In vitro evaluation of incompatibility between simplified adhesive systems and dual-cure resin cement

Avaliação in vitro da incompatibilidade entre sistemas adesivos e cimento resinoso dual

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ABSTRACT

Objective
The aim of this study was to evaluate the incompatibility between simplified adhesive systems and dual-cure resin cement by means of marginal micro-leakage testing.

Methods
Standardized cavity preparations were performed in 30 bovine teeth. The adhesive systems Adper Scotchbond Multipurpose Plus (3M ESPE, St. Paul, MN, USA) - Group A, Excite (Ivoclar/Vivadent, AG, Liechtenstein) - Group B, and Adper Prompt L-Pop (3M ESPE, St. Paul, MN, USA) - Group C, were applied and the cavities were filled with dual-cure resin cement (Enforce - Denstply, Milford, DE, USA). The samples were submitted to five hundred thermal cycles, after which they were immersed in 2% buffered methylene blue.

Results
Group A presented the lowest mean percentage of micro-leakage (3.34%), followed by Group B (13.60%) and Group C (48.33%). Significant differences were found among the groups after the application of the Kruskal-Wallis statistical test (P=0.00).

Conclusion
The highest mean marginal micro-leakage values were shown with the use of the one-bottle etch-and-rinse adhesive system and the all-in-one self-etch system, which demonstrates the incompatibility between these materials.

Indexing terms: Adhesives. Dental cements. Dental leakage.

RESUMO

Objetivo
Avaliar a incompatibilidade entre sistemas adesivos e cimento resinoso dual através de teste de microinfiltração marginal.

Métodos
Foram realizados preparos cavitários padronizados em 30 dentes bovinos divididos em 3 grupos: A) Adper Scotchbond Multipurpose (3M ESPE, St. Paul, MN, USA); B) Excite (Ivoclar/Vivadent, AG, Liechtenstein) e C) Adper Prompt L-Pop (3M ESPE, St. Paul, MN, USA). Após aplicação dos adesivos, as cavidades foram preenchidas com cimento resinoso dual Enforce (Denstply, Milford, DE, USA), submetidos à ciclagem térmica (500 ciclos) e imersos em azul de metileno 2% tamponado.

Resultados
O grupo A apresentou os menores percentuais de microinfiltração (3,34%), seguido pelo grupo B (13,60%) e pelo grupo C (48,33%). Após aplicação do teste estatístico Kruskal-Wallis, foram detectadas diferenças estatisticamente significativas entre os três grupos (P=0.00).

Conclusão
Houve incompatibilidade na associação entre os sistemas adesivos, convencional de dois passos (Excite, Ivoclar/Vivadent, AG, Liechtenstein) e autocondicionante de passo único (Adper Prompt L-Pop (3M ESPE, St. Paul, MN, USA), e o cimento resinoso dual Enforce (Denstply, Milford, DE, USA).

Termos de indexação: Adesivos. Cimentos dentários. Infiltração dentária.

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INTRODUCTION

There is an increasing demand for indirect restorations that match the shade of natural teeth, and this is undoubtedly due to the many advantages they offer, such as mechanical functional improvements and enhanced esthetic appearance, when compared with direct restorations[1-2].

When use of the indirect restorative technique is proposed, it is necessary to have knowledge of adhesive agents, as there are numerous materials available on the market, sold in various formulations with different properties. Nevertheless, indirect aesthetic restorations are now routinely bonded to the tooth substrate with the use of adhesive resin cements[3]. These resin cements basically present the same composition as those of the resin composites, an organic matrix consisting of monomers, Bis-GMA (bis-phenol A glycidyl methacrylate) or UDMA (urethane di-methacrylate) and TEGMA (tri-ethylene glycol di-methacrylate), and inorganic particles surrounded by a bonding agent[4-5]. Nevertheless, in order to achieve an effective bond between resin cement/ dental substrate, it is necessary to apply an adhesive system capable of influencing the retention and bond of the restoration[6].

There is a very common clinical trend towards conciliating adhesive systems and resins (or dual-cure resin cements) from different manufacturers and of different classifications. Although no adverse reaction will occur with the majority of the associations, it has been observed that dual activated resins are incompatible with all-in-one self-etch adhesives and one-bottle etch-and-rinse adhesives, both of which are formulations designated as simplified[6-7].

In the case of the simplified adhesives, in addition to other di-methacrylates, the oxygen inhibition layers contain acidic vinyl monomers with carboxylic or phosphate ester groups. When these adhesives are used together with chemical-cured or dual-cured composites, there is an interaction of the residual acidic resin monomers from the adhesive inhibition layer with the binary peroxide-amine catalytic components which are commonly used in the chemically-cured or dual-cured composites. This will result in slow or no polymerization, depending upon the acidity and concentration of the acidic resin monomers. The non polymerization of these materials produces a zone of high ionic concentration (hypertonic) and results in water movement from the dentin substrate into the non-polymerized interface. This leads to an immiscible blend of hydrophobic and hydrophilic monomers accumulating at the adhesive/dual-cure resin cement interface in the form of blisters and resin globules, challenging the durability of resin-dentin bonds produced. The origin of this permeability may probably be attributed to modifications in the adhesive formulations, in an attempt to make them more hydrophilic[8-11].

In view of the above explanation, the aim of this study was to evaluate *in vitro* the incompatibility existing between adhesive systems and dual-cure resin cements by means of marginal micro-leakage testing.

METHODS

For this research, 30 recently extracted bovine mandibular incisors were selected, cleaned under running water, using periodontal instruments and pumice stone paste and water.

Subsequently, circular cavities were prepared in the middle region of the vestibular surface of teeth, using of a diamond tip No. 2294® (KG Sorensen), mounted in a high speed hand piece under constant air-water cooling. On conclusion, the cavities were 1.5 mm deep and had an internal diameter of 1.5 mm corresponding to the active tip of the diamond tip used.

After cavity preparation, the teeth were randomly divided into 3 groups according to the type of adhesive system used: Group A: (n=10) 3-Step Etch-and-Rinse Adhesive System: Adper Scotchbond Multipurpose Plus (3M ESPE, St. Paul, MN, USA); Group B: (n=10) One-Bottle Etch-and-Rinse Adhesive System: Excite (Ivoclar/Vivadent, AG, Liechtenstein); Group C: (n=10) All-in-One Self-Etch Adhesive System Adper Prompt L-pop (3M ESPE, St. Paul, MN, USA).

In the next step the procedures of bonding to dentin were performed in accordance with the manufacturers' recommendations. Afterwards, the cavities were filled with dual-cure resin cement Enforce (Denstply/ Caulk) inserted by the incremental technique and light activated for 20 seconds (XL 3000 - 3M ESPE (St. Paul, MN, USA) - 450 mW/cm^2). The finishing and initial polishing were performed with rubber cups and points (Flexicups and Flexipoints, Cosmedent Inc., Chicago, IL, USA) at low speed. Final polishing was performed using Enamelize paste (Cosmedent Inc., Chicago, IL, USA) and a diamond felt disc (FGM Joinville, Santa Catarina, Brazil) at low speed.

The test specimens were stored in distilled water at 37°C for 24 hours. The compositions of the materials used are shown in Chart 1.

Thermal cycling consisted of 500 thermal cycles, with water baths of 5° C and 55° C, for 1 minute each. The root apexes were first sealed with resin composite and two coats of fast setting epoxy resin (Araldite, Brascola, São Bernardo do Campo, Brazil) to avoid possible penetration by the dye solution. After this the test specimens were sealed with two coats of coloured nail varnish, ensuring that the restorations and an area of 1.0 mm around them were not covered by varnish[12]. They were then immersed for 4 hours in a buffered 2% methylene blue dye solution. After that, the teeth were washed under running water, sealing was removed and they were submitted to longitudinal cuts through the centre of the restoration, using a double faced diamond disk (KG Sorensen) under cooling.

Dye penetration was assessed by stereomicroscopy (Zeiss® Axiotech) at 20x magnification, using a digital capture image device. The images were stored in TIFF format files on a computer hard drive. The analysis of leakage measurement was performed on a computer using the Image Tool program (Soft Imaging System SIS - analySIS®) and the following ratio was recorded: (length of methylene blue micro-leakage/total cavity length) x 100 (Figure 1).

RESULTS

After microscopic evaluation of dye solution penetration at the tooth-resin cement interface, it was observed that Adper Scotchbond Multipurpose Plus (3M ESPE, St. Paul, MN, USA) presented the lowest mean percentage of marginal micro-leakage, followed by Excite (Ivoclar/Vivadent, AG, Liechtenstein), with an intermediate value and Adper Prompt L-Pop (3M ESPE, St. Paul, MN, USA), which presented the highest mean percentage of micro-leakage (Table 1).

Statistical data analysis was based on the Kruskal-Wallis non parametric test used at a level of confidence of 95%, and significant differences were found among the groups (P = 0.00).

In Figure 2 micro-leakage can be observed for each of the systems used. After analysis of the quantiles and the median, it was found that the differences among the group were considered significant.

Table 1. Mean percentages of micro-leakage according to the adhesive system used

Adhesive System	Number of cavities (n)	Mean percentage of micro-leakage
AdperScotchbond Multipurpose Plus	10	3.34%
Excite	10	13.60%
Adper Prompt L-Pop	10	48.33%

Chart 1. Manufacturer and composition of the materials used.

Adhesive system	Manufacturer	Composition
Adper Scotch Bond Multipurpose Plus	3M ESPE (St. Paul, MN,USA)	Primer: 2-hydroxyethylmethacrylate (HEMA) Poly-alkenoic Acid Adhesive: Bis Glycidylmethacrylate (Bis GMA) Photo-initiators 2-hydroxyethylmethacrylate (HEMA)
Excite	Ivoclar/Vivadent (AG, Liechtenstein)	Adhesive: 2-hydroxyethylmethacrylate (HEMA) Di-methacrylates Phosphinic acid acrylate Dispersed silica dioxide Initiators
Adper Prompt L-POP	3M ESPE (St. Paul, MN,USA)	Liquid A: Mono and di-hema phosphates Di-methacrylates Camphoroquinone Aromatic amine Liquid B: Water Substituted Phenol Methacrylate polycarbonic acid 2-hydroxyethylmethacrylate (HEMA)
Dental Cement Enforce	Dentsply/Caulk (Milford, DE,USA)	Basic paste: TEGMA BDMA Boron glass Aluminum silicate Silanized Barium Camphoroquinone Benzyl Peroxide Catalyzer paste TEGMA Bis-GMA Titanium dioxide Mineral pigments

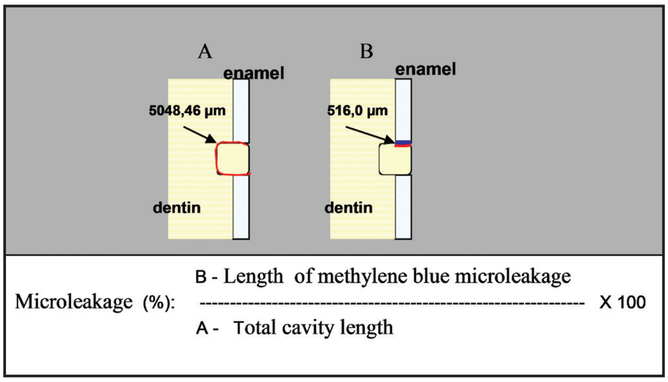

Figure 1. Leakage measurement method used.

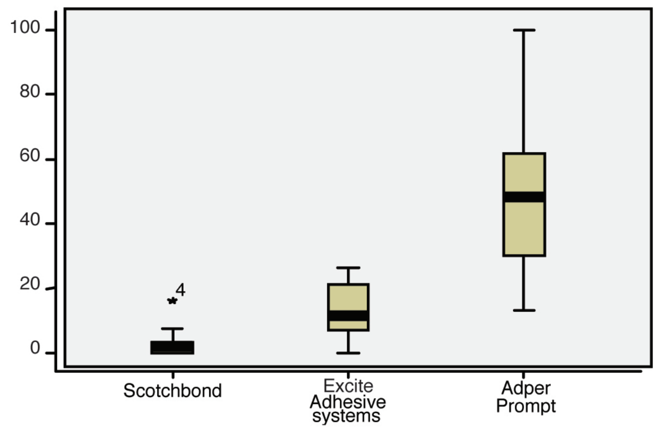

Figure 2. Box-Plot with the measurements of the centre of dispersion and the variability for micro-leakage according to the adhesive system used.

DISCUSSION

The clinical association of simplified adhesive systems with chemically activated or dual-cure resin cements results in an interaction that negatively affects the success of cementation procedures of indirect restorations or intraradicular posts. Indeed, the use of these adhesive systems produces a superficial layer with a high concentration of residual acidic resin monomers, due to the inhibition of polymerization by the superficial oxygen inhibited layer. These residual acidic resin monomers interact with binary peroxide-amine catalytic components that are commonly used in chemically-cured or dual-cured resin composites, thus affecting the polymerization reaction[10].

This study tested the incompatibility between different products, by using the marginal micro-leakage methodology. As a result it was found that there were statistically significant differences among the groups when adhesive systems and dual-cure resin cement were associated, corroborating the findings of previous studies[10,13].

The 3-step etch-and-rinse adhesive Group, Adper Scotchbond Multipurpose Plus (3M ESPE, St. Paul, MN, USA), presented the lowest marginal micro-leakage values (Table 1) among the adhesives researched. This was possibly due to the absence of acidification on the non polymerized surface, thus not resulting in the deleterious effects of the incompatibility between the acid resin monomers and chemical reaction activators of the dual-cure resin cement (peroxide/amine system). Latta et al.[14] also found that this conventional adhesive system showed better performance in bond strength tests, when compared with another simplified system. Therefore, the 3-step etch-and-rinse adhesive Adper Scotchbond Multipurpose Plus (3M ESPE, St. Paul, MN, USA) could be considered the positive control in this study, due to its excellent attributes. Franco et al.[13] also reported that the 3-step etch-and-rinse adhesive system considerably decreased the acidity of the non polymerized surface. They attributed this to the clinical protocol for the 3-step etch-and-rinse adhesive system requiring the application of a hydrophobic adhesive (Bis-GMA and UDMA) separately from the hydrophilic primer (HEMA).

There were statistically significant differences between the levels of micro-leakage observed for the one-bottle etch-and-rinse adhesive Excite (Ivoclar/Vivadent, AG, Liechtenstein) when compared with those of Adper Scotchbond Multipurpose Plus (3M ESPE, St. Paul, MN, USA). Sanares et al.[10], in a study that quantified the reduction in tensile strength, observed that the interaction between the one-bottle etch-and-rinse adhesive (simplified) with chemically activated composites was indeed prejudicial to the restorative bond.

This phenomenon occurs not only with chemically polymerized resin, but also with all dual polymerization resin materials, such as dual-cure resin cement, probably due to the one-bottle adhesives producing a more acid non polymerized surface than the 3-step etch-and-rinse adhesives. Moreoover, single-bottle adhesives behave as permeable membranes after polymerization[15]. It is known that the bond strength of the one-bottle etch-and-rinse adhesives is usually lower to that of the 3-step etch-and-rinse adhesives with light polymerized composites[16-17]. Nevertheless, the previously mentioned incompatibility may aggravate this situation, decreasing the bond strength and thus increasing the micro-leakage when these adhesives systems are used with dual-cure resin cement.

Among the classes of adhesives used in this research, the all-in-one self-etch system Adper Prompt L-Pop (3M ESPE, St. Paul, MN, USA) presented the highest mean percentages of marginal leakage, with statistically significant differences in comparison with the Adper Scotchbond Multipurpose Plus (3M ESPE, St. Paul, MN, USA) and Excite (Ivoclar/ Vivadent, AG, Liechtenstein) groups. These results allow one to suggest that the great influence of adhesives systems stems from the pH and its interaction with the components for activating the dual-cure resin cement, since Adper Prompt L-Pop (3M ESPE, St. Paul, MN, USA) had the lowest pH (<1.0), and presented the highest rates of marginal micro-leakage measured. This low hydrogen ionization potential is probably owing to the large presence of acid esters in its composition.

In addition to the interaction between the chemical activation mode of dual-cure resin cements and the all-in-one self-etch adhesives being responsible for the reduction in bond effectiveness[18], dentinal permeability is another important factor to consider[11]. This is because all-in-one self-etch adhesives behave like permeable membranes after polymerization, because of the absence of a more hydrophobic resin component, and consequently not providing hermetic dentinal sealing[11].

Strong self-etch adhesives (all-in-one) usually have a pH of 1 or below. This high acidity results in rather deep demineralization effects. It has often been documented that such adhesives presented rather low bond strength values and quite a high number of pre-testing failures when assessed after a micro-tensile bond strength approach. This more acid formulation also largely favours the effect of incompatibility, even when compared with a 2-step self-etch system (primer - acid + adhesive), since the latter makes use of a final hydrophobic layer[19].

Therefore, it is important to consider that adverse chemical reactions that affect the adhesive interface with dual-cure resin cements may be directly related to the pH of the adhesive used, i.e., to their composition in terms of acid monomers[20].

CONCLUSION

According to the results obtained, it was possible to conclude that there was incompatibility between the simplified adhesive systems (Adper Prompt L-Pop, 3M ESPE, St. Paul, MN, USA; and Excite, Ivoclar/Vivadent, AG, Liechtenstein), and the dual-cure resin cement (Enforce), as demonstrated by the higher mean percentages of marginal leakage. The three-step etch-and-rinse system (Adper Scotchbond Multipurpose Plus, 3M ESPE, St. Paul, MN, USA) presented better interaction with the dual-cure resin cement and lower mean percentages of marginal micro-leakage.

Collaborators

RJA FIGUEIREDO and AKM ANDRADE were responsible for performing the microleakage tests and writing the article. RM DUARTE and MAJR MONTES participated in writing and correcting the article. FDSCM SILVA and ICM PORTO were responsible for the statistical analysis and writing the article.

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