

# Time sensitivity associated with application of HEMA-free adhesive

## Abstract

**Background:** Acetone is one of the solvents incorporated in HEMA-free dental adhesives. Since acetone is very sensitive with high vapor pressure, acetone-based adhesives necessitate great care and attention in handling and application.

**Objective:** The aim of this study was to determine the optimal application time of acetone-based HEMA-free adhesive by conducting micro tensile bond strength test (MTBS) at different time intervals.

**Materials and methods:** Twelve freshly extracted non-carious human molars were collected. Two-thirds of the roots were removed and the coronal part of the teeth was subjected to horizontal cross-sectioning in order to remove their cusps and expose the superficial coronal dentin. Then, the teeth were randomly divided into 4 groups (3 teeth each) according to the adhesive application time before curing. One- step self-etch HEMA-free adhesive (G bond; GC, Japan) and universal resin composite (Kalore; GC, Japan) were used in this study. In group (A), application of adhesive was carried out for 15sec as recommended by the manufacturer instructions. In groups (B), (C) and (D), the adhesive was applied for 30sec, 1min and 5min, respectively, before curing. After 24-hour storage in distilled water at 37°C, each tooth was cross-sectioned into micro beams and subjected to MTBS test by using a universal testing machine.

**Results:** The tests showed statistically significant differences among the groups ( $P = 0.00$ ) with each group is different from the others ( $P=0.00$ ). Group A had the highest mean strength value followed by groups B, C and then D with 57.7, 41.6, 29.7 and 17.1 MPa, respectively.

**Conclusion:** The tested adhesive might be sensitive to application time as shown by superior MTBS results when its application time lasts for 15 seconds. This might be explained by evaporation of acetone in one-step acetone-based self-etch adhesives.

**Keywords:** adhesive, dentin, HEMA-free, micro tensile bond strength, tooth

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Aljafari A,<sup>1</sup> Abuzaina O,<sup>1</sup> Qari M,<sup>1</sup> Banakhar N,<sup>1</sup> Bakry AS,<sup>2</sup> Jamleh A,<sup>3</sup> Bakhsh TA<sup>2</sup>

<sup>1</sup>General Dentist, Saudi Arabia

<sup>2</sup>Department of Operative Dentistry, Saudi Arabia

<sup>3</sup>Department of Dentistry, Saudi Arabia

**Correspondence:** Abdulmalek Aljafari, General Dentist, King Abdulaziz University, Saudi Arabia, Tel +966506969576, Email [abdulmalikjt@gmail.com](mailto:abdulmalikjt@gmail.com)

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## Introduction

Methacrylate-based materials are commonly used in the dental profession as bonding agent, filling materials, provisional crowns and bridges, and dental prostheses.<sup>1</sup> Among these methacrylate monomers, especially 2- hydroxyethyl methacrylate (HEMA) is often used in adhesives material.<sup>2</sup>

The water-soluble HEMA has polar properties with small dimension to enhance the wetting properties of the adhesive solution and the penetration efficacy of the adhesive into demineralized dentin.<sup>3</sup> HEMA has been reported to be positively influence bond strength to dentin. Because of its hydrophilic character, it is also frequently added to improve miscibility of hydrophobic and hydrophilic components in an adhesive solution. Van Landuyt et al.<sup>4</sup> found that when the concentration of the HEMA is 10% performed best result regarding to bond strength than the other group with the different concentration of the HEMA (0,19,36 %).<sup>5</sup>

The major drawback of the use of HEMA is its high allergenic potential. Fast penetration of this monomer through gloves and through skin due to its low molecular weight can cause contact dermatitis (allergic reaction type IV).<sup>6,7</sup> HEMA may also deteriorate

the mechanical properties of the polymerized adhesive, which can result in inferior mechanical strength of the cured adhesive, enhanced water uptake, swelling and marginal staining. To overcome these shortcomings, HEMA-free adhesives were introduced. Excluding HEMA, these adhesives are similar to HEMA-containing adhesives and consist of a complex blend of hydrophilic/hydrophobic ingredients, water and an organic solvent (acetone or ethanol) that is added in the formulation to avoid the phase separation and to keep this blend stable.<sup>8,9</sup>

Therefore, the aim of this study was to quantify the optimum application time of acetone-based HEMA-free adhesive by measuring the micro tensile bond strength (MTBS) at different interval-times.

## Material and methods

### Tested materials

Twelve freshly extracted non-carious human molars were collected. The teeth were stored in 4°C in saline solution with 0.02% thymol until use in the experiment. A one-step self-etch HEMA-free adhesive.<sup>5</sup> (G bond; GC, Japan) and a universal resin composite (Kalore; GC, Japan) were used in this study,<sup>10,11</sup> (Table 1).

**Table 1** Tested materials

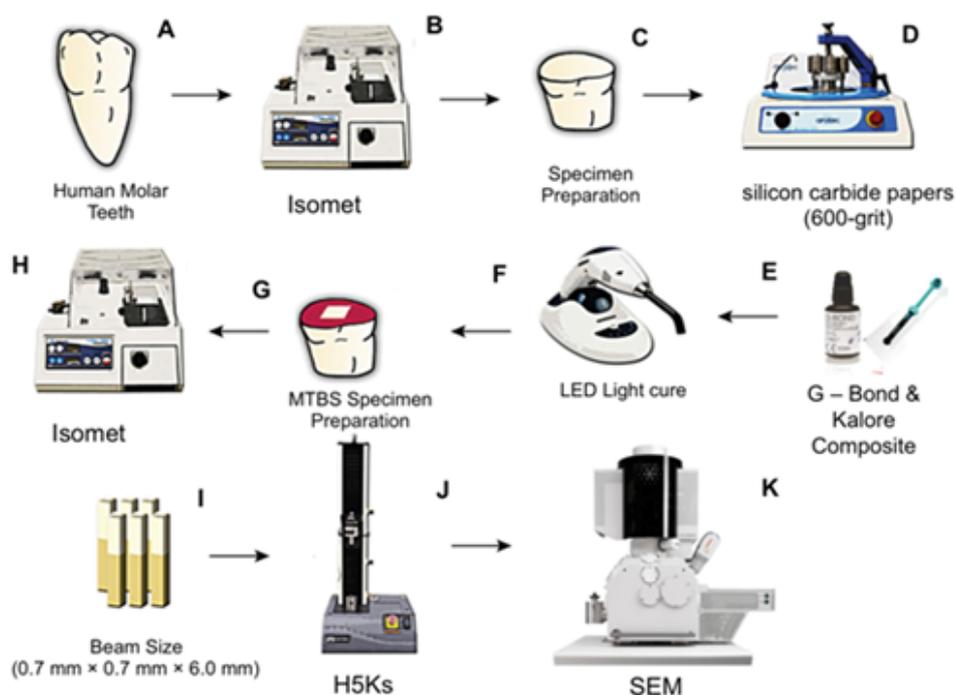
	Composition	Lot Number	Manufacturer
G-Bond Adhesive	4-MET, phosphoric ester-monomer, UDMA, TEGDMA, acetone, water, stabilizer, silica filler, water, photo-initiator	1401111	GC, Tokoyo, Japan
G-Bond Composite	Matrix ( Urethane dimethacrylate, Fillers (Fluoro aluminosilicate glass, pre-polymerized filler, silicone dioxide), Photo initiator, pigment	1203222	GC, Tokoyo, Japan

### Sample preparation

Two-thirds of the root structure was removed in all the teeth using a low speed diamond saw (Isomet; Buehler, USA) under water lubrication. The coronal part of the samples was subjected to horizontal cross-sectioning to remove the cusps of the specimen and to expose the superficial dentin. Furthermore, the occlusal surfaces of the samples were polished with silicon carbide papers (600-grit) under running water to create standard smear layer.

Then, the samples were randomly divided into 4 groups (n=3) according to the differences in the adhesive application time before

curing. In group (Figure 1) (A), application of adhesive was carried out according to the manufacturer instructions within 15 seconds, while the application of the adhesive in (B) was carried out for 30sec before curing. The adhesive in group (C) was applied for 1min before curing and for 5 min in group (D) before curing. Subsequently, each specimen was subjected to air-drying and curing using LED curing unit (400nm-500nm) according to the manufacturer instructions followed by restoration with a universal composite that was applied in two increments (each increment 2mm thickness) and cured according to the manufacturer instruction.

**Figure 1** Sample preparation.

### MTBS specimen preparation and measurement

After 24hours storage in a distilled water at 37°C, each group was cross-sectioned into beams (0.7mm×0.7mm×6.0mm) using a low speed diamond saw (Isomet; Buehler, USA) and subjected to micro tensile bond strength using a universal testing machine (H5Ks, Hounsfield Tinius Olsen, UK) to measure the differences at cross-head speed of 1.0mm/minute. Then each beam was measured using a digital caliper (CD15, Mitutoyo, Kawasaki, Japan).

### Results

Shapiro-Wilk test showed normality in the groups ( $P>0.05$ ), results for the micro tensile strength (MPa) were statistically analyzed using a one way analysis of variance (ANOVA) and LSD post hoc test.<sup>12</sup> The analyses were performed at a significance level of 5% using SPSS 16 software.

It was shown that there are statistically significant differences among the groups ( $P=0.00$ ), with group A having the highest mean

strength value followed by groups B, C and then D with 57.70, 41.60, 29.70 and 17.10 megapascal, respectively.

## Discussion

Although acetone is a common solvent accompanied in HEMA-free adhesives, they are very sensitive and handling them should be accomplished with a great care and attention due to the high vapor pressure (181mm Hg at 20°C).

The results showed an inverse relation between the micro tensile bond strength and the timing of adhesive activation. The absence of HEMA in G-bond adhesive will reduce the water movement from the dentinal tubules into the adhesive layer. However, extending the application time of the adhesive beyond manufacturer recommendation would result in solvent evaporation over the time. Depending on the amount of the evaporated solvent before adhesive curing, the extent of phase separation will occur and this in turn will negatively affects the adhesive mechanical properties over the time.<sup>13</sup>

Also, based on the previous findings, it could be speculated that the behavior of acetone would be more complicated and critical in-vivo situations at 37°C. Thus, delayed activation of the acetone-based HEMA-free adhesives could result in inferior mechanical properties and bond strength.<sup>7</sup>

## Conclusion

The tested adhesive might be sensitive to application time as shown by superior MTBS results when its application time lasts for 15seconds. This might be explained by evaporation of acetone in one-step acetone-based self-etch adhesives and thus manufacturer recommendation should be followed strictly.

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## Conflicts of interest

Authors declare that there is no conflict of interest.

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