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Original article

EVALUATION OF THE BOND STRENGTH BETWEEN BIOACTIVE RESTORATIVE SYSTEMS AND PRIMARY TEETH DENTIN: AN IN VITRO STUDY

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ABSTRACT

Purpose: Comparative evaluation of the bond strength between four bioactive restorative materials and the dentin of primary teeth.

Materials and Methods: The study included 120 extracted primary teeth, divided into four groups according to the restorative material used. The tested materials were the giomer Beautifil II with the adhesive system Clearfil SE Protect, the compomer Dyract with the adhesive system Evetric, the nanofilled composite Estelite Sigma Quick with the adhesive system Bond Force II, and the conventional glass ionomer cement Fuji IX GP. After the preparation of the samples they were subjected to thermocycling. Prismatic sticks (approx. 2×2×20 mm) were sectioned from each sample and tested for microtensile bond strength (ìTBS) at a crosshead speed of 1 mm/min. The results were statistically analyzed using one-way ANOVA and post hoc Tukey test ($p < 0.05$).

Results: The giomer Beautifil II and the compomer Dyract showed the highest mean values of the adhesive bond strength—5.59 MPa and 5.29 MPa, respectively—while the lowest values were recorded for the conventional GIC Fuji IX GP (2.61 MPa). Beautifil II showed statistically significant higher values compared to all the other materials (Fuji IX GP, Estelite Sigma Quick, and Dyract XP). No statistically significant differences were found between Dyract XP and Estelite ($p = 0.899$).

Conclusion: The highest bond strength was observed with Beautifil II + Clearfil SE Protect, followed by Dyract + Evetric, ESQ + Bond Force II, and Fuji IX GP. The superior performance is achieved by the combination of a bioactive restorative material and a two-step self-etch adhesive system, which provides both chemical and micromechanical retention, due to the incorporation of 10-MDP monomer in its structure.

Keywords: Primary teeth, dentin bonding, bioactive restorative materials, giomer, compomer, glass ionomer cement,

INTRODUCTION

Maintaining a healthy primary dentition that can optimally perform its functions is a prerequisite for the normal psychological and physical development of the child and plays a significant role in their quality of life. Primary and permanent teeth have a similar structure and chemical composition, which makes the mechanism and pathophysiology of carious lesions identical in both dentitions [1,2]. However, anatomical and physiological differences exist between the two dentitions, leading to certain variations in the development of carious lesions [2,3,4]. The enamel of primary teeth is thinner—1.14 mm [1,3,4,5]—compared to that of permanent teeth—2.58 mm [4,6]. In primary teeth, the enamel is of equal thickness in the cusp and cervical areas [3] and has greater porosity and permeability [7]. The proportion of interprismatic enamel (the more soluble fraction) is higher in the primary dentition; enamel prisms are smaller and the arrangement of crystals is more irregular [1,5]. These structural differences, together with lower mineral content [3,4,5] and higher levels of carbonate ions, which further increase its solubility [1,4,6], make the enamel of primary teeth more permeable and more susceptible to the development and progression of carious lesions than the enamel of permanent teeth [1]. The dentin of primary teeth is also thinner. Similar to enamel, it has a lower degree of mineralization [3] and is characterized by faster and more extensive involvement as carious lesions progress [3,5]. The inherently thinner dental structures lead to greater exposure of the pulp chamber in the primary dentition, which appears relatively larger compared to the rest of the tooth and is closer to the tooth surface [1]. These specific characteristics of primary teeth favor the more rapid progression to complicated lesions compared to permanent teeth [3]. Carious lesions in the primary dentition progress rapidly, often affecting large portions of the tooth crown within a short period of time [8]. This is due to a combination of potent etiological factors such as high intake of sticky carbohydrate-rich foods, unstable oral hygiene habits in children, the structural characteristics of primary teeth, and changes in the protective functions of the pulp. The specific features of primary teeth influence both the shape and size of cavity preparations as well as the choice of restorative material. Ensuring the long-term survival of restorations in the primary dentition is important not only for the patient's health but also from an economic standpoint [9]. The restoration of primary teeth aims to preserve the tooth in the dental arch until the time of its physiological exfoliation. Therefore, restorations should ideally last until tooth shedding. Restoring the tooth can make it more vulnerable to the development of new carious lesions, which is related to the cavity preparation, the properties of the restorative material, the adhesive system and the application protocol, as well as the polishing and finishing of the final restoration [9,10]. The accumulation of biofilm on the restoration facilitates the material wear, leads to degradation, surface roughening, and reduced microhardness, which in turn promotes further biofilm accumulation. As a result, conditions are created for colonization at the adhesive interface between the restorative material and tooth structure, leading to the development of secondary (recurrent) caries lesions [11]. Good marginal sealing is essential for the longevity of restorations, while its absence is a risk factor for the occurrence of secondary caries. Marginal sealing is primarily ensured by the adhesive systems; therefore, their quality and composition directly influence the clinical durability of restorations [12].

Traditionally, the approach to treating carious lesions has involved the use of bioinert materials, which replace lost hard dental tissues without eliciting a biological response. In recent years, there has been a shift in philosophy regarding the requirements that restorative materials must fulfill. Scientific attention is now focused on the development of bioactive materials that induce a biological response at the restorative material-hard tissue interface. These materials interact with the oral environment through the release of fluoride, calcium, and phosphate ions. These ions influence acidity levels, affect bacterial metabolism, and facilitate the remineralization of hard dental tissues through the formation of hydroxyapatite and fluorapatite [13] Our focus is on evaluating the bond strength achieved when using representatives of the main classes of bioactive restorative materials: the compomer Dyract, the giomer Beautifil II, and the conventional glass ionomer cement (GIC) Fuji IX GP, along with one composite material, Estelite Sigma Quick. These were combined with two one-step self-etch adhesives (Evetric, Bond Force II) and one two-step self-etch adhesive (Clearfil SE Protect).

MATERIALS AND METHODS

Experimental Samples

The study used intact, extracted primary teeth. The teeth were collected from healthy children aged between 4 and 9 years, following signed informed consent from the parents for the use of these teeth in the experiment. The primary teeth were extracted either due to physiological exfoliation or for orthodontic reasons. After extraction, the teeth were placed in a 10% formalin solution for 10 minutes, and subsequently stored in physiological saline until the time of the specific experimental procedure.

The distribution of the experimental samples is presented in Table 1.

Table 1. Distribution of the specimens into groups according to the type of composite system used.

Group	Number of samples	Restorative material and adhesive system
Group 1	N=30	Conventional GIC Fuji IX GP
Group 2	N=30	Compomer Dyract XP and Evetric, seventh-generation self-etch adhesive, without fluoride
Group 3	N=30	Giomer Beautifil II and ClearFil SE Protect, sixth-generation two-step self-etch adhesive system , containing fluoride
Group 4	N=30	Estelite Sigma Quick and Bond Force II, seventh-generation self-etch adhesive, containing fluoride

Tooth Surface Preparation

All teeth were prepared to create a flat dentin surface. This was achieved by removing the enamel with a high-speed turbine bur under water-air cooling, followed by polishing with abrasive discs. Using a round turbine bur (ISO 806 314 001534 012) and a high-speed handpiece with water–air cooling (Tiger 500 Bio Series High-Speed Handpiece), a cut was made in a mesio-distal direction along the central occlusal fissure. With a turbine diamond fissure bur (ISO 806 204 108524 835010) and a high-speed handpiece with water–air cooling, a cut parallel to the occlusal surface was made at the controlled depth determined by the initial cut with the round bur, in order to remove the occlusal enamel and parts of the dentin. The resulting surface was then leveled using an abrasive disc, replaced for each specimen.

Restorative Procedures

Each restorative material and adhesive system were applied to the prepared smooth dentin surface according to the manufacturer's instructions.

Preparation of the samples for the Microtensile Bond Strength Test

After 24 hours of storage in deionized water at 37°C, the restored teeth underwent thermocycling (1500 cycles between 5°C and 55°C; Thermocycler SD Mechatronik). They were then embedded in acrylic resin and sectioned mesiodistally with a diamond disk to obtain prismatic specimens measuring approximately 2 × 2 × 20 mm. Only beams containing a clearly distinguishable interface between dentin, adhesive, and restorative material were used for the evaluation of the bond stregth.

Evaluation of bond strength using the microtensile test

For the ìTBS test, the restored teeth specimens were embedded in self-curing transparent orthodontic acrylic resin and oriented with the restoration facing upwards. After complete polymerization, each specimen was sectioned in two perpendicular directions (longitudinal and transverse) to obtain prismatic sticks with dimensions of approximately 2 × 2 × 20 mm. Only sticks with cross-sections entirely comprising dentin–adhesive–composite interfaces were selected for testing, thereby ensuring the correct perpendicular alignment of the adhesive bond relative to the applied force. The stick-shaped micro-specimens were fixed to the testing device plates (LMT-100) using cyanoacrylate adhesive, with their longitudinal axis aligned with the loading direction. Each specimen consisted of dentin, an adhesive layer, and composite material. The microtensile bond strength (ìTBS) test was performed at a crosshead speed of 1 mm/min until specimen failure. The maximum failure load was recorded in MPa.

Statistical Analysis

Data analysis was performed using SPSS software. One-way ANOVA was used for group comparisons, followed by post hoc analysis (Tukey HSD). Values with $p < 0.05$ were considered statistically significant.

Table 2. Mean values and variability of ìTBS for the tested materials (Groups 1–4).

	n	Mean	Std	Min	25%	50%	75%	Max
Group 1 Fuji IX GP	9	2.61	0.9	1.4	2.3	2.6	2.8	4.4
Group 2Dyract XP	11	5.29	0.98	3.8	4.7	5.2	5.75	7.4
Group 3Beautifil II	21	5.59	3.63	1.6	3.2	4.7	5.9	16
Group 4Estelite Sigma Quick	11	2.97	1.48	1	1.9	3	3.9	5.7

RESULTS

In the present study, the microtensile bond strength of the adhesive interface between four restorative systems and the dentin of primary teeth was evaluated. The results were subjected to statistical analysis.

The highest mean bond strength values were observed in specimens from Group 3 (restorative material Beautifil II and adhesive system Clearfil SE Protect) – 5.59 MPa, followed by Group 2 (Dyract XP with adhesive system Evetric) – 5.29 MPa, Group 4 (ESQ with adhesive system Bond Force II) and Group 1 (Fuji IX GP), with 2.97 and 2.61 MPa, respectively (Table 2).

For comparison of the mean bond strength values among the different groups, a post-hoc Tukey HSD analysis was applied. The results are presented in Table 3.

Table 3. Comparative analysis of the mean bond strength among the different groups (Post-hoc analysis Tukey HSD).

Comparison	**Difference**	**p-value**	**Significance**
Group 3 vs Group 2 (Beautifil II vs Dyract XP)	4.87	0.001	p < 0.05
Group 3 vs Group 4 (Beautifil II vs Estelite)	3.81	0.01	p < 0.05
Group 3 vs Group 1 (Beautifil II vs Fuji IX GP)	8.87	0.001	p < 0.05
Group 2 vs Group 4 (Dyract XP vs Estelite)	-1.06	0.899	p > 0.05
Group 2 vs Group 1 (Dyract XP vs Fuji IX GP)	4	0.01	p < 0.05
Group 4 vs Group 1 (Estelite Sigma Quick vs Fuji IX GP)	5.06	0.001	p < 0.05

The post-hoc Tukey HSD analysis revealed that specimens from Group 3 (Beautifil II) exhibited statistically significantly higher mean bond strength values compared to all other groups (Table 3, $p < 0.05$). The mean bond strength of specimens from Group 1 (Fuji IX GP) was significantly lower than that of Groups 2, 3, and 4. Specimens from Group 2 (Dyract XP) showed higher mean bond strength values compared to Group 4 (Estelite, Table 3), although the difference was not statistically significant. Failure mode analysis showed that the majority of failures were adhesive, occurring at the interface between the dentin and the adhesive resin. This was particularly evident in Group 1 (Fuji IX GP) and Group 4 (Estelite Sigma Quick).

DISCUSSION

Achieving a strong bond between the restorative material and dentin is crucial for the longevity of restorations. Adhesion of restorative materials can be achieved through the use of adhesive systems, or by using restorative materials that bond to dentin without an adhesive system, as is the case with GICs [14]. The other materials examined in our study—compomer, giomer, and composite—establish their bond with dentin through two one-step self-etch adhesive systems and one two-step self-etch adhesive.

Application of an adhesive system creates a bond with dentin through micromechanical retention. Such retention occurs when the adhesive fully penetrates the dentin surface, forming a hybrid layer [15]. The failure mode analysis revealed that the majority of fractures were adhesive. These results suggest that the interface remains the weakest link in the restoration of primary teeth. In Group 3, although the failure was also primarily adhesive, the integration provided by the 10-MDP monomer and the S-PRG fillers likely shifted the stress distribution, resulting in higher resistance compared to the other restorative systems.

The bond strength depends more on the quality rather than the thickness of the hybrid layer [16]. The quality of this layer depends on strict adherence to the application protocol, as well as both the etching potential of the self-etching adhesives and the chemical interactions between functional monomers (such as 10-MDP) and the dental tissues. All three adhesives investigated in this study are classified as mild self-etch systems. This would suggest similar bond strength values among specimens in the three groups. However, according to the results of this study, there was a statistically significant difference in the values for all restorative materials in combination with their respective adhesive systems, except for the difference between Dyract XP and Estelite (Table 3).

Dentin as a substrate can also influence the adhesive bond strength. According to data from the specialized literature the thickness of the remaining dentin, as well as the localization of the lesion, affect bond strength, with bonding to deep dentin appearing to be weaker [17]. Since the preparation of the specimens ensured their uniformity with respect to these characteristics, we assume that the differences in the obtained mean values are attributable to the different composite systems used.

It is widely recognized in the scientific literature that the strongest bond to dentin is achieved when using total-etch adhesives [18, 19, 20]. Shortening the procedure by applying self-etch adhesives limits the penetration of monomers into dentin, which can lead to areas without hybridization or with uneven hybridization, and consequently, to a weaker bond to dentin [18]. There are opinions that two-step self-etch adhesive systems provide lower bond strength compared to three-step total-etch adhesives, but achieve better bonding than one-step self-etch adhesives [18]. The results of the present study support these statements, as the bond achieved with the two-step self-etch adhesive Clearfil SE Protect (Group 3) was significantly stronger than that obtained using one-step self-etch adhesives and glass ionomer cement (Table 3). The higher bond strength values of this adhesive may be attributed, on one hand, to its two-step self-etching application protocol, which results in a more uniform and acid-resistant bond with the hard dental tissues. [19, 21] On the other hand, the adhesive contains the 10-MDP monomer, which establishes a strong chemical bond with the tooth substrate through the formation of insoluble Ca-MDP salts, thereby reinforcing the hybrid layer. Consequently, the adhesion is both micromechanical and chemical. Furthermore, this adhesive possesses antibacterial properties, which is particularly advantageous in the treatment of primary teeth. [19, 21]. The S-PRG (Surface Pre-Reacted Glass) fillers within the giomer's composition may also contribute to the enhanced adhesion to dentin achieved by that composite system. In a moist environment, the absorption of water triggers the release of ions, facilitating a constant exchange of fluoride, calcium, strontium, and sodium ions with the hard dental tissues. [22] This mechanism potentially promotes a more stable and chemically integrated interface between the restorative material and the tooth structure.Our findings are consistent with other studies, which report that the application of a two-step self-etch adhesive system results in a stronger bond compared to the use of one-step adhesives and glass ionomer cement [18,19,20].

CONCLUSION

The results of this study indicate that the restorative material used and its corresponding adhesive system have a significant influence on the bond strength to dentin in primary teeth. The highest values were recorded for the giomer Beautifil II in combination with the two-step self-etch adhesive system Clearfil SE Protect. The combination of S-PRG fillers within the giomer's composition and the 10-MDP monomer in the adhesive interface, which provides a stable chemical bond with the hard dental tissues, may be responsible for the superior results observed in the specimens from Group 3.These findings highlight the need for careful consideration when selecting the material and adhesive protocol to ensure long-lasting restorations in the primary dentition.

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