromatography (HPLC) devices.

1 ml of dentin bonding material was added to 100 mg of type I collagen before each sample was irradiated for 20 seconds by means of a light curing unit (DBA, Guilin Woodpecker, China) at a wavelength of 550nm and then immersed in 10 ml ethanol. Standard solutions and samples were taken using a 1 ml syringe and then injected into a filter holder previously filled with nylon membrane and accommodated in a closed vial. In the final step, they were analyzed using HPLC (Agilent 1100 series, Agilent, Germany).

The time at which a specific sample is eluted is referred to as the retention time. The samples measured by HPLC for the analysis of residual monomers require a standard reference from the monomers to be detected and compared with existing standards to determine the monomers to be measured. The HPLC results in the form of a chromatogram provide information about the retention time and sample area (Figure 1). Reading the chromatogram involves looking at the treatment area results. The results of the sample area are compared to those of the standard area before being calculated using a formula in order that quantitative results are obtained.

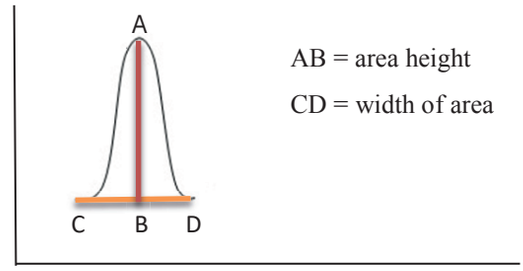


**Figure 1.** Illustration of HPLC calculation result

The results of the remaining monomers were calculated by percentage. To determine the percentage of residual monomers from the research sample after treatment the following formula can be used:[7]

$$\% \text{ Residual monomer} = \frac{\text{sample area } (AB \times CD)}{\text{standard area } (AB \times CD)} \times 100\%$$

## RESULTS

Based on the results of the study, it can be seen that the UDMA with acetone solvent + type I collagen group produces the lowest number of residual monomers, while the HEMA with ethanol solvent + type I collagen group produces the highest (Table 1).

To identify the distribution of data in the study groups, a Kolmogrov-Smirnov test was conducted. Of the four groups tested statistically, all showed a value of $p > 0.05$ signifying normal data distribution. Furthermore, in the four study groups, homogeneity tests conducted by means of a Levene test produced a result of $p = 0.003$ ($p > 0.05$) indicating that the samples were not homogeneous. These were then subjected to a Kruskal-Wallis test as a means of identifying differences between groups, the $p = 0.000$ ($p < 0.05$) result obtained confirmed differences between the four study groups.

In order to establish the differences in each experimental group a Tukey HSD analysis test was conducted. The test results had a value of $p < 0.05$ across all experimental groups confirming significant differences between them. This showed that the amount of residual dentin bonding monomers of UDMA acetone + type I collagen, UDMA ethanol + type I collagen, HEMA acetone + type I collagen, and HEMA ethanol + type I collagen had significant differences (Table 2).

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## DISCUSSION

Dentin is a hard tissue containing approximately 60% inorganic ingredients, 30% organic matter and 10% water. Organic matter consists of 90% collagen, while the remaining 10% is non-collagen material.[8] Dentin bonding is an agent used as a material to combine composite restorations with dental tissues. It is usually employed in combination with composite resins to reduce the occurrence of microleakage between the material lodged and the tooth surface, while also increasing retention of the filling material.[2]

Several studies of dentin bonding state that specific functional monomers can interact chemically with dental tissues.[9] The polymerization of resin-dentin bonding is obtained using visible light from a light curing unit. This resin can polymerize with visible light because it contains a photo-initiator, camphorquinone.[2] Various dentin bonding, for example HEMA and UDMA[10], containing a range of basic materials has been available on the market. Solvents are often added to bonding adhesive materials, serving to dilute thick monomers and help monomers penetrate demineralized dentine. The most commonly used solvents are acetone and ethanol because they have the optimum physical and chemical properties compared to other solvents.[4]

This research is based on the existence of a number of clinical phenomena potentially causing postoperative pain after filling with composite resin. Several previous studies have posited that the free monomers in dentin bonding material are believed to be one source of pain. It is important to realize that complete polymerization rarely occurs and that residual monomers emerge, consequently causing postoperative pain.[11]

The results showed that ethanol-coated HEMA dentin bonding material contained the largest amount of residual monomer, while acetone-coated UDMA dentin bonding material contained the smallest compared to acetone-coated UDMA and ethanol-coated HEMA dentin bonding material in this study group. An important factor influencing the release of residual monomers is the size of monomers in resin materials since more smaller molecules than larger ones are released.[5] HEMA possesses small, heavy molecules[12], although UDMA has a greater molecular weight.[13]

Solvents play an important role in the process of penetrating dentin bonding to collagen.[4] Monomers that use solvents have a higher shear strength than those that do not. This is due to the acetone and ethanol solvents having strong evaporation power which helps to both evaporate the moisture content of the dentine surface and penetrate the monomers into collagen fibrils.[14]

Acetone, also known as a water-chaser[2], is an effective solvent which helps remove water from dentine. Because the evaporation pressure of acetone is high the remaining water on the dentine surface is reduced, causing the monomer material to be easier to penetrate into collagen. The more monomers that bind to collagen, the stronger the resulting chemical bonds so that the adhesive strength is also greater.[15]

Unlike acetone, the water-chasing ability of ethanol is weak.[2] Because ethanol evaporation is not as great as that of acetone, considerable amounts of water remains on the surface of the dentine and it becomes difficult for the monomer material to penetrate the collagen fibrils. The fewer the monomers that bind to collagen, the weaker the chemical bonds that occur with the result that the adhesive strength is also lower.[15] It can be concluded that ethanol-based HEMA dentin bonding produces the largest number of residual monomers, while acetone-coated UDMA produces the smallest.

**Table 1.** Mean and standard deviation of monomers of residual HEMA and UDMA dentin bonding with acetone and ethanol solvent (%).

| Sampel | n | Mean (%) | SD |
|---|---|---|---|
| HEMA acetone + type I collagen | 7 | 10.6914 | 0.20260 |
| HEMA ethanol + type I collagen | 7 | 13.9314 | 0.24498 |
| UDMA acetone + type I collagen | 7 | 2.8871 | 0.82625 |
| UDMA ethanol + type I collagen | 7 | 7.4843 | 0.17174 |

**Table 2.** Multiple comparisons of Tukey HSD data on HEMA and UDMA dentin bonding with acetone and ethanol solvent.

| Group | UDMA acetone + type I collagen | UDMA ethanol + type I collagen | HEMA acetone + type I collagen | HEMA ethanol + type I collagen |
|---|---|---|---|---|
| UDMA acetone + type I collagen | _ | 0.000* | 0.000* | 0.000* |
| UDMA ethanol + type I collagen | | _ | 0.000* | 0.000* |
| HEMA acetone + type I collagen | | | _ | 0.000* |
| HEMA ethanol + type I collagen | | | | _ |

* $p \leq 0.05$ = there are significant differences

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