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Shear Bond Strength of MDP-Containing Adhesives with and without HEMA: An in Vitro Study

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ABSTRACT

Background and Objectives: The primary role of dental adhesives is to deliver a good bond between the filling material and the tooth. Universal adhesive (UA) systems brought a novel concept by adding active resin monomers that facilitate both micromechanical and chemical adhesion. 10-methacryloyloxydecyl dihydrogen phosphate (MDP) is a vital component of these adhesives, noted for its strong ionic interaction with hydroxyapatite (HAp) in both enamel and dentin, which leads to the formation of nanolayers of MDP-calcium salts. Evidence suggests that 2-hydroxyethyl methacrylate (HEMA), a monomer that is hydrophilic and improves the adhesive's wetting ability on dental surfaces, may affect MDP's interaction with hydroxyapatite powder. In view of this, the present study aimed to assess the differences in shear value of bond strength between two MDP-containing multi-mode bonding systems: Futurabond M+ (VOCO, Germany), which contains HEMA, and G-Premio Bond (GC, Japan), a HEMA-free alternative.

Materials and Methods: Forty extracted human maxillary premolars were separated into two groups (n=20). The occlusal portion was removed to gain a smooth dentin surface. Adhesives were applied per manufacturers' instructions, followed by composite resin build-up (Palfique LX5, Tokuyama, Japan). After 24-hour water storage at 37°C, using a universal testing machine (TERCO, MT 3037, Sweden) at 1.0 mm/min, shear bond strength was tested. To analyze the data, an independent t-test ($p < 0.01$) was used.

Results: Futurabond M+ (HEMA-containing) exhibited significantly higher shear bond strength (19.74 ± 4.80 MPa) than G-Premio Bond (HEMA-free) (14.33 ± 5.80 MPa) ($p = 0.001$). The mean difference (5.41 MPa) and large effect size (Cohen's d = 1.017) show that the greater bond strength attained with VOCO Futurabond M+ is statistically important and expressive in a practical, clinical situation.

Conclusion: HEMA-containing Futurabond M+ produced higher shear bond strength to dentin compared to HEMA-free G-Premio Bond, probably due to HEMA, ethanol, and higher pH. The adhesive act is dependent on numerous factors; therefore, additional investigations are necessary.

Keywords: HEMA, Shear bond strength, Universal adhesives, MDP.

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INTRODUCTION

With the advent of aesthetic composite materials, a breakthrough in evolving dental adhesive technologies has been landmarked. Establishing a strong bond to the tooth substrate is crucial before placing these esthetic restorative materials.[1] Durable and visually pleasing restorative materials like resin-based composites used with strong enamel-dentin adhesive play a key role in contemporary dentistry. The performance of the restorative material in the clinic can be enhanced by formulation modifications, the incorporation of novel, formulation-tailored fillers (such as composite filler clusters or nano-fillers), or by refining the bonding characteristics of the multi-mode adhesives used.[2]

The introduction of universal adhesive (UA) systems in 2011[3-5] offered an exceptional viewpoint within this area of study. The incorporation of functional resin monomers in bonding formulations allows chemical adhesion, making these agents applicable in both direct and indirect restorations to bond to various substrates, including enamel, dentin, and ceramic.[6, 7] MDP (10-methacryloyloxydecyl dihydrogen phosphate) stands out as the main component used in these adhesive systems. It is very effective because it reacts ionically with hydroxyapatite in enamel and dentin, forming stable calcium-MDP nanolayers on the surface. These nanolayers strengthen the chemical bond with the tooth surface and help protect the hybrid layer from hydrolysis.[8] Still, doubts remain about the bonding effectiveness of MDP-containing adhesives.[9] Research suggests that 2-hydroxyethyl methacrylate (HEMA) may influence the interaction between MDP and HAp powder.[10] As a hydrophilic monomer, HEMA improves the wetting ability of bonding systems on tooth surfaces. This feature promotes adhesion by increasing the diffusion and mixing of adhesive components, supporting the formation of the hybrid layer,[11] and reducing phase separation.[12]

However, HEMA also accelerates the development of an unsteady water-based gel that is vulnerable to breakdown due to hydrolysis.[13, 14] An additional disadvantage of using HEMA in universal adhesives (UAs) is its undesirable interface with MDP, which significantly decreases the HAp demineralization. As a result, fewer MDP-Ca salts are formed, and the deposition of nano-layers, which is critical for strong chemical bonding with dentin, is partially inhibited.[15]

Given these findings, the current study was designed to assess the shear bond strength between two, MDP-containing nano-filled universal adhesives: Futurabond M+ (VOCO, Germany), which includes HEMA, and G-Premio Bond (GC, Tokyo, Japan), which does not contain HEMA, as shear bond strength is the most commonly used lab measure for evaluating the performance of dentin bonding agents.[16] It was hypothesized that the HEMA-free adhesive would demonstrate better shear bond strength to dentin, owing to reduced water absorption and enhanced chemical interaction between MDP and hydroxyapatite.

METHODS

Preparation and grouping of the specimens

For this study, forty sound maxillary premolars were collected from healthy individuals aged 12 to 18 years who had undergone extractions for orthodontic purposes. All selected teeth were free of caries and had intact crowns. The research protocol received approval from the Scientific Ethical Review Board at the Dental College of Hawler Medical University (Reference No. HMUD-2425122). The teeth were then inspected with a stereoscopic microscope at 40× magnification. Each sample was vertically embedded in a cold-polymerizing acrylic resin via a surveyor and a prefabricated PVC house pipe that was 1 cm wide by 2 cm high. Each tooth was positioned inside the tube in a way that its long axis stood upright, creating a right angle with the base, guaranteeing correct position during the cutting procedure. This arrangement ensured that the dentin surface was perfectly aligned and ran parallel to the base of the supporting block. Each specimen's occlusal surface was cut along the mesiodistal plane using a diamond bur (NTI, Kahla GmbH, Germany) to uncover the underlying smooth dentin surface. To avoid heat generation, a continuous water spray was applied for cooling. After the initial cut, silicone paper discs were used to smooth the exposed dentin surface for approximately 20 seconds to simulate a clinically relevant smear layer.[17,38] Although both adhesives used in this study are classified as multi-mode systems, they were applied through a self-etch approach, without etching by 37% acid gel. This approach maintained the smear layer and ensured consistent surface conditions across all samples.

Following that, two groups of 20 specimens each were formed by randomly and blindly assigning the teeth (Figure 1) using a computer-generated list to ensure equal and unbiased group allocation. According to the dentin bonding agent applied, Group A received Futurabond M+ (VOCO), a HEMA-containing adhesive, and Group B received G-Premio Bond (GC), a HEMA-free adhesive, as shown in Table 1.

Table 1. Adhesives tested in this research

Group name	Brand name	Manufacturer	pH	Composition
Group A	Futurabond M+	(VOCO, Cuxhven, Germany)	2.3	HEMA, 10-MDP Ethanol/Water
Group B	G-Premio Bond	(GC, Tokyo, Japan)	1.5	10-MDP, 4-MET, MDTP, GDMA, Acetone/Water

Table 2. Adhesive application instructions according to the manufacturers

Group A: Futurabond M+ (VOCO)	Group B: G-Premio Bond (GC)
• After the surface was prepared, it was cleaned using water and with a gentle stream of air, dried off. • A drop of Futurabond M+ was placed on a mixing pad. • Using a disposable, flexible micro-brush with a superfine 1.5 mm tip, the adhesive was carefully applied and rubbed for 20 seconds on the prepared surface. • The adhesive coating was dried off for 5 seconds with dry, oil-free air to eliminate any solvents. • The adhesive was cured with light cure for 10 seconds.	• A disposable, flexible micro-brush with a 1.5 mm superfine tip was used to apply the adhesive to the prepared surface. • The adhesive remained on the prepared area for 10 seconds. • The adhesive was completely dried with maximum air pressure for 5 seconds. • The adhesive was cured for 10 seconds via light cure.

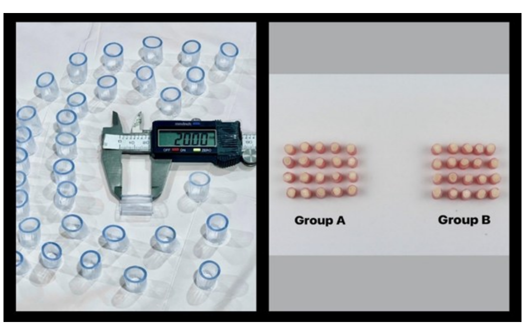

Figure 1. Steps of preparation of the samples and grouping.

Application of bonding agents

Following the manufacturer's directions (which are shown in Table 2), the adhesives were used on every specimen in its respective group (Figure 2).

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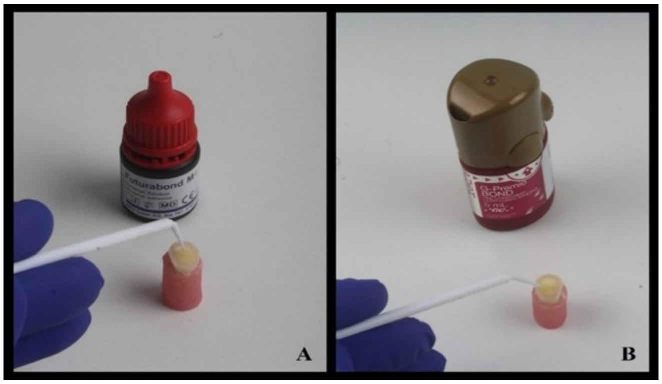

Figure 2. Bonding application, Futurabond M+ (A), G-Premio Bond (B).

Composite Resin Build-up

Following securing the acrylic mold on a bench vice, using an elastic mold measuring 2 x 2 mm, composite (Palfique LX5, Tokuyama, Japan) cylinders were placed on the bonded surfaces and light-cured (Figure 3 A, B, C, and D). The dimensions of every sample were rechecked using a digital vernier.

The light-curing unit's intensity was checked with a radiometer (Hilux Curing Light Meter, Benlioglu Dental Inc.) before and after each light-cure polymerization step to guarantee precise light intensity.17 All specimens at each step of bonding and composite resin build-up were light-cured using the 3M™ Elipar™ DeepCure-L LED Curing Light, which had a light intensity of 1,470 mW/cm<2, and a light guide with a 10 mm diameter. Light source's distance from the surface was 1mm.37

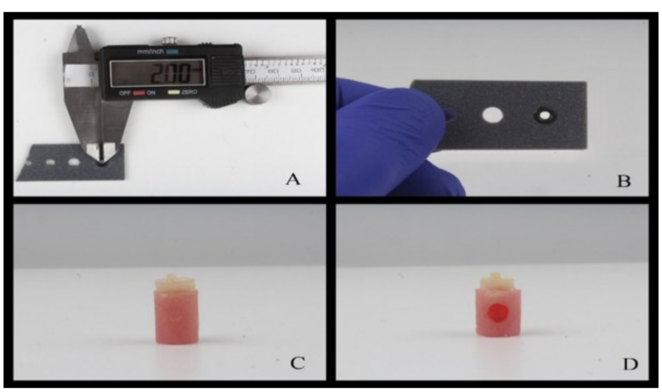

Figure 3. Composite placement on the bonded surface with the aid of the elastic mold (Elastic mold measured by digital Caliber 2mm by 2mm A and B, composite cylinder sample of each group C and D).

Shear Bond Strength Test

For twenty-four hours, the prepared specimens were kept at a regulated temperature of 37°C in a distilled water bath (Figure 4). This storage period only represents short-term bond strength and is a limitation of the present study.

Including long-term aging methods, such as thermocycling or extended water storage, would have improved the clinical relevance of the findings. Afterward, each specimen was secured to the testing apparatus, and a 12 mm diameter adapter was used to compensate for the uneven surface of the 1 cm prefabricated PVC pipe used as the cylinder mold. Shear force was exerted on every specimen under compression mode with a universal testing machine (UTM) (TERCO, MT 3037, Sweden) at a 1.0 mm/min crosshead speed (Figure 5)18 A knife-edge shearing blade (0.5 mm wide)34 was positioned perpendicular to the composite–dentin interface because the bonded composite cylinder was oriented horizontally. Samples were loaded until failure occurred. Shear bond strength was determined by dividing the force applied (in Newtons) by the cross-sectional area (in mm²), and expressed in megapascals (MPa)33 The test was conducted in the Mechanical Engineering Department, Faculty of Engineering, Salahaddin University, Erbil.

Figure 4. The prepared samples were stored in distilled water at 37°C for 24 hours

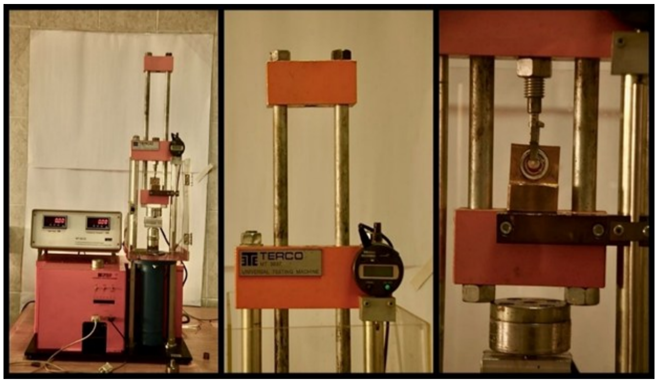

Figure 5. Universal Testing machine (TERCO, MT 3037, Sweden), one sample under the test (right photo)

RESULTS

(Table 3) showed the Shapiro-Wilk test at a significance level of $p < 0.01$, neither of the adhesive groups demonstrated a significant departure from a normal distribution. (Group A) Futurabond M+ had a p-value of 0.020, and (Group B) G-Premio Bond had a p-value of 0.101. Although the (Group A) value is below 0.05, it remains above the stricter 0.01 threshold. Therefore, the null hypothesis of normal distribution is not rejected for either group. This indicates that the shear bond strength data for both adhesives are approximately normally distributed, and the use of parametric tests such as the independent samples t-test is statistically appropriate.

Table 3. Tests of Normality for Shear Bond Strength (MPa)

Group	Kolmogorov-Smirnov Statistic	df	Sig.	Shapiro-Wilk Statistic	df	p-value
(Group A) Futurabond M+	0.203	20	0.030	0.883	20	0.020
(Group B) G-Premio Bond	0.158	20	0.200	0.920	20	0.101

(Table 4) showed that the statistical analysis revealed a notable difference in shear bond strength between the two adhesives tested: Futurabond M+ and G-Premio Bond. Futurabond M+, a HEMA-containing adhesive, had a higher average bond strength of 19.74 ± 4.80 MPa, while G-Premio Bond, which is HEMA-free, showed a lower mean value of 14.33 ± 5.80 MPa. The mean difference between the two groups was 5.41 MPa. An independent samples t-test was conducted to assess the significance of this difference, resulting in a t-value of 3.315 and a p-value of 0.001. Since the p-value is below the 0.01 threshold, the result is considered highly statistically significant. However, failure mode analysis (e.g., adhesive, cohesive, or mixed) was not performed in this study. Due to time constraints, we couldn't conduct this evaluation, but it is recommended for future research to improve understanding of bond strength results.

Table 4. Comparison of Shear Bond Strength Between VOCO Futura bond M+ and GC G- Premio Bond

Group	N	Mean ± SD	Mean Difference	t-test	p-value
(Group A) Futurabond M+	20	19.74 ± 4.80	5.41	3.315	0.001
(Group B) G-Premio Bond	20	14.33± 5.80			

**Significant at level ($p<0.01$)

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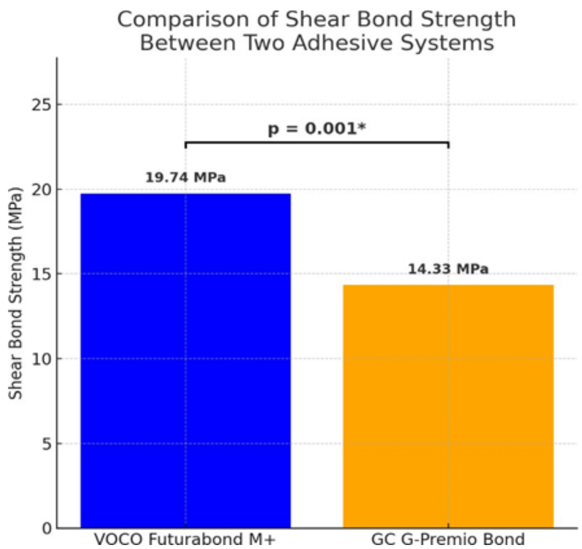

The effect size analysis (Table 5) demonstrated a large and clinically significant difference in shear bond strength between VOCO Futurabond M+ and GC G-Premio Bond.

The calculated values—Cohen's d = 1.017, Hedges' g = 0.996, and Glass's delta = 0.933—all surpassed the 0.8 benchmark for a large effect. Moreover, the 95% confidence intervals for each metric excluded zero, confirming statistical reliability: Cohen's d [0.350, 1.671], Hedges' g [0.343, 1.638], and Glass's delta [0.237, 1.610]. Collectively, these results indicate that the superior bond strength of VOCO Futurabond M+ is both statistically significant and clinically meaningful.

Table 5. Independent Samples Effect Sizes

Effect Size Type	Standardizer	Point Estimate	95% CI (Lower)	95% CI (Upper)
Cohen's d	5.32234	1.017	0.350	1.671
Hedges' g	5.43035	0.996	0.343	1.638
Glass's delta	5.79637	0.933	0.237	1.610

DISCUSSION

The success of composite restorations largely depends on the shear bond strength established at the adhesive interface. weak bonding systems remain to pose a major problem in restorative and prosthetic dentistry, often leading to restoration failures, such as marginal staining, secondary caries, and eventual debonding.[19]

Dentin bonding systems were planned to prevent such failures by permitting strong adhesion between tooth structure and resin composites. Over time, these bondings have minimized the necessity for aggressive tooth reduction, permitting conservative cavity designs, less dependence on mechanical retention, and better preservation of supporting enamel .[20]

Several variables can affect shear bond strength outcomes; these include specimen-related factors (such as dentin depth, storage medium, and age), technical factors (test design, loading rate, and cross-sectional bonding area), and material-related parameters. Manufacturers have constantly wanted to simplify bonding procedures while improving their efficiency. Yet, this simplification has not always resulted in improved performance. Notably, the seventh generation of adhesives, though intended to streamline the clinical protocol, often exhibited reduced dentin bond strength and increased microleakage.[21]

To address such limitations, universal (or multimode) adhesives were introduced. These systems can be used with etch-and-rinse (ER), self-etch (SE), and selective-etch techniques. Also, they can bond to a wide range of substrates (enamel, dentin, ceramics, and metals) in both direct and indirect procedures. Universal adhesive formulations normally include monomers like 10-MDP, which is widely used for its strong attraction toward hydroxyapatite and capacity to create durable adhesion.[22]

It attains this durable bonding by creating stable calcium salt nanolayers with hydroxyapatite, contributing to both chemical and micromechanical retention.[23] 10-MDP's concentration differs across different adhesives, and if its presence is higher, it will give a better-quality bond strength.[10]

Many simplified adhesives include 2-hydroxyethyl methacrylate (HEMA) in their formulation, which is a hydrophilic monomer that enhances wettability and facilitates adhesive penetration into the exposed collagen network. HEMA also acts as a solvent-like agent, stabilizing the formulation by lowering phase separation between hydrophilic and hydrophobic components.[11] Still, its high-water attraction poses a challenge, as it can increase water sorption and, over time, trigger hydrolytic weakening of the adhesive interface.[24]

In adhesives containing 10-MDP, HEMA might affect the chemical interaction between 10-MDP and calcium, possibly inhibiting MDP-Ca salt formation.[25] As a solution, HEMA-free adhesives were developed.[35] But, their absence may result in phase separation,[26, 27] causing water droplets to form inside the polymerized adhesive; a condition described as water-tree nanoleakage. This happens because residual water in the dentin tubules can seep in, potentially weakening the adhesive layer.[26, 28]

HEMA's effect on clinical outcomes remains a topic of debate. While some studies suggest there's no significant difference between adhesives that contain HEMA and those that are HEMA-free, others have shown that the presence and absence of HEMA can lead to different clinical outcomes.[24]

In this study, a statistically significant difference in shear bond strength was observed between the two universal adhesives tested. Unlike the hypothesis, Futurabond M+, which contains HEMA and uses ethanol as a solvent, attained a higher mean shear bond strength. At the same time, the HEMA-free, acetone-based G-Premio Bond recorded a lower mean value. The mean difference, and the result was very significant, supporting that the enhanced performance of Futurabond M+ is not only statistically meaningful but also of practical importance. These results support the idea that HEMA improves adhesive penetration, particularly into moist dentin, and enables better micromechanical interlocking.

Multiple investigations have confirmed that the formulation chemistry of an adhesive system greatly impacts its clinical efficiency.[29] The effect of the adhesive solvent must also be considered. Ethanol, used in Futurabond M+, is known to solidify demineralized collagen and preserve interfibrillar spaces during solvent vanishing.[17] It promotes effective monomer infiltration and improves bonding in hydrophilic conditions.[30] Conversely, acetone, used in G-Premio Bond, due to its water-chasing effect, needs a moist bonding approach to prevent breakdown of the exposed collagen network.[31] Excess water or evaporation mismanagement can compromise bond strength, as acetone is less forgiving in terms of working time and moisture sensitivity.[32] These differences in solvent systems might explain the superior performance of the ethanol-based adhesive in this study.

pH is another aspect of bonding performance. Universal adhesives are naturally classified as ultra-mild (pH > 2.5), mild (pH ~2), or intermediately strong (pH 1–2).7 Authors assume that milder adhesives preserve more hydroxyapatite, enabling stronger chemical bonds with MDP.[36] Futurabond M+ (pH 2.3) is classified as mild, while G-Premio Bond (pH 1.5) is intermediately strong. This difference may somewhat explain the variance in bond strength detected in this study.

Supporting this study's findings, a 2022 study by Pimentel de Oliveira et al. compared self-etch adhesives with varying HEMA and 10-MDP content.[36] Their outcomes showed the highest bond strength in the group containing both HEMA and 10-MDP, assigning the strength primarily to the presence of 10-MDP. This further highlights that while HEMA might play a helpful role in improving handling and penetration, the chemical interaction between MDP and hydroxyapatite remains the foundation of strong adhesion.

The clinical argument around HEMA continues. While its removal helps reduce hydrolytic degradation, it may also compromise adhesive stability due to increased phase separation. On the other hand, the presence of HEMA improves handling and early bond strength but might hinder long-term durability. In view of these variables, adhe-

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sive performance depends on a combination of several interrelating factors, including monomer type, solvent system, acidity, and formulation design. Further in vitro and long-term clinical studies are needed to clarify these interactions in more detail and optimize adhesive design.

CONCLUSION

This study found that the HEMA-containing Futurabond M+ revealed significantly higher shear bond strength to dentin than the HEMA-free G-Premio Bond. The presence of HEMA, along with ethanol as a solvent, and a less acidic pH appears to enhance adhesive performance. Since bonding outcomes are influenced by several interacting parameters, additional long-term laboratory and clinical research is recommended to validate these results.

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CONFLICT OF INTEREST

The author(s) declared no conflicts of interest.

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