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Tooth-restoration adhesive interfacial characterization using different universal dental adhesives

Investigation Monography

Master Degree in Dental Medicine

Faculty of Dental Medicine, Oporto University

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À minha família, especialmente à minha mãe, que é e sempre será o meu porto de abrigo.

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Abstract

Background: In adhesive dentistry, an adequate and long-term bond to dentin and enamel is required to achieve clinical success**.** Due to the increased demand for flawless restorations, it became necessary to evaluate if the new adhesive systems are effective in reducing the marginal microleakage.

Aim: The interaction characterization between universal adhesives and the tooth structure and the evaluation of this interaction effects, through a qualitative analysis of the tooth/restoration marginal microleakage.

Materials and Methods: Thirty intact caries-free human third molars were collected. Class I cavities were prepared with an occlusal depth of approximately 3 mm. The samples were randomly divided into three different adhesive groups: Group I: Prime&Bond active™ (Dentsply Sirona, Konstanz, Germany) (PBA); Group II: G-Premio BOND (GC, Tokyo, Japan) (GB); Group III: Futurabond M+ (VOCO, Cuxhaven, Germany) (FB). The adhesives were applied following the respective manufacturers' instructions and the cavities were restored with composite resin Grandio®SO (VOCO, Cuxhaven, Germany). All specimens were thermocycled for 500 cycles (dwell time of 60 seconds). Microleakage was tested with a dye penetration method followed by macroscope analysis. Microleakage scores were statistically analyzed through Binomial and Chi-Square tests.

Results: No statistically significance differences were found in the probability of success/failure of each adhesive regarding the presence/absence of marginal microleakage (p=0,5). No statistically significance differences were found in the relationship between the different types of adhesives and the presence/absence of marginal microleakage (p=0,627).

Conclusion: The different chemical properties of the evaluated adhesives, specifically solvents, had no influence on the tooth – restoration marginal microleakage.

KEYWORDS: "Universal adhesives", "Marginal microleakage", "Solvents", "Dentin-adhesive interface"

Resumo

Introdução: Na dentisteria adesiva, para alcançar o sucesso clínico é obrigatória a obtenção de uma adesão adequada e duradoura à dentina e ao esmalte. Devido ao aumento da demanda por restaurações imaculáveis, tornou-se necessário avaliar se os novos sistemas de adesivos universais são eficazes na redução da microinfiltração marginal.

Objetivo: Caracterizar a interação de três adesivos universais com a estrutura dentária e avaliar os efeitos dessa relação através de uma análise qualitativa da microinfiltração marginal.

Materiais e Métodos: Para este estudo foram coletados trinta terceiros molares humanos, íntegros e sem cárie. Classes I foram preparadas com cerca de 3mm de profundidade. As amostras foram aleatoriamente divididas por três grupos, de acordo com diferentes adesivos universais: Grupo I: Prime&Bond active™ (Dentsply Sirona, Konstanz, Germany) (PBA); Grupo II: G-Premio BOND (GC, Tokyo, Japan) (GB); Grupo III: Futurabond M+ (VOCO, Cuxhaven, Germany) (FB). Os adesivos aplicaram-se de acordo com as instruções dos fabricantes e as cavidades foram restauradas com a resina composta Grandio®SO (VOCO, Cuxhaven, Germany). Todas as amostras foram submetidas a um processo de termociclagem de 500 ciclos (tempo de permanência de 60 segundos). Os dentes foram depois sujeitos a um método de penetração de tinta para posterior avaliação da microinfiltração marginal em lupa macroscópica. Os níveis de microinfiltração foram estatisticamente analisados através dos testes Binomial e Qui-Quadrado.

Resultados: Não foram encontradas diferenças estatisticamente significativas na probabilidade de sucesso/insucesso de cada adesivo em relação há presença/ausência de microinfiltração marginal (p=0,5). Não foram encontradas diferenças estatisticamente significativas na relação entre os diferentes tipos de adesivos e presença/ausência de microinfiltração marginal (p=0,627).

Conclusão: As diferentes composições químicas dos adesivos avaliados, mais concretamente no que respeita os solventes, não tiveram influência na microinfiltração marginal dente-restauração.

PALAVRAS-CHAVE: "Adesivos Universais", "Microinfiltração marginal", "Solventes", "Interface adesivo-dentina".

Abbreviations

Bis-EMA Ethoxylated bisphenol A glycol dimethacrylate

Bis-GMA Bisphenol A glycidyl methacrylate

FB Futurabond M+

FDI World Dental Federation

GB G-Premio BOND

HAp Hydroxyapatite

HEMA 2-hydroxyethyl methacrylate

MDP 10-methacryloxydecyl dihydrogen phosphate

PB Prime&Bond active

Units

°C - Celsius degree

mm - Milimetre

mW/cm2 – Milliwatt per square centimetre

Symbols

% - percentage

N – Sample size

p – Statistical significance

α – Level of significance

χ^2 – Independence Chi-square test

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Introduction

In the last few years adhesive technology evolved rapidly.[1] Modern adhesive systems have a major impact on daily clinical practice as they simplify bonding procedures by reducing the number of application steps. Single-component or "all-in-one" self-adhesive systems combine acid-etching, primer application and the adhesive resin in a single step that require no mixing.[2] This self-etch approach provides simultaneous etching and priming of tooth structures, thereby allowing a protocol less technique sensitive and a reliable bonding performance.[3]

Recently, universal or multimode adhesives were launched in the market. These new single-bottle adhesives can be used in either self-etch or etch-and-rinse modes, depending on the clinical situation and the operators' personal preferences.[2] Despite their versatility, multimode adhesives are mainly self-etch adhesives. However, several studies demonstrated that the bond strength was improved when prior selective-enamel-etching was performed in the cavity.[4]

Based on micromechanical adhesive mechanism, the bonding performance of the various self-etching adhesives depend largely on the ability of adhesives functional monomers to penetrate the dental substrates.[5, 6] In addition, solvents also seem to play a major role in the formation of the anchoring structure: the hybrid layer.[7] Resin bond strength to dentin can be considered as the sum of individual adhesive forces arising from the penetration of resinous monomers into the intertubular (hybrid layer) and intratubular (resin tags) dentin.[8]

Universal adhesive systems are distinguished from previous adhesive generations by incorporating monomers capable to chemically bond to calcium in hydroxyapatite (HAp), which allows adhesive interfaces more resistant to biodegradation over time.[1, 2] In these adhesive systems, water is required in order to dissociate the acidic monomers and allow the self-etch strategy.[3] However, organic solvents such as ethanol, acetone and tertiary butanol should be added to prevent poor polymerization caused by the excess of residual water. The incorporation of these volatile solvents aid to dislodge the water from the dentin surface thus facilitating the penetration of resin monomers into nanospaces of the exposed collagen network after demineralization.[9] Furthermore, resin tags can be larger or shorter depending on the solvent influence in the dentinal tubules.

Because they promote intensification of wettability and resin infiltration, solvents proved to be, through scientific evidence, a component capable of increasing the adhesive bond strength.[8]

Therefore, it is important to determine if the variation in the composition of universal adhesives, namely the use of different solvents, could influence the dentin-resin interface.

In order to address this issue, the present study aims to characterize the interaction between universal adhesives and the tooth structure and to evaluate the effects of this interaction, through a qualitative analysis of the tooth/restoration marginal microleakage.

Material and Methods

Materials

The *in vitro* short-term experimental study, used the following materials: three universal adhesive systems, G-Premio BOND (GC, Tokyo, Japan), Futurabond M+ (VOCO, Cuxhaven, Germany), Prime&Bond active™ (Dentsply Sirona, Konstanz, Germany), all of them utilized in the self-etch approach; and an universal nano-hybrid restorative material, composite resin Grandio®SO (VOCO, Cuxhaven, Germany) (Figure 1).

Figure 1 - Universal adhesives and composite resin.

Table I - Materials, composition and application procedure.

Adhesives/Resin Composite	*Lote No.*	*Composition*	*Application Procedure*
G-Premio BOND (GC, Tokyo, Japan)	1902141	2-Hydroxy-1,3 dimethacryloxypropane, Acetone, Methacryloyloxydecyl dihydrogen phosphate, 2,2'-ethylenedioxydiethyl dimethacrylate, Diphenyl(2,4,6-trimethylbenzoyl)phosphine oxide, Butylated hydroxytoluene, Water	1. Apply to enamel and dentin surfaces using the disposable applicator; 2. Leave undisturbed for 10 seconds; 3. Dry thoroughly for 5 seconds under maximum air pressure; 4. Light cure for 10 seconds.
Futurabond M+ (VOCO, Cuxhaven, Germany)	1917225	2-Hydroxyethyl methacrylate, BIS-GMA, Ethanol, Acidic adhesive monomer, Urethanedimethacrylate, Catalyst, Pyrogenic silicic acids, Water	1. Apply the adhesive to the surfaces and rub it in for 20 seconds with the disposable applicator; 2. Dry off the adhesive layer with dry air for at least 5 seconds; 3. Light cure the adhesive layer for 10 seconds.
Prime&Bond active™ (Dentsply Sirona, Konstanz, Germany)	1812000802	Phosphoric acid modified acrylate resin, Multifunctional acrylate, Bifunctional acrylate, Acidic acrylate, Isopropanol, Water, Initiator, Stabilizer	1. Apply the adhesive to completely wet the surfaces to be treated; 2. Keep the adhesive slightly agitated for 20 seconds; 3. Treat every surface with a moderate air flow for at least 5 seconds; 4. Light cure adhesive for 10 seconds.
Grandio®SO (VOCO, Cuxhaven, Germany)	1919124	Bis-GMA, Triethylene glycol dimethacrylate, Bis-EMA	Apply the selected shade of GrandioSO in layers that are a maximum of 2mm thick, adapt with a suitable instrument and light-cure for 10 seconds (A3)

Abbreviations: Bis-GMA, bisphenol A glycidyl methacrylate; Bis-EMA, ethoxylated bisphenol A glycol dimethacrylate.

Specimen Preparation

Thirty intact caries-free human third molars, extracted for orthodontic or periodontal motives, were collected under a protocol approved by the Ethics Committee of Porto University School of Dentistry. The teeth were cleaned with an ultrasonic tip to remove periodontal fibers and bone remnants. After being disinfected in 0,5% chloramine, the teeth were stored for no more than 6 months in distilled water at 4°C. Class I cavities were prepared with an occlusal depth of 2,5/3 mm using both 1,2 mm rounded and cylindrical diamond turbine burs with continuous water irrigation. The cavity depth was measured with a calibrated periodontal probe.

The samples were divided into three different adhesive/solvent groups (n=10): Group I: Prime&Bond active™ (Dentsply Sirona, Konstanz, Germany) (PBA); Group II: G-Premio BOND (GC, Tokyo, Japan) (GB); Group III: Futurabond M+ (VOCO, Cuxhaven, Germany) (FB). The adhesives were applied following the respective manufacturers' instructions (Table 1) by rubbing each enamel/dentin surface using a microbrush, then air-dried and light-cured for 10 seconds (C02-C, Premium Plus, USA). Cavities were restored with 2mm increments of composite resin, Grandio®SO (VOCO, Cuxhaven, Germany). Each increment was light-cured for 10 seconds, except for the last one, which was cured for 20 seconds. Light curing was performed with an output of 1400 mW/cm^2 (C02-C, Premium Plus, USA). The occlusal surfaces were finished and polished with a diamond flamed shaped bur and a silicone diamond polisher slow hand-piece cup.

Marginal Microleakage Evaluation

After the cavities were restored, all bonded specimens were placed in the laboratory oven (Heraeus, T5028, Germany) at 37°C with 100% humidity for 24h, to decrease polymerization stress. Then, all specimens were thermocycled (Ethik Technology, 621-1D 220V 13L, Brazil) in baths of 5°C and 55°C, with a dwell time of 60 seconds for 500 cycles (Figure 2). In order to distinguish the groups, the samples of each group were wrapped into three gauzes with three different colored sewing threads (Figure 3). In the following step, the entire tooth surface was double coated with nail varnish to within 1/2mm of the restoration (Figure 4). The specimens were immersed in a 2% methylene blue solution dye for 24h in the laboratory oven at 37℃ (Figure 6).

Figure 3 - Distinction of each group for thermocycling procedure.

Figure 2 - Specimens in thermocycling procedure.

Figure 4 -Specimens after application of two layers of nail varnish.

After dye penetration, the teeth were thoroughly rinsed with distilled water and embedded in self-cured acrylic resin (Figure 5) to facilitate the cutting procedure. Each specimen block was buccolingually sectioned (Figure 7) in halves using a low-speed diamond saw on a hard tissue microtome (Acuttom®, Struers, Denmark) under continuous water irrigation. Twenty readable sections (ten blocks) per group were obtained (Figure 8) for dye penetration evaluation under photomacroscope (Wild

Heerbrug, M420, Switzerland/ Leica, DFC295, Germany) at 25x magnification. Digital images were acquired at 16x magnification (N=60).

Dye penetration scoring was performed using the following criteria regarding class I cavities:[10]

0 – No dye penetration;

1 – Dye penetration up to half of the restoration depth;

2 – Dye penetration greater than half of the restoration depth (to the pulpal floor);

3 – Dye penetration including the pulpal floor.

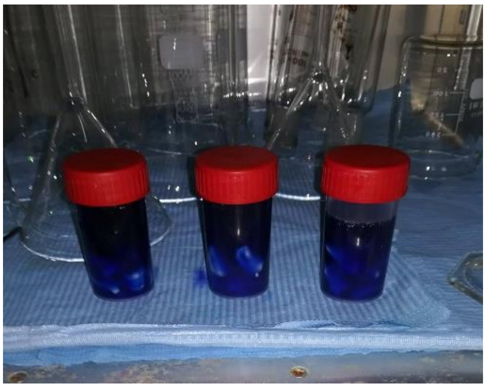

Figure 6 - Teeth immersed in a solution of 2% blue methylene for dye penetration.

Figure 5 - Teeth embedded in epoxy resin.

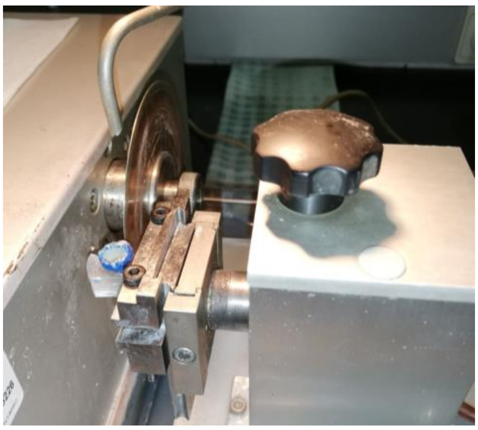

Figure 7 - Microtome cutting procedure.

Figure 8 - Three halves of each group prepared and labeled for dye evaluation.

Statistical Analysis

Statistical database analysis was performed with SPSS program (Statistic Package for Social Sciences; IBM® SPSS® Statistics version 25). Each half of the tooth was considered a statistical unit. The sixty halves were analyzed and scored twice by the same investigator, under the same conditions but at two different times. In order to evaluate internal consistency, the resulting halves scores were correlated in reliability analysis, via Cohen's kappa coefficient (k) (Table II).

Table II - Reliability analysis between Microleakage - 1st and Microleakage - 2nd

			Microleakage - 2nd				
			No dye penetration	Dye penetration up to half of the restoration depth	Dye penetration greater than half of the restoration depth	Dye penetration including the pulpal floor	Total
Microleakage – 1st	No dye penetration	Count	24	4	0	0	28
		% of Total	40,0%	6,7%	0,0%	0,0%	46,7%
	Dye penetration up to half of the restoration depth	Count	7	21	0	0	28
		% of Total	11,7%	35,0%	0,0%	0,0%	46,7%
	Dye penetration including the pulpal floor	Count	0	0	2	2	4
		% of Total	0,0%	0,0%	3,3%	3,3%	6,7%
Total		Count	31	25	2	2	60
		% of Total	51,7%	41,7%	3,3%	3,3%	100,0%

Microleakage – 1st: first time evaluation; Microleakage – 2nd: second time evaluation.

The reliability analysis demonstrated low internal consistency, k=0,615, and for this reason the obtained scores were reevaluated, concluding that the most accurate were the second ones (Microleakage – 2nd).

It should be noted that faced with such a low number (4/60) of scores “2” and “3”, the necessary assumptions for statistical inference analysis are not fulfilled. Consequently, the two last scores (“2” and “3”) were aggregated resulting in a new dichotomous variable: Absence or presence of microleakage (Microleakage A/P).

To demonstrate the probability of success and failure of each adhesive in relation to the presence or absence of microleakage, the statistical Binomial test was performed at a 0,5 probability. The Independence Chi-square test (χ^2) was proposed to evaluate whether there is a significant relationship between the three types of adhesives and the possibility of marginal microleakage. Both statistical tests were performed at a 5% level of significance (α=5%).

Results

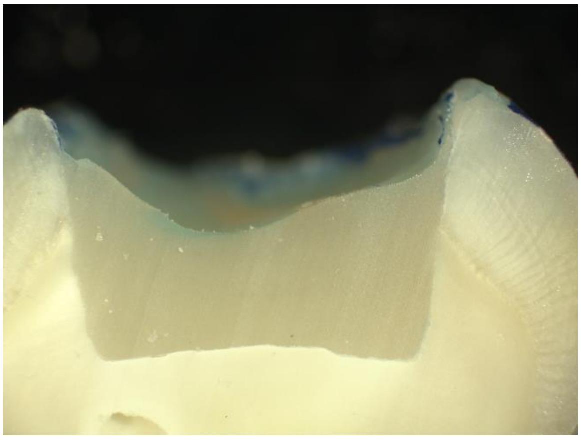

Figure 9 - Score "0" with Futurabond M+ adhesive.

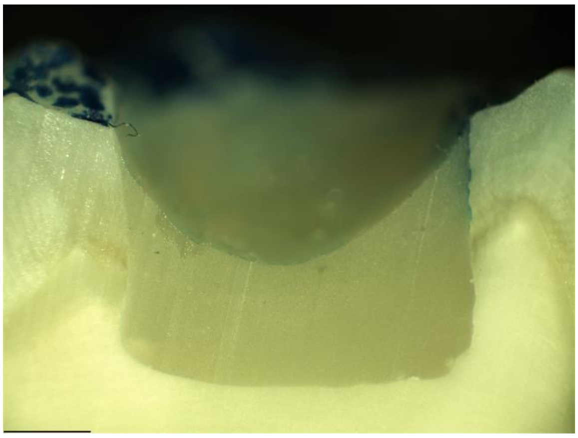

Figure 10 - Score "1" with G-Premio BOND adhesive.

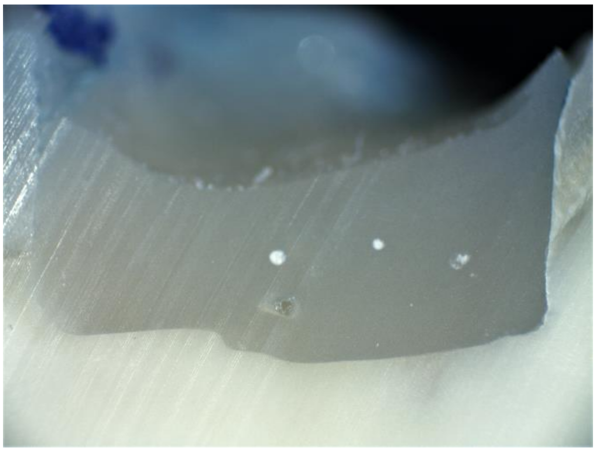

Figure 11 - Score "2" with Prime&Bond active adhesive.

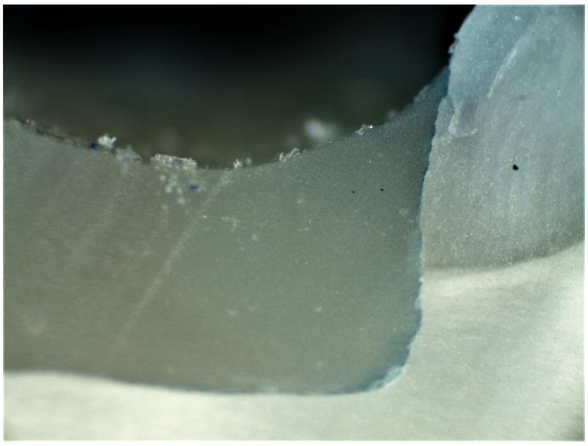

Figure 12 - Score "3" with Prime&Bond active adhesive.

Thought descriptive analysis (Figure 13), it was observed that: the most prevalent scores were “0”(No dye penetration) and “1”(Dye penetration up to half of the restoration depth), with 51,7% and 41,7% respectively; Simply four statistical individuals were reported as scores “2”(Dye penetration greater than half of the restoration depth) and “3”(Dye penetration including the pulpal floor). It is worthy of note that the four specimens included in the highest scores belong to Prime&Bond active adhesive group.

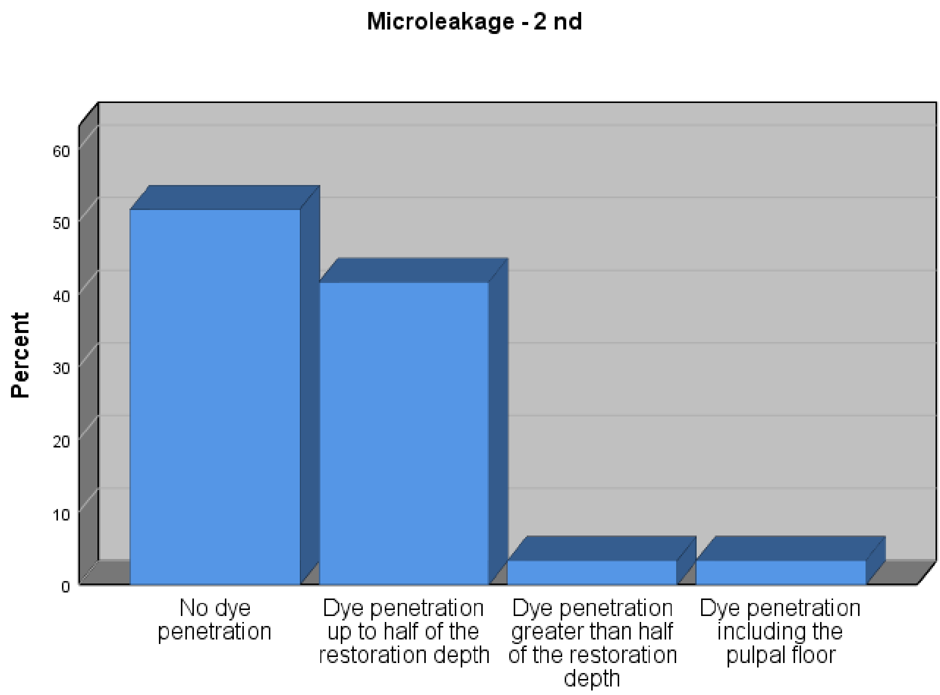

Figure 13 - Prevalence of the microleakage different scores.

Tabela III - Binomial test analysis (*p*=0,5) for the microleakage of the adhesives.

Group			Category	N	Observed Prop.	Test Prop.	*p* (2-tailed)
Prime &Bond active	Microleakage (A/P)	Group 1	Absent	9	,45	,50	,824
		Group 2	Present	11	,55		
		Total		20	1,00		
G-Premio Bond	Microleakage (A/P)	Group 1	Present	10	,50	,50	1,000
		Group 2	Absent	10	,50		
		Total		20	1,00		
Futura bond M+	Microleakage (A/P)	Group 1	Absent	12	,60	,50	,503
		Group 2	Present	8	,40		
		Total		20	1,00		

N – sample size; Observed Prop. – the proportions of the specimens with/without microleakage; Test. Prop – the probability of success; (*p*=0,5)

Binomial test (*p*=0,5) is demonstrated in Table III. For the three groups of universal adhesives, the null hypothesis is accepted. With regard to Prime&Bond active, 45% specimens without microleakage is not statistically significant counter to a probability of 0,5 (*p*=0,824). With 50% of specimens without microleakage, G-Premio Bond also does not present statistically significant differences (*p*=1,000). The same applies to Futurabond M+ with 60% of specimens (*p*=0,503).

Futurabond M+ was the adhesive that presented less specimens with marginal microleakage, while Prime&Bond active showed the highest number of specimens with dye penetration. Despite that, as the Chi-square test (χ^2) confirms (Figure 14), the null hypothesis is accepted since the differences found are not statistically significant (χ^2=0,934; df=2), *p*=0,627.

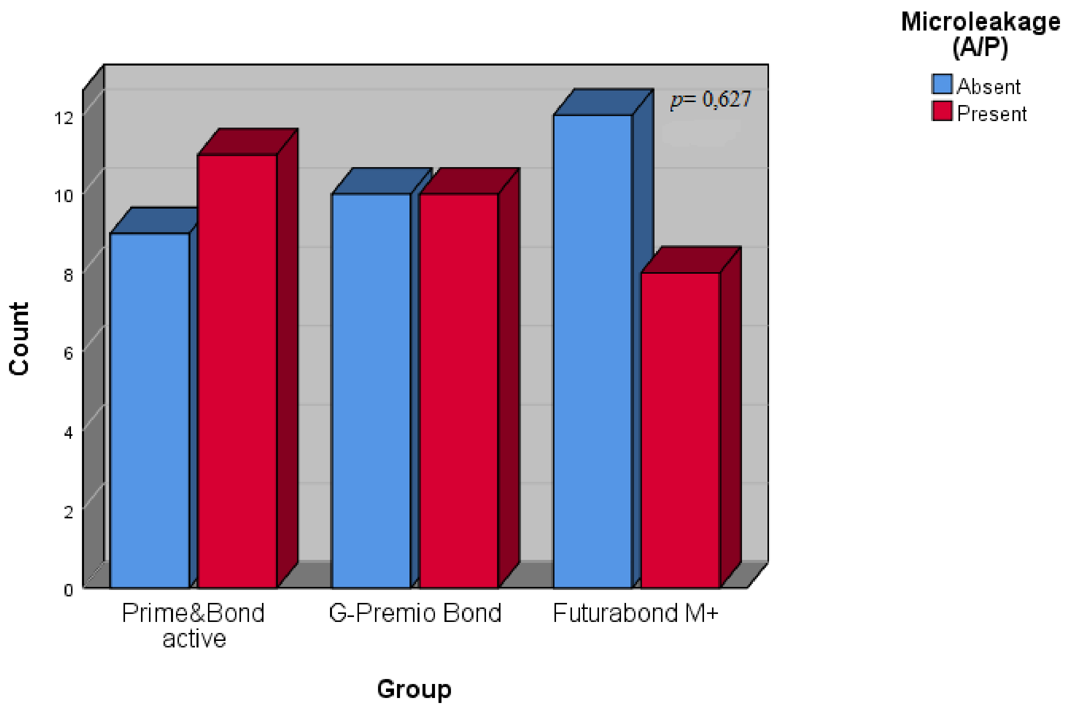

Figure 14 - Chi-square test (*p*=0,627) analysis to relate the type of adhesive with the presence/absence of microleakage.

Discussion

To achieve clinical success in adhesive dentistry, an adequate and durable bond to both dentin and enamel is mandatory.[11] Due to the increased demand for flawless restorations, it became necessary to evaluate if the new adhesive systems are effective in reducing the marginal microleakage.[12] According to few authors, the inability to maintain a reliable seal (microleakage) between tooth-restoration is mainly due to three factors: composite polymerization shrinkage; bonding surfaces (enamel/dentin/cementum); and chemical properties of the adhesive systems.[10, 13-15] On the other hand, Kalmowicz *et al*. agreed that tooth surface, namely, enamel morphology, was the most important factor regarding to microleakage of the composite resin systems utilized in their experimental study.[10] Nevertheless, it becomes pertinent to understand if there is a correlation between the adhesives composition and the restorations marginal integrity.

In order to blend hydrophilic and hydrophobic monomers, solvents are needed in the single bottle adhesives. Universal adhesive systems contain water in their composition, as it activates the ionization process of the functional monomers and at the same time improves wettability, thus increasing monomer penetration into the collagen interfibrillar spaces.[9, 16] Although water is a brilliant rewetting agent, it also has a low vapor pressure, which makes it harder to evaporate from the tooth surface. Residual solvent retention is associated with porous hybrid layer and a higher probability of water sorption. Water movement creates vulnerability at the adhesive-dentin interface, since unconverted monomers extracted from the hybrid layer can affect the integrity of the dentin-resin bond and thus decrease bond strength.[17, 18] Therefore, incorporation of organic solvents such as ethanol and acetone turn out to be imperative.[3] Acetone has a high vapor pressure, which makes it an easier solvent to evaporate and less likely to be entrapped in the bonding layer. Even so, acetone has low H-bonding power, and therefore dentin should not be over dried, which requires a more sensitive technique.[19, 20] G-Premio BOND (GB) is an acetone/water based adhesive. In the present study, GB only demonstrated microleakage at enamel margins (Figure 10), showing an effective sealing with the dentin substrate, regarding dye penetration evaluation. On the other hand, some studies concluded that GB presented poor adhesive-dentin interface, when submitted to shear bond and fatigue strength tests. The authors also noted that the interfacial phaseseparation porosities and large number of voids could be related to residual solvent entrapment due to the high content of acetone in this adhesive.[21-23] Hence, it is important to better understand, in an isolated way, the mechanism inherent to the complex dentin-solvent.

Futurabond M+ (FB) was the adhesive that presented more cases of "Absence of microleakage" (Figure 9), when compared with the other adhesives, although without statistical significance ($p > 0,05$). This result may be related to the fact that FB has in its composition the hydrophilic monomer HEMA. HEMA has the ability to increase dentin wettability and re-expand the collagen network.[17, 20] However, it is though that excess of HEMA can lead to water sorption and compromise bonding effectiveness.[18, 20] Further, FB has ethanol as co-solvent, which, due to its high solubility and osmotic pressure, assists in the exit of residual water and transports the polymerizable monomers into the dentinal tubules.[13, 17] Therefore, the latter can lead to greater adaptation of the adhesive to the tooth surface, decreasing marginal microleakage.

Prime&Bond active (PB) showed the largest number of samples with marginal microleakage. In addition, only this adhesive obtained microleakage at the dentin margins (Figures 11-12), although only four samples were infiltrated. PB is a relatively recent adhesive and so there is still not enough literature that can corroborate with this study results. PB is an isopropanol/water based adhesive. Isopropanol seems to be a completely water-soluble alcohol that provides optimal balance between wettability properties and polarity. According to Ahmed *et al*. Prime&Bond active resulted in the highest microtensile bond strength compared with G-Premio Bond and Clearfil Universal Quick (Kururay Noritake) in both etch-and-rinse and self-etch modes.[21]

In the present study, the tooth-restoration interaction was evaluated using three universal adhesives with different solvents. The interfacial characterization was obtained through qualitative analysis of the marginal microleakage. In order to allow a reliable comparison of the adhesives, all samples were equally prepared in the same conditions by a single investigator. For the above reasons, only one type of composite resin, Grandio®SO, was used. All bonded specimens were submitted to an aging process since thermal stress often occurs in the tooth-restoration interface.[24] Therefore, to simulate thermal variations that take place in the oral environment, the teeth were thermocycled in baths of 5°C and 55°C, with a dwell time of 60 seconds for 500 cycles. According to Carreira *et al*. a dwell time inferior to 50 seconds is not sufficient for the whole specimen to reach bath temperature.[25]

Universal adhesive systems appear in the market with the purpose of simplifying and reducing the number of application steps. This new adhesive systems can be used in both self-etch and etch-and-rinse modes.[1, 2, 26-28] In the current study, the three universal adhesives were applied following the self-etch approach. However, several authors agree that single-bottle adhesives are significantly more efficient when prior phosphoric acid preetching of enamel is applied. [24, 26-31] Regarding clinical trials, both Çakir *et al.* and Loguercio *et al.* concluded that there was no significant difference among bond strategies (self-etch or etch-and-rinse) when the universal adhesive was applied. Nevertheless, both authors reported, according to FDI criteria, signs of marginal degradation (marginal incompatibility or discoloration) when some of the adhesives were used in the self-etch approach, which is in agreement with what was previously mentioned regarding selective enamel etching.[1, 32] Similarly, but an *in vitro* study performed by Prya *et al.* that evaluated the effect of pre-etching enamel (3 seconds) on microleakage with universal adhesives, also demonstrated significant improvement in marginal integrity when the enamel margins were pre-etched.[29] In contrast, the dentin bonding ability is rather more complex and controversial. Some researchers pointed out that etching mode was dependent on the universal adhesive composition.[2, 28] Takamizawa *et al.* concluded that regardless of etching mode, both shear bond and fatigue strengths of the universal adhesives demonstrated equivalent bonding performance to dentin.[26] Conversely, Kermanshah *et al.* compared class V restorations microleakage using Scotchbond™ Universal (in both self-etch and etch-and-rinse modes) with Scotchbond Multi-Purpose and concluded that Scotchbond™ Universal used in self-etch approach exhibited the bests results in the dentin margins, possibly due to the present of MDP (10-methacryloxydecyl dihydrogen phosphate) in the adhesive chemical properties.[14] Furthermore, it is thought that when phosphoric acid attacks dentin hydroxyapatite (HAp) crystals, partly dissolving them, poor chemical bond between the functional monomers and the substrate can be expected.[26, 30] Therefore, in order to prevent the accidental occurrence of dentin pre-etching only in certain specimens, it was decided not to perform selective enamel etching in any case. Even though this *in vitro* study did not evaluated the different modes of adhesive application, 41,7% of the samples showed microleakage signs only at the enamel margins. These results may be in accordance with the literature referred to above, since enamel pre-etching was not performed.

Bonding performance is dependent on many factors, one of them being the chemical composition of the adhesives. Then, it is valid to affirm that adhesives with different solvents will bond differently, depending on the substrate and in which mode (2SE, SE or ER) they are applied. This *in vitro* short-term experimental study showed some limitations and to capable infer whether there was solvent influence in the microleakage, some points should have been taken into account: reduced sample, single investigator evaluating samples and reduced number of cycles in thermocycling (short aging process). Occlusal load and pulpal tests would yield more authentications, since they replicate better the environment of the oral cavity. Similarly, it would have been beneficial the use of scanning electron microscopy for a more detailed and extended characterization of the adhesive-restoration interface. Finally, further research should be carried out considering the limitations mentioned above.

Conclusion

Within the limitations of this experimental study, it can be conclude that:

There are no arguments to state that there is a relationship between the three adhesives evaluated and the presence/absence of marginal microleakage;

It can be admitted that for each adhesive, success (absence of microleakage) and failure (presence of microleakage) have the same probability of occurrence.

Different chemical properties of the adhesives, specifically solvents, had no influence on the tooth – restoration marginal microleakage.

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Anexos

Anexo 1: Parecer da Comissão de Ética da FMDUP

U. PORTO
FACULDADE DE MEDICINA DENTÁRIA
UNIVERSIDADE DO PORTO

Exmª Senhora
Patrícia João Rebelo Castela
Faculdade de Medicina Dentária da U. Porto

000131 04 FEV 2019

(CC à Orientadora Srª. Prof. Doutora Ana Portela)

Assunto: Parecer relativamente ao Projeto de Investigação nº 7/2019.
(Tooth-restauration adhesive interfacial characterization using diferente universal dental adhesives).

Informo V. Exa. que o projeto supracitado foi analisado na reunião da Comissão de Ética para a Saúde, da FMDUP, no dia 1 de fevereiro de 2019.

A Comissão de Ética é **favorável** à realização do projeto tal como apresentado.

Subject: Recommendation on the research project nº 7/2019.
(Tooth-restauration adhesive interfacial characterization using diferente universal dental adhesives).

I hereby inform that the aforementioned project was analyzed on 1st february, 2019 by the Ethics Committee for Health of the Faculty of Dental Medicine,
The Ethics Committee is **favourable** to the project execution.

Com os melhores cumprimentos,
A Presidente da Comissão de Ética para a Saúde, da FMDUP

Inês Morais Caldas

Prof. Doutora Inês Alexandra Costa Morais Caldas

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Anexo 2: Declaração de Autoria do Trabalho

U.PORTO

FACULDADE DE MEDICINA DENTÁRIA
UNIVERSIDADE DO PORTO

Declaração de Autoria do Trabalho

Monografia de Investigação/Relatório de Atividade Clínica

Declaro que o presente trabalho, no âmbito da Monografia de Investigação/Relatório de atividade, integrado no MIMD, da FMDUP, é da minha autoria e todas as fontes foram devidamente referenciadas.

Porto, 05 de julho de 2019

A Investigadora:

Patrícia João Rebelo Castela

(Patrícia João Rebelo Castela)

Anexo 3: Parecer do Orientador para entrega definitiva do trabalho apresentado

U.PORTO
FACULDADE DE
MEDICINA DENTÁRIA
UNIVERSIDADE DO PORTO

Parecer

Declaro que o Trabalho de Monografia desenvolvido pela estudante Patrícia João Rebelo Castela, do 5º ano do Curso de Mestrado Integrado em Medicina Dentária da FMDUP, subordinado ao tema: "Tooth-restoration adhesive interfacial characterization using different universal dental adhesives" está de acordo com as regras estipuladas pela FMDUP. Mais informo que o referido trabalho foi por mim conferido e se encontra em condições de ser apresentado e defendido em provas públicas.

Porto, 05 de julho de 2019

Orientadora
(Ana Isabel Pereira Portela)
Professora auxiliar da FMDUP