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ORIGINAL ARTICLE

Impact of Different Photoinitiers of Adhesive Systems on Mechanical Properties, Sorption, Solubility, and Microtensile Bond Strength to Dentin

Luciane Ribeiro Saraiva[1], Rayssa Ferreira Cavaleiro de Macêdo[1], Anna Júlia Leão Pereira Martins[2], Darlon Martins Lima[1], Edilausson Moreno Carvalho[2], Jose Bauer[1]

[1]Department of Dentistry I, School of Dentistry, Federal University of Maranhão, São Luís, MA, Brazil.
[2]Postgraduate Program in Dentistry, University Ceuma, São Luís, MA, Brazil.

Corresponding author: Edilausson Moreno Carvalho **E-mail:** edilausson.carvalho@ceuma.br

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ABSTRACT

Objective: To evaluate the influence of photoinitiators on the physicochemical properties of simplified conventional adhesive systems. **Material and Methods:** Three adhesive systems were tested: Adper Single Bond 2 (3M), Ambar (FGM), and Ambar APS (FGM). For the cohesive strength (CS) test, specimens (n =10) were prepared and subjected to mechanical testing in a universal testing machine (Instron 3342). For the cross-link density (CLD) test, eight specimens of each material were prepared and subjected to Knoop microhardness measurements before and after immersion in ethanol for 24 hours. For the sorption and solubility (SS) test, ten specimens (n=10) of each material were prepared and tested over 28 days. For the dentin bond strength (BS) test, human molars (n=6) were restored, sectioned, and tested in a universal testing machine at a 1 mm/min speed. The data were analyzed using one-way ANOVA, two-way repeated measures ANOVA, and Holm-Sidak for mean contrasts (α=0.05). **Results:** Statistical analysis revealed no significant differences between the materials in the BS (p=0.40), CS (p=0.356), CLD (p=0.189), and solubility (p=0.157) tests. However, for the sorption test, a significant difference was observed between the adhesive systems, with Single Bond showing the highest values compared to Ambar and Ambar APS, which were similar. **Conclusion:** The APS photoinitiation system provides no advantage in the analyzed properties.

Keywords: Dentin-Bonding Agents; Mechanical Tests; Polymerization.

Introduction

Monomers in adhesive systems polymerize through an addition reaction, requiring initiators - molecules with low dissociation energy that form free radicals upon thermal, light, or chemical activation [1,2]. Depending on the initiator-activator system, adhesives can be classified as photoactivated, chemically activated, or dual-activated [3,4].

The efficiency of the photoinitiator in adhesive systems is directly linked to the degree of conversion, which is frequently associated with enhanced mechanical properties of these materials [5,6]. These properties include cohesive strength, microhardness, and resistance to solvent softening. Another crucial factor related to adhesive systems is the amount of hydrophilic monomers, such as HEMA, in their composition. These monomers can hinder the effectiveness of some photoinitiators and co-initiators, essential for the transition from monomers to polymers [7,8].

Although it has disadvantages, a widely used photoinitiator system in adhesive materials features camphorquinone (CQ) [9,10]. These include aesthetic issues due to its intense yellow color and a low polymerization rate, necessitating the presence of a tertiary amine as a co-initiator to enhance the conversion reaction [11]. Additionally, the CQ-amine system can cause composites to change color after photoactivation, as CQ is consumed during polymerization [12].

To address these drawbacks, alternative photoinitiator systems to camphorquinone have been proposed. Recently, a new photoinitiation system, APS (Advanced Polymerization System), was introduced to the dental market, incorporating a unique polymerization system developed by the manufacturer FGM [13]. The APS system combines various photoinitiators that enhance the degree of conversion, thereby improving mechanical properties, and is practically colorless, avoiding any aesthetic interference [14].

Therefore, this study aimed to evaluate the physicochemical properties and bond strength of simplified conventional adhesive systems with different activation components. The null hypothesis tested was that there would be no difference in the physicochemical properties and bond strength among the tested adhesives.

Material and Methods

Adhesive Systems

Three conventional adhesive systems were used: Adper Single Bond 2 (3M ESPE, St. Paul, MN, USA), Ambar (FGM Dental Group, Joinville, SC, Brazil), and Ambar APS (FGM Dental Group, Joinville, SC, Brazil). The composition, batch numbers, manufacturers, and application modes for the bond strength test are described in Table 1.

Cohesive Strength

Ten specimens of each adhesive were prepared in a dumbbell shape, measuring 10 mm in length, 2 mm in depth, and 2 mm in width, with a cross-sectional area of 1 mm^2. After lubricating all mold surfaces with vaseline, the adhesives were applied to the mold and filled. An air jet was applied for 10 seconds at 10 cm for solvent evaporation. A transparent polyester strip (3M ESPE, St. Paul, MN, USA) was placed over the surface, followed by light-curing for 60 seconds (Radii-cal, SDI - 1200 mW/cm^2). The specimens were stored for 24 hours in an incubator at 37°C. Subsequently, they were subjected to cohesive strength testing using a universal testing machine Instron 3342 (Instron, Norwood, MA, USA) at a 1 mm/min speed until specimen failure. The load values in Newtons (N) were converted to Megapascals (MPa).

Table 1. Composition, application mode, and batch number of the adhesive systems.

Material / Batch Number	Composition	Application Procedure*
Adper Single Bond 2 (3M/ESPE) (1760262)	Dimethacrylate resins, Bis-GMA, HEMA, Vitrebond copolymer, filler, ethanol, water, initiators (not disclosed by the manufacturer)	Application of 37% phosphoric acid on dentin for 15 s; Rinse for 15 s; Drying with absorbent paper; Application of adhesive drop (10 s) vigorously; Repeat step 4; Air-blowing for 10 s at a distance; Photoactivation (10 s)
Adper Single Bond 2 (3M/ESPE) (1760262)	Dimethacrylate resins, Bis-GMA, HEMA, Vitrebond copolymer, filler, ethanol, water, initiators (not disclosed by the manufacturer)	Application of 37% phosphoric acid on dentin for 15 s; Rinse for 15 s; Drying with absorbent paper; Application of adhesive drop (10 s) vigorously; Repeat step 4; Air-blowing for 10 s at a distance; Photoactivation (10 s)
Ambar APS (FGM) (170117)	MDP (10-Methacryloyloxydecyl dihydrogen phosphate); Methacrylate monomers; Photoinitiator composition [Phenyl bis (2,4,6- trimethylbenzoyl)-phosphine oxide - APS], Co-initiators, and stabilizer. Silica nanoparticles and ethanol	Application of 37% phosphoric acid on dentin for 15 s; Rinse for 15 s; Drying with absorbent paper; Application of adhesive drop (10 s) vigorously; Repeat step 4; Air-blowing for 10 s at a distance; Photoactivation (10 s)

*The adhesive systems were applied according to the manufacturer's instructions.

Cross-Link Density

Twenty-four specimens (n = 8) of each adhesive system were prepared using a cylindrical metal mold (5 mm diameter × 2 mm thickness; Odeme Dental Research, Luzerna, SC, Brazil). The adhesives were applied to the mold after lubricating all mold surfaces with Vaseline (Quimidrol Comércio, Indústria e Importação Ltda, Joinville, SC, Brazil). An air jet was applied for 10 seconds at 10 cm for solvent evaporation. A transparent polyester strip (3M ESPE, St. Paul, MN, USA) was placed over the surface before light-curing. An LED curing light Radii-cal (SDI Limited, Bayswate, Australia) with a power density of 1200 mW/cm² was used for 60 seconds on each specimen. After polymerization, the specimens were carefully removed from the mold with a scalpel blade No. 15, and any remaining burrs were removed. The specimens were then stored in a dry incubator at 37°C for 24 hours. After this period, four indentations (Knoop indenter) were made on the surface of each specimen using a microdurometer HMV-2 (Shimadzu Corp., Kyoto, Japan) with a load of 10g for 15 seconds. Following the initial measurement, the specimens were stored in 100% ethanol solution at 37°C for 24 hours, after which new measurements were taken. Cross-link density was estimated by the softening effect promoted by ethanol, indicated by the decrease in superficial hardness [15].

Sorption and Solubility

For these tests, 10 specimens were prepared using a metal mold (5 mm diameter × 2 mm thickness; Odeme Dental Research, Luzerna, SC, Brazil). The specimens were prepared as previously described for the Cross-Link Density test. After polymerization, the specimens were carefully removed from the mold with a scalpel blade No. 15, and any remaining burrs were removed. The specimens were then stored in a desiccator containing silica at room temperature for 60 days and subsequently weighed (M1).

After this period, the specimens were stored in distilled water at 37°C and weighed at specific intervals: every 24 hours for 7 days and at 14, 21, and 28 days. An analytical balance AUX-220 (Shimadzu Corp., Tokyo, Japan) with a precision of 0.0001 g was used. The volume of water for immersion was 5 ml per sample. Before weighing, each specimen was carefully dried with absorbent paper, and the weight was recorded as M2. The specimens were then desiccated for 30 days and weighed daily until a final stable mass (M3) was obtained. Water sorption (WS) and solubility (SL) values (in μg/mm³) were calculated using the following formulas: $WS = (M2 - M3) / V$; $SL = (M1 - M3) / V$. The volume of each specimen was determined using the formula $V = \pi r^2 h$, where πr^2 is the area of the specimen, and h is the average height (thickness) measured with a caliper (Absolute Digimatic). The protocol for this study was adapted according to ISO 4049 standards.

Microtensile Bond Strength Test

Sample Preparation

Flat dentin surfaces were created on all teeth used in the study by removing the occlusal enamel with a low-speed diamond disk Isomet 1000 (Buehler, Lake Bluff, IL, USA) under irrigation (n=6). The exposed, enamel-free dentin was polished with 600-grit silicon carbide sandpaper for 10 seconds to standardize the smear layer [16]. The dentin surfaces were examined under an optical microscope at 40x magnification to confirm the absence of enamel.

Restorative Procedure

After acid conditioning and adhesive application (Table 1), restorations were performed using Filtek Z350 composite resin (3M ESPE, St. Paul, MN, USA) in increments of approximately 6.0 mm in height, applied in three 2.0 mm layers. Each resin increment was light-cured for 20 seconds using an LED curing light Radii-cal (SDI Limited, Bayswate, Australia) at 1200 mW/cm². The specimens were then stored in distilled water and placed in an incubator at 37°C for 24 hours.

Preparation of Specimens

Each tooth was secured with sticky wax to a cutting machine device ISOMET 1000 (Buehler, Lake Bluff, IL, USA) with the bonding interface perpendicular to the cutting disc. Two sequences of longitudinal and perpendicular cuts were made to obtain stick-shaped specimens with a rectangular cross-sectional area of 0.8±0.1 mm². The number of sticks that were prematurely lost during preparation was recorded.

Bond Strength Test

Each specimen had its adhesive interface measured with a digital caliper with 0.001 mm precision (Mitutoyo Corp., Kanagawa, Japan). The specimen was then fixed with cyanoacrylate gel glue (BSI Inc., Hailsham, UK) in a microtensile grip (Odeme Dental Research, Luzerna, SC, Brazil). This grip was attached to a universal testing machine Instron 3342 (Instron, Norwood, MA, USA), ensuring that tensile stresses occurred perpendicular to the bonding interface. The tests were conducted at a 1 mm/min speed until specimen failure, and results were expressed in MPa using BlueHill software.

Statistical Analysis

Statistical analysis was performed using SigmaPlot software (SigmaPlot 13.0, Systat Software Inc., San Jose, CA, USA). All data were subjected to the Shapiro-Wilk normality test (α=0.05), confirming a normal distribution in all evaluations. A test power of 80% was selected for all analyses. Cohesive strength, microhardness, and sorption and solubility data were analyzed using one-way ANOVA and the Holm-Sidak post-test (α=0.05). For the microtensile bond strength test, the tooth was defined as the experimental unit. The mean of the sticks from each tooth (in each evaluation period) was used for statistical purposes. Bond strength values for each group were calculated as the mean between the teeth in each group. Bond strength data were analyzed using two-way ANOVA (adhesive system x storage time) and the Holm-Sidak post-test (α=0.05).

Ethical Clearance

This research was approved by the Research Committee of the Federal University of Maranhão (Opinion No. 3.469.428).

Results

The means and standard deviations for bond strength (MPa), cohesive strength (MPa), cross-link density (%), and sorption and solubility (µg/mm³) tests are described in Table 2. The analysis of variance revealed no statistically significant differences between the groups for cohesive strength (p=0.356), cross-link density (p=0.489), and microtensile bond strength tests (p=0.40). However, the sorption/solubility tests showed a significant difference between adhesive systems, with the Single Bond material exhibiting higher water sorption values ($p<0.05$) compared to the Ambar and Ambar APS adhesives, which showed similar values to each other ($p>0.05$).

Table 2. Means and standard deviation of the tests performed.

Material	Bond Strength (MPa)	Cohesive Strength (MPa)	Cross-Link Density (%)	Sorption (µg/mm³)	Solubility (µg/mm³)
Single Bond 2	52.8 ± 8.1^{A}	11.3 ± 2.2^{A}	54.0 % ± 20.4^{A}	148.6 ± 9.6^{A}	56.0 ± 5.8^{A}
Ambar	53.8 ± 16.1^{A}	9.7 ± 1.5^{A}	55.4% ± 3.3^{A}	130.6 ± 12.1^{B}	61.1 ± 17.2^{A}
Ambar APS	45.2 ± 8.8^{A}	9.6 ± 1.8^{A}	50.2% ± 13.1^{A}	124.9 ±10.5^{B}	66.7 ± 21.3^{A}

*Equal letters indicate statistically similar results within the columns.

Discussion

In recent years, adhesive dentistry and photopolymerization science have gained increasing relevance, leading to new technologies to enhance resin materials' physicochemical and mechanical properties, including adhesive systems [17]. This study investigated the impact of alternative photoinitiators on cohesive strength, microhardness, solvent softening, sorption and solubility, and bond strength of three simplified conventional adhesive systems with different photoinitiators in their compositions.

Cohesive strength and cross-link density tests are crucial for evaluating the polymerization efficiency of adhesive systems. In this study, no statistically significant differences were observed in the values obtained from both tests (Table 2). These results suggest that the photoinitiator agents used in the evaluated adhesive systems, specifically camphorquinone and the APS system, perform similarly in forming the three-dimensional polymer network. This network directly influences the material's mechanical performance, supporting the findings observed for the cohesive strength of these materials [15,18]. A high cross-linkage density tends to confer more excellent resistance to degradation [19]. However, beyond the photoinitiator type and concentration, other factors, such as the type of monomers used and light intensity, also influence the formation and subsequent degradation of the polymer [20,21].

Regarding water sorption, the Adper Single Bond 2 adhesive absorbed significantly more water than the other adhesives, consistent with previous studies [15,22]. One possible explanation for this result is the presence of hydrophilic monomers like HEMA in the adhesive composition, which, at higher concentrations, may have increased the hydrophilicity of this material [19,23].

Solubility is a phenomenon that degrades polymers through the release of unreacted or hydrolyzed components, depending mainly on the adhesive composition and polymerization efficacy [6,24]. In contrast to sorption testing, the solubility results in this study showed no significant difference among the tested groups. Despite its considerable water sorption, Adper Single Bond exhibited solubility values similar to those of the other tested adhesive systems.

Stabilizing the adhesive interface remains a significant challenge in dentistry, as the deterioration of this layer directly affects the success of the restorative procedures. This degradation can occur due to the activity of

metalloproteinases on the organic collagen matrix, the action of water on the polymer components of the adhesive [24,25], or insufficient conversion of monomers into polymers during photoinitiation, which increases the permeability of the adhesive layer [26], making it more susceptible to degradation. Therefore, adhesives with higher water sorption are assumed to exhibit more significant degradation and subsequent monomer leaching. However, this behavior was not observed in the experimental groups evaluated in this study, as demonstrated by the solubility data. This phenomenon may be attributed to the short analysis period or insufficient water absorbed by the material.

The composition of the APS (Advanced Polymerization System) photoinitiation system present in the tested commercial adhesive system, Ambar APS, was not disclosed by the manufacturer, complicating the study, as knowing each component of the composition would be essential. In this study, adhesives containing alternative photoinitiators showed bond strength values statistically similar to those of the adhesive with camphorquinone. Similar results were found by Hass et al. [27]. This suggests that replacing camphorquinone with the APS system does not interfere with the quality of the hybrid layer formed and, consequently, the mechanical performance of these materials.

Conclusion

The findings of this study suggest that replacing camphorquinone with the APS photoinitiation system does not interfere with the analyzed properties. However, further tests are needed to confirm the clinical advantages of using these alternative materials.

Authors' Contributions

LRS	https://orcid.org/0009-0003-2871-729X	Methodology, Validation, Investigation, and Writing - Original Draft.
RFCM	https://orcid.org/0000-0002-8364-2505	Writing - Review and Editing.
AJLPM	https://orcid.org/0009-0005-6855-5808	Writing - Review and Editing.
DML	https://orcid.org/0000-0001-9113-1206	Methodology and Supervision.
EMC	https://orcid.org/0000-0003-3475-3687	Formal Analysis and Data Curation.
JB	https://orcid.org/0000-0003-3761-0331	Conceptualization, Project Administration, and Funding Acquisition.

All authors declare that they contributed to a critical review of intellectual content and approval of the final version to be published.

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Conflict of Interest

The authors declare no conflicts of interest.

Data Availability

The data used to support the findings of this study can be made available upon request to the corresponding author.

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