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Proceeding Paper

Bond Strength Properties of a Dental Adhesive with a Novel Dendrimer—G-IEMA †

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Abstract: The objectives of this study were to characterize the microtensile bond strength to enamel of two experimental adhesive systems, one containing a novel monomer and the other having the same composition as commercial adhesive systems, and comparing them to commercial materials. Two experimental adhesive systems were developed in the lab, one with Bisphenol A diglycidyl methacrylate (Bis-GMA) and the other with G(2)-isocyanatoethyl methacrylate (G-IEMA) as a substitute for Bis-GMA. Twenty healthy human permanent molars were cut into halves and randomly divided into eight groups based on the application mode. The experimental universal adhesive system without Bis-GMA demonstrated comparable adhesive strength to enamel as the other universal adhesive systems containing Bis-GMA.

Keywords: dendrimer; experimental monomer; experimental dental adhesive; G-IEMA

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1. Introduction

The major paradigm shift in adhesion to tooth structure occurred in 1955 with the introduction of phosphoric acid etching to enamel by Buonocore. Since then, the evolution of adhesive techniques has allowed dentists to adopt a minimally invasive philosophy in clinical practice [1,2]. Regarding material choice, currently, there is a wide offering of adhesive systems available for bonding to tooth tissues, using different adhesive strategies [3]. To simplify the use of these materials, universal adhesive (UA) systems were introduced, allowing clinicians to choose the best adhesive strategy according to different clinical scenarios, whether it is an etch-and-rinse, self-etch or selective enamel etching approach [4–6].

Replacing Bis-GMA while still improving the physicochemical and mechanical properties of adhesives has been researched in recent studies [7,8]. Specifically, some authors have examined the introduction of dendrimers as base constituents UAs. A second-generation dendrimer derived from isocyanatoethyl methacrylate, G-IEMA, has been investigated as a candidate for potential replacement of Bis-GMA-based systems [9,10]. Traditional linear crosslinking monomers can be replaced successfully by dendrimer G-IEMA without influencing the resulting properties. Not only did this monomer significantly improve the experimental UA's degree of conversion, but it was also responsible for reducing the co-polymer shrinkage and controlling water sorption [11]. Further to this, the same authors also observed that G-IEMA formulations could increase the bond strength to dentin, and later on, showed that they have promising interfacial properties [11,12].

However, to research and prove the beneficial applicability of G-IEMA, further studies are needed. It is therefore important to investigate the role of G-IEMA-based systems on the bond strength to enamel using two different adhesive strategies, while also evaluating their impact on the contact angle of enamel surfaces, which is as of yet unknown.

2. Materials and Methods

Two experimental adhesive systems, one with Bis-GMA (EM1) and another with G-IEMA, as a substitute for Bis-GMA (EM2), were developed in our lab. Two commercial adhesives, Futurabond® M+ (VOCO) (FUT) and ScotchbondTM Universal (3M ESPE) (SBU) were chosen as controls. Twenty healthy human permanent molars, obtained with informed consent (approved by the Ethics Committee of Egas Moniz School of Health & Science), were cut into halves and randomly divided into eight groups (n = 5) according to the application mode (self-etch or etch-and-rinse): FUT_ER, FUT_SE, SBU_ER, SBU_SE, EM1_ER, EM1_SE, EM2_ER, and EM2_SE. Afterwards, each specimen was polished with a 600 SiC grit paper for smear layer simulation and the adhesives were applied according manufacturer's directions. The etch-and-rinse method employed Octacid orthophosphoric acid (37%) (Clarben). Resin build-ups were conducted using the Schmidt Composite Nanohybrid (MADESPA), with increments shaped as rectangular prisms. The resin build-ups were applied in 2 mm increments, achieving a total height of 6 mm. The materials were light-cured using the EliparTM DeepCure-S (3M ESPE) system, following the manufacturer's instructions, with a 40 s cure time specifically applied to the experimental universal adhesive systems. This system employs blue LEDs for the light-curing process. Its peak irradiance was 1200 mW/cm^2 which was confirmed with a radiometer. After processing, the specimens were kept in distilled water for 24 h at 37 °C. Beams (1 ± 0.2 mm^2) were obtained through additional sectioning and tested using a universal testing machine (μTBS) with a cross head speed of 0.5 mm/min until failure. The data analysis was performed using SPSS (version 28.0, IBM Corp., Armonk, NY, USA) with linear mixed models (LMMs) incorporating fixed effects, while maintaining a significance level of 5%.

3. Results

The effects of adhesive ($p = 0.033$) and method, or protocol, ($p < 0.001$) on the microtensile bond strength were significant and independent of one another. There was no interaction between the adhesive used and the technique adopted ($p = 0.985$) (Table 1). Independent of application procedure, SBU displayed a considerably greater μTBS than the experimental EM2 ($p = 0.031$). No variations were found between any other adhesive pairings.

Table 1. Linear mixed model analysis—Type III Tests of Fixed Effects considering the variables: (1) adhesive and (2) adhesive strategy or protocol.

	Type III Tests of Fixed Effects [a]			
Source	**Numerator df**	**Denominator df**	**F**	**Sig.**
Intercept	1	426	531.262	0.000
Adhesive	3	426.000	2.947	0.033
Strategy	1	426	79.606	0.000
Adhesive * strategy [b]	3	426.000	0.050	0.985

[a] Dependent variable: microtensile bond strength (MPa). [b] In the "Adhesive * strategy" term above, the asterisk (*) denotes the interaction between main factors Adhesive and strategy.

4. Discussion

Ongoing concerns about the use of biocompatible dental materials have called into question the incorporation of Bis-GMA in resin-based restorative materials, due to its Bisphenol A (BPA) content, which can elute and have systemic health implications [10]. The demand for new materials without Bis-GMA has emerged as a preventive measure to reduce exposure to Bisphenol A [13]. The same adhesive systems who formulated and subsequently evaluated the physicochemical properties and adhesion to dentin experimental universal adhesive systems without Bis-GMA, patented in Portugal (holder: Egas Moniz School of Health & Science). This study followed the same line of research, assessing the adhesion to enamel, which has not been studied until now [11,12].

According to the current scientific evidence, for enamel, the best adhesive strategy continues to be the use of orthophosphoric acid prior to the application of the adhesive system [13,14]. Several in vitro studies demonstrated that universal adhesive systems have higher values of adhesive resistance to enamel when used according to the etch-and-rinse (versus self-etch) protocol, as was observed in this study [5,15]. These results are justified by the reduced demineralization capacity of a universal adhesive system compared to orthophosphoric acid, resulting in an incomplete creation of microporosities, which inevitably reduces the micro-retention of the adhesive [2]. The chemical composition of the adhesive systems can also contribute to the differences observed in this study because, although the experimental adhesive systems were formulated according to the chemical composition of the commercial adhesives, the specific percentage of each component was not discriminated.

In this study, most of the adhesive systems used had a mild pH (Futurabond® M + and experimental adhesives), with Scotchbond™ Universal registering a more basic pH, falling within the ultra-mild category. Although a good adhesive behavior to dentin is associated with mild self-etching (pH $\cong$ 2), these solutions are unable to effectively condition the enamel, leading to increased microleakage; a mild pH is essential prior to etching dentin to obtain a micromechanical retention effective [16,17]. The pH of a universal adhesive system is a relatively important property because, while an acidic medium is required for the dissolution of the smear layer and smear plugs (opening the dentinal tubules), an excessively acidic adhesive system can remove excess calcium, decreasing its ability to adhere to 10-MDP, which becomes particularly important in adhering to dentin [16,17]. It is essential to select adhesive systems that contain 10-MDP, taking into account their molecular structure, their hydrophobic behavior and the characteristics of the adhesive interface that favor adhesion [18]. The microtensile bond strength to dentin using the same adhesive systems and protocols as the present study, there were no significant differences between the adhesive systems studied, suggesting that the experimental universal adhesive system without Bis-GMA could be used effectively in dentin [12].

5. Conclusions

The four universal adhesive systems examined (Futurabond® M+, ScotchbondTM Universal, an experimental universal adhesive system with Bis-GMA, and an experimental universal adhesive system without Bis-GMA) showed no statistically significant differences in adhesive strength to enamel when using either the etch-and-rinse or self-etch adhesive strategies. The experimental universal adhesive system without Bis-GMA exhibited comparable adhesive strength to enamel as the other universal adhesive systems containing Bis-GMA. The promising behavior of the experimental Bis-GMA-free universal adhesive system indicates the need for further investigations. These studies should focus on exploring the potential of the G-IEMA dendrimer as a substitute for Bis-GMA in the composition of adhesive systems.

6. Patents

This work resulted in a national patent, registered under No. 115,064—Formulation for a universal dental adhesive system containing a second-generation dendritic cross-linking monomer (2019); Vasconcelos e Cruz, J., Gonçalves, L. L. and Polido, M., Moniz School of Health & Science.

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Conflicts of Interest: The authors declare no conflict of interest.

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