FACULDADE DE ODONTOLOGIA DE PIRACICABA
UNIVERSIDADE ESTADUAL DE CAMPINAS

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EFEITO DO TEMPO E TIPO DE FOTOATIVAÇÃO E DA CICLAGEM MECÂNICA E TÉRMICA NA INFILTRAÇÃO MARGINAL DE RESTAURAÇÕES ADESIVAS.

Dissertação apresentada à Faculdade de Odontologia de Piracicaba, da Universidade Estadual de Campinas, para obtenção do Título de **Mestre** em Clínica Odontológica.

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“Aprender é a única coisa de que a mente nunca se cansa, nunca tem medo e nunca se arrepende.”

Leonardo da Vinci

RESUMO

A manutenção da integridade entre dente e restauração é importante para o sucesso clínico de restaurações adesivas, influenciando a longevidade das mesmas. Assim, o objetivo deste trabalho *"in vitro"*, composto por dois experimentos, foi avaliar a infiltração marginal em cavidades Classe II: 1) confeccionadas com um sistema adesivo e compósito dental, variando a fonte de luz e o tempo de fotoativação do sistema adesivo, submetidos aos testes de ciclagem mecânica e térmica; 2) confeccionadas com um sistema adesivo e um compósito dental, variando a fonte de luz e o tempo de fotoativação da resina composta, submetidos ou não aos testes de ciclagem mecânica e térmica. Para tanto foram utilizados 270 dentes bovinos no qual foram realizadas restaurações Classe II variando o aparelho e o tempo de fotoativação, aplicado no sistema adesivo ou resina composta. Após isto, as restaurações foram submetidas ao teste infiltração marginal. De acordo com os resultados obtidos (Análise de Variância (p=0,05) e teste de Tukey) e sob as condições experimentais deste estudo, pôde-se concluir que: 1) o aumento do tempo de fotoativação do sistema adesivo não mostrou melhores resultados no processo de infiltração marginal, independente da fonte luz utilizada; 2) o aumento do tempo de fotoativação da resina composta não mostrou melhores resultados no processo de infiltração marginal, independente da fonte luz utilizada; 3) a utilização de aparelhos fotopolimerizadores com maiores intensidades de luz não mostrou melhores resultados quando comparados ao de menor intensidade, quando utilizados para

fotoativação da resina composta; 4) houve aumento da infiltração marginal após os testes de ciclagem mecânica e térmica nos grupos LED 3ª geração e halógena quando estes foram utilizados para fotoativação da resina composta.

Palavra-chave: infiltração marginal, resina composta, sistema adesivo, aparelhos fotopolimerizadores.

ABSTRACT

The maintenance of the bond integrity between tooth and resin systems is important for the clinical success of composite resin restorations. Thus, the aim of this *"in vitro*" study composed of two manuscripts, was to evaluate the microleakage of Class II dental restorations: 1) made with an adhesive system and a dental composite, varying light curing time and light curing modes of the adhesive system, submitted to the thermal and mechanical loading cycling; 2) made with an adhesive system and a dental composite, varying light curing time and light curing modes of the resin, submitted or not to the thermal and mechanical loading cycling. 270 bovine teeth were used for this study, and prepared Class II restorations varying the time and the light curing modes for the adhesive or composite resin. After that, the restorations were submitted to the microleakage test. Based on the results (Analysis of Variance (p=0,05) and Tukey test), within the experimental limits of this study, it can be concluded that: 1) the increase of the light curing time of an adhesive system did not show better results in the process of marginal microleakage, regardless the light curing modes used; 2) increasing the photoactivation time of the resin composite did not reduce the microleakage of the cervical interface of the restoration, regardless the light curing modes used; 3) use of light curing units with high irradiance did not show better results when compared with low irradiant energy, for the two first layers of resin composite; 4) the thermal

and mechanical loading cycling increased the microleakage in groups LED 3[a] generation and Conventional (QTH) for the two first layers of resin composite.

Key Word: microleakage, resin composite, adhesive system, light curing unit.

SUMÁRIO

1 INTRODUÇÃO

O uso de resinas compostas e sistemas adesivos visam atender a demanda estética, e seu preparo cavitário promove maior conservação de tecido dental sadio e, conseqüentemente, maior resistência para o elemento dental. Apesar da grande melhoria nas características estruturais desses materiais, alguns fatores indesejáveis ainda podem ser observados e podem levar a falhas nas restaurações, como coeficiente de expansão térmica diferente ao dos tecidos dentais, pigmentação superficial, desgaste, contração de polimerização (Lutz *et al*., 1986; Soh *et al*., 2004) e deformação plástica quando sujeitas às forças mastigatórias (Burgess *et al*., 2002).

A durabilidade da restauração adesiva está intimamente relacionada com a integridade da interface de união dente/material restaurador. As limitações desta restauração são clinicamente observadas através de manchamento superficial, sensibilidade pós-operatória, desenvolvimento de cáries secundárias, inflamação pulpar, que são indicativos dos sinais e sintomas da passagem de fluidos, moléculas e toxinas nas falhas produzidas entre a parede cavitária e o material restaurador (Kidd, 1976; Opdam *et al*., 1998; Van Meerbeek *et al*., 1998; Attar *et al*., 2004).

Muitos fatores podem gerar tensões elevadas sobre as margens cavitárias, contribuindo, significativamente, para a falta de integridade marginal, deterioração da união entre estrutura dentária e material restaurador e conseqüente infiltração marginal. Dentre estes estão: a contração de polimerização (Mandras *et al*., 1991; Davidson *et al*., 1984), o módulo de elasticidade do material restaurador e as condições do meio bucal que estão sujeitas às variações térmicas e mecânicas (Feilzer *et al*., 1987; Davidson & Abdalla, 1993; Da Cunha Mello *et al*., 1997; Nikaido *et al.,* 2002).

A polimerização adequada é essencial para obtenção de uma restauração com propriedades ideais (Leonard *et al.* 2002) e para a manutenção da integridade

na interface da restauração (Castro *et al*., 2004; Kubo *et al*., 2004; Ferracane & Condon, 1999). A polimerização dos compósitos ocorre devido à presença de fotoiniciadores capazes de absorver luz em determinados comprimentos de onda (Burgess *et al*., 2002). O fotoiniciador comumente usado na resina composta é a canforoquinona, que absorve energia com pico de absorbância em 468nm (Nomoto, 1997). A formação de radicais livres desencadeada pela excitação da canforoquinona, se dá através da conversão das moléculas de monômeros em uma rede de polímeros, o que causa a aproximação molecular, levando à contração dos materiais (Davidson & Feilzer, 1997). Quanto maior o número de monômeros resinosos convertidos em polímeros, melhores serão as propriedades físico-mecânicas do material formado (Feilzer *et al.,* 1995; Yoon *et al.,* 2002; Price *et al.,* 2003). Contudo, quanto maior este grau de conversão, maior será a contração volumétrica (Rueggeberg, 1999), podendo ser responsável por forte tensão na interface dente/restauração, possibilitando um rompimento de união e a formação de fendas entre o material restaurador e a estrutura dental (Davidson *et al*., 1984) podendo ocorrer, assim, a desadaptação do material na cavidade (Jorgensen *et al*., 1985).

A tensão de polimerização não está associada apenas a contração do material restaurador, mas também depende da configuração da cavidade (Fator C), técnica de inserção do material, distância da fonte fotoativadora, intensidade e o tipo da luz ativadora (Caughman *et al,*1995; Pires *et al*, 1993). As tensões resultantes de todos esses fatores são responsáveis pelo desempenho clínico da restauração.

Deficiente polimerização pode ocorrer em cavidades profundas. É comum a distância entre o sistema adesivo e o primeiro incremento de compósito com a ponta do aparelho de fotoativação ser aproximadamente 8 mm, o que reduziria a intensidade de luz que atinge a superfície do sistema adesivo ou do compósito, diminuindo o grau de conversão (Aguiar *et al.,* 2005). Segundo Prati *et al.* (1999), apenas 1mm de ar interposto entre a ponta do aparelho de fotoativação e a superfície do material resinoso reduz a intensidade de energia luminosa em

aproximadamente 10%. O sistema adesivo ou o compósito não polimerizado adequadamente, quando em contato com o meio bucal, poderá ser solubilizado, possibilitando infiltração marginal e cárie secundária (Asmussen & Peutzfeldt; 2001).

Como a distância entre a ponta do aparelho de fotoativação e a superfície do sistema adesivo ou do incremento do compósito é difícil de ser controlada, pois depende da extensão da cárie e da profundidade da cavidade após o preparo, deve-se atentar para outros fatores, como: intensidade de luz da fonte do aparelho de fotoativação, a especificidade de luz emitida pelo aparelho e tempo de fotoativação, na tentativa de se minimizar os efeitos da redução na irradiância de luz provocada pelo distanciamento da ponta do aparelho de fotoativação.

A falta de integridade marginal e conseqüente infiltração pode ser resultado direto de uma hibridização inadequada e de restaurações adesivas que não foram capazes de suportar as forças geradas durante a contração de polimerização do material restaurador (Mandras *et al.*, 1991; Davidson *et al.*, 1984).

Mesmo após a obtenção de selamento adequado, o desenvolvimento da infiltração marginal pode ocorrer após algum tempo devido ao rompimento de união causada pela degradação da interface adesiva causada por desafios químico, térmico e/ou mecânicos (Da Cunha Mello *et al*., 1997, Abdalla & Davidson, 1996). O teste de infiltração marginal é o mais usado, em estudos laboratoriais, para avaliar a integridade marginal e a habilidade de selamento dos materiais restauradores (Cardoso *et al*., 2002). Os testes de ciclagem mecânica e térmica também têm sido introduzidos para avaliar as tensões na interface união simulando situações *"in vivo" (*Da Cunha Mello *et al.*, 1997; Leibrock *et al*., 1999). Assim, o estudo dos fatores que influenciam um adequado selamento é de grande relevância para a obtenção de longevidade clínica das restaurações adesivas.

2 PROPOSIÇÃO

O objetivo deste estudo *"in vitro"*, composto por dois artigos científicos, foi avaliar a infiltração marginal em restaurações Classe II:

- confeccionadas com um sistema adesivo e um compósito dental, variando a fonte de luz e o tempo de exposição do sistema adesivo (Capítulo 1);
- confeccionadas com um sistema adesivo e um compósito dental, variando a fonte de luz e o tempo de exposição da resina composta submetidos ou não a ciclagem mecânica e térmica (Capítulo 2).

3 – CAPÍTULOS

Capítulo 1

TITLE:

Effect of light polymerization time and mode of adhesive system on the microleakage in adhesive dental restorations made with composite resin

ABSTRACT:

The aim of this *"in vitro"* study was to quantify the microleakage on restorations made with two step etch-and-rise adhesive system (Adper Single Bond 2 3M- Dental Products) and nanofilled composite resin (Filtek Z350- 3M Dental Products) after thermal and mechanical loading cycling. Ninety cavities were prepared on proximal surfaces of incisives teeth, and were randomly divided according to the photoactivation time (10, 20 or 30 seconds) and light curing modes (QTH - 450 mW/cm^2, LED 2nd generation - 1100 mW/cm^2 or LED 3rd generation - 700 mW/cm^2) of an adhesive system. Following restorative procedures and the thermal and mechanical loading cycling, the samples were immersed in methylene blue for 2 hours. The samples were ground and the powder was prepared for analysis in an absorbance spectrophotometer. All results were statistically analyzed by the ANOVA and 5% Tukey test. Results showed no significant statistical difference of microlekage between the light curing modes and light curing time. It was concluded that different curing modes and increasing the curing time were not factors that influenced the process of microleakage.

CLINICAL SIGNIFICANCE

The polymerization of the adhesive systems is desirable to decrease marginal microleakage and increase of the clinical longevity of composites restorations. However, the increase photoactivation time and the use of light curing modes to polimerizate the adhesive system did not reduce marginal microleakage in adhesive restorations submitted to thermal and mechanical cycling.

INTRODUCTION

The increasing demand for esthetic restorations in clinical dentistry has generated intensive research in adhesive materials[1]. The durability of a restoration is largely based on maintenance of an effective sealing of the tooth/restoration interface. If could be achieved, it would be possible to minimize microleakege and its consequences[2].

One of the factors that may reduce the sealing of the restoration is increasing degree of conversion of the adhesive or resin composite. The amount of light energy is the main factor for the degree of conversion of the composite and adhesive system.[3] However, it has been proven that the resin material is not totally polymerized, because it contains little amount of residual monomers among the polymeric structures formed.[4-5]As the conversion degree is related to the physical properties of that material[6], the amount of remaining monomers is a co-determining feature of the physical properties of the resulting polymer.[4]

There are many factors that may affect the amount of light energy that reaches the top and bottom surfaces of a composite increment or adhesive.[7] These factors might be: type and size of the tip of the photoactivation unit, distance from this tip to the surface of the composite, intensity and specificity of the light emitted by the unit, interaction between light wavelength and the initiator agent of

reaction polymerization, photoactivation length, composition, color, opacity and thickness of the composite or adhesive.[8-15]

If the adhesive system or the increment of the composite do not receive enough total energy for an appropriate polymerization reaction, several problems can appear determining the clinical failure of the restoration. It is common to observe marginal discoloration, secondary decay lesions and post-restorative sensibility and they contribute to the constant substitution of proximal adhesive restorations.[16-18] The location and depth of the cavity walls, orientation and density of the dentine tubules can significantly influence the adhesion to the dentine.[19-22] Besides, many factors as the thermal alterations, malocclusion, traumas, and increase in the taxes of wearing and degradation, increase of the potential cytotoxicity due to the presence of the residual monomer, decrease in the elastic modulus, weak bonding of tooth / adhesive and composite, [11, 24-26] impose tensions to the system tooth/restoration and they can affect the adhesive bonding.[27]

Among the factors that can reduce the light intensity that reaches the composite or the adhesive, the only one that cannot be controlled by the dentist while accomplishing a deep cavity restoration is the distance between the tip of the photoactivation unit and the bottom or surrounding walls of the cavity. According to Prati and others, [28] 1mm of air interposed between the tip of the photoactivation unit and the surface of the resin reduces the intensity of light energy in approximately 10%.

When in contact with the buccal environment, the adhesive system or the composite not appropriately polymerized can be solubilized, making possible marginal leakage and secondary decay.[4] The lack of marginal integrity and consequent leakage can be a direct result from an inadequate hybridizing and from adhesive systems that were not capable to support the forces generated during polymerization contraction.[29-30] Besides, the monomer liberated from the

solubilization of the adhesive system or from the composite can irritate soft adjacent tissue, stimulate the growth of bacteria, and promote allergic reactions.[31]

In the same way, if this adhesive and/or inadequately polymerized composite is in contact with the axial and pulpal walls of the cavity, the remaining monomer can cause postoperative sensibility due to its toxicity. These monomers can easily diffuse dentine tubules and affect odontoblast cells, causing loss of function and inflammatory reaction in the dental pulp, resulting in sensibility. If this process persists without any clinical action, the process can take to the pulpal necrosis.[32]

Because it is difficult to control the distance between the tip of the photoactivation unit and the surface of the adhesive system or the increment of the composite due to the extension of the decay and depth of the cavity, one should be attentive to other factors, such as: irradiance of light from the light curing unit, the light specificity emitted by the light curing unit and length of photoactivation, to avoid the radiant exposure reduction caused by the distance between the tip of the photoactivation unit. According to Xu and others[33], increasing the curing time could compensate for the loss of bond strength due to the increased curing distance. When polymerized an adhesive in deep proximal cavities, the conventional 10 second curing time is insufficient, it should be increased to compensate for the decreased bond strength owing to loss of radiant exposure[33].

The aim of this "*in vitro*" study was to measure the microleakage of Class II dental restorations made with an adhesive system and a dental composite, varying light curing time and modes of the adhesive system, submitted to the thermal and mechanical loading cycling. The tested null hypothesis was that the increase of length of polymerization and/or the use of polymerization modes with high irradiance not reduces the marginal microleakage.

METHODS AND MATERIALS

Ninety extracted bovine incisors were collected, cleaned with a periodontal curette, polished with a rubber cup and pumice paste under water and then they were stored in distilled water until they were used.

In order to enable the compressing tip of the mechanical cycling machine to touch the proximal surface of the teeth - the faces on which the restorations were made - the root portions of the bovine teeth were embedded in polystyrene resin (Piraglass, Piracicaba, SP, Brazil). The teeth had a part of their roots embedded in cold cure polystyrene resin and afterwards, were split obliquely, 10 mm from the amelodentinal proximal junction (Figure 1A and B) using a double-faced diamond disc (KG Sorensen, Barueri, SP, Brazil). After cutting, they were finishing with water abrasive papers #600, to obtain a smooth, flat incisal surface.

Specimen Preparation: Cavities were made using a diamond tip #3146 (KG Sorensen, Barueri, SP, Brazil). All the cavities were prepared with a water-cooled high-speed turbine coupled to a unit of cavity preparation on the most plane proximal, simulating Class II, measuring 8 mm height, 4mm of wide and 1,5mm deep (Figure 1C), under irrigation with air/water jet. The burs were substituted every 5 preparations. The cavities were randomly restored, following the manufacturers' instructions. The preparations were etched for 15 seconds using 35% phosphoric acid (3M ESPE, St Paul, MN, USA), then washed for 15 seconds and gently air dried to prevent excessive dentin drying. The adhesive system Adper Single Bond 2 (3M ESPE, St Paul, MN, USA) was applied in two consecutive coats, gently air dried for 10 seconds and light-polymerized was carried out in agreement with each experimental groups (Table 1). The nanofilled resin composite Filtek Z350 (3M ESPE, St Paul, MN, USA) was inserted in four horizontal increments 2mm thick each and polymerized for 20 seconds (XL 3000 – 3M – 3M ESPE, St Paul, MN, USA). After 24 hours storage in water at 37º C, the restorations were finished and polished with aluminum oxide disk Sof-Lex Pop-on

(3M ESPE, St Paul, MN, USA), in decreasing order of granulation.

Thermal and Mechanical Load Cycling: The groups were submitted to aging tests thermal cycled for 1000 times (5 ± 2º C and 55 ± 2º C) with a dwell time of 1 minute at each temperature, and transfer interval of 5 seconds. After these procedures samples were submitted to the mechanical cycling.

The samples were coupled to the mechanical cycling machine (ERIOS International, São Paulo, SP, Brazil) and submitted to 200 thousand load cycles, frequency of 1 cycle/second to receive an intermittent vertical load of 80N on the restoration. During the test, the samples were maintained in distilled water at 37ºC. *Dye Immersion:* After these procedures, the entire sample (except the interface between the restoration and the tooth) was protected with two layers of fast setting cyanoacrylate-based adhesive Superbonder (Henkel Loctite Adhesives, LTDA. Iatpevi-SP, Brazil. 06690-111). Before dye immersion, a 1mm strip of adhesive tape was placed around the area that was infiltrated (Figure 1D) and two layers of nail varnish (Figure 1E) were applied. Then, the specimens were totally immersed in 2% neutral methylene blue solution for 2 hours. After this period, the blocks were removed from dye solution, washed in running water and dried. The nail varnish was removed with the use of a periodontal curette and the dye on the restoration was removed wearing off 0,05 mm from the surface, controlled by a caliper.

Sample Trituration: To take a reading of infiltrated dye color, specimens - dental block + restoration- (Figure 1F and G) were initially split up and weighed. After the weighing the specimens were triturated in a hard tissue mill (Marconi Equip. Ltda. Piracicab-SP. Brazil, 13400) in order to obtain a powder composed of tooth/restoration and then weighed again. If the difference between the initial and final weight was higher than 10%, the specimen would be discarded. In this study, no specimen was discarded.

Dissolution: After trituration, the powder obtained from each sample was separately immersed, in test tube, containing 4 ml of absolute alcohol PA, for 24 hours, to

dissolve the dye that leaked through the tooth/restoration interface. The solution obtained was centrifuged at 3000 rpm for 3 minutes (Tomy- IC 15AN- Tomy Ind. Tokyyo, Japan), so that the powder and other elements decanted. The supernatant of the centrifuged solution was submitted to quantitative analysis of the dye present in the solution by a Spectrophotometry (DU 65, Beckman – Instruments, Inc., Fullerton – CA, USA, 92631-5) unit through absorbance reading.

The absorbance reading was taken in an adjusted unit at a wavelength of 668 nm, corresponding to the maximum absorbance of methylene blue dye. Prior to the readings, the Spectrophotometry unit had been adjusted by spectral reading with pattern solutions at the concentrations of 0.1; 0.2; 0.3; 0.5; 1; 2; 4; 6 µg / ml, to obtain the maximum spectral absorbance wavelength. Readings of the solutions were made using the wavelength value to find the maximum value of spectral absorbance. Through the ABS-concentration system, one obtains the r^2value (0.9998) and the equation of the line (y = a + bx). The following regression was obtained: Absorbance = 0.2716 x (dye Concentration) - 0.0075. From this regression, dye concentration could be calculated. A graph of lines in a Cartesian system of axes was drawn, using the values of dye concentration in micrograms per milliliters on the axis of the abscissas and the optical density obtained on the axes of the ordinates. The linear regression was obtained from Y as a function of X to determine the equation of the line, from which the concentration of dye was calculated.

RESULTS

The exploratory analysis of the data indicated that they met the presuppositions of a parametric analysis. They were also submitted to two-way ANOVA and Tukey's Test at a 5% level of significance. According to the proposed method and factors of study, the results obtained are presented in Table 2. Results showed that the increase in polymerization time and the light curing unit used were

factors not capable of interfering in the process of marginal microleakage in Class II cavities.

Considering the units used for photoactivation, there were no statistical differences among the groups, regardless the photoactivation time (p=0,2737) and of the power density in the tip of the photoactivation unit (p=2,1159). According to the data obtained, it was possible to note that there was not interaction between photoactivation X time (p=1,4919). Increasing of light curing time did not show better results in the process of marginal microleakage, regardless the light curing modes used.

DISCUSSION

Marginal microleakage is an important factor for the clinical action of restoring materials and it has been used in laboratory studies, as an indicator of the maintenance of this materials.[34] The clinical effects of the marginal microleakage are the penetration of bacteria and their sub-products in the interface tooth-restoration, contributing to the formation of secondary decays and postoperative sensibility. In this study, the dye blue methylene 2% was used for the evaluation of the technique of marginal microleakage. The whole infiltrated dye was measured by through technique spectrophotometry. In this manner, it was possible to quantify the marginal microleakage of specimens.

It has been suggested that the adhesive systems can be polymerized in an incomplete way either on the surface or within the hybrid layer.[35] In the surface, the polymerization of the adhesive is inhibited by the presence of oxygen in the buccal cavity. In the hybrid layer, other factors collaborate in a negative way for the complete polymerization, including the presence of water of the dentine, primers solvents and the incompatibilities between the components of the adhesives.[35-36] An incomplete polymerization can also be associated with the low irradiance that reaches the surface of the adhesive systems[37], due to the low radiant incidence emitted by the photoactivation unit or to the insufficient length of photoactivation.[37]

Cadenaro and others suggested the increase of the time of photoactivation to improve the polymerization and to reduce the permeability of the adhesives, and consequently, improve their clinical performance[36].

Moreover, the radiant incidence of light emitted by different units and lengths of photoactivation were analyzed in this study, in cavities 8 mm deep. Deep cavities are associated with low irradiance in the gingival and pulp walls, and consequently low polymerization quality of the polimerics materials.[14-15]

The results showed that the increase of photoactivation time of or the type of unit used was not a factor capable of interfering in the process of marginal microleakage in Class II cavities. In other words, when the length of photoactivation was doubled or tripled in relation to the manufacturer's recommendations, microleakage values were not reduced. Considering the photoactivation units used (Bluephase 16i - Vivadent, Ultralume LED 5 - Ultradent, XL 3000 - 3M Espe), there were also no statistical differences among the groups, regardless the length of photoactivation and the radiant incidence on the tip of the photoactivation unit (Table 2).

The low thickness of the hybrid layer formed and the adhesive system Single Bond 2 might be the factors that explain the equality among the experimental groups. Light irradiance emitted by the units could take to a conversion degree similar to the adhesive system, regardless the emitted value (Table 1), taking to an adhesive interface that answers in the same way of the aging tests applied – Thermocycling and Mechanic Cycling. The low thickness of the adhesive system also affects the time to reach the phase of glazing and the damage caused by oxygen the tax of conversion degree.

The adhesive systems reach the phase of glazing quickly (post gel phase), hindering the polymerization of the residual monomers and the evaporation of solvents after this period.[35] This way, the polymerization period after the vitrification phase would not bring any beneficial effect for the degree of conversion of the

adhesive system and consequently for the adhesive interface. It is estimated that the necessary time to reach this phase would be smaller than 10 second.[35]

A third factor to be considered is the thickness of the adhesive layer inhibited by oxygen. Cadenaro and others[36] suggested that simplified adhesives (primer and adhesives in the same bottle), as used in this study, are more affected by oxygen than systems that come in separated bottle the primer and the adhesive, for the smallest thickness of the first ones after the application. Besides, simplified adhesives are more hydrophilic, and they present larger difficulty of evaporation of solvents, harming the conversion degree and increasing the permeability. Cadenaro and others [36] still suggested the application of a layer of hydrophobic adhesive on these adhesives to help increase the conversion degree and to reduce the permeability. This last hypothesis can be tested, added to the factors examined in this study, in order to obtain an adhesive interface with better polymerization degree, smaller permeability and larger resistance to the degradation caused by the several forms of aging of the restoration.

According to this study, the null hypothesis was accepted; therefore it was not found difference among the groups after the increase of the light curing time of the adhesive system and of the light curing modes used.

CONCLUSION

Within the limits of this study, it could be concluded that the increase of the light curing time did not show better results in the process of marginal microleakage, regardless the light curing modes used.

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TABLE

Table1: Experimental Groups

Groups	Light Curing Unit	Polymerization mode	Irradiance* (mW/cm^2)	Irradiance* (mW/cm^2) at 8mm	Radiant Exposure** (J/cm^2)	Light Curing Time
1	Bluephase 16i	Light-emitted diodes (LED) 2º generation	1100	540	11,0	10s
2	Ultra-Lume LED5	LED 3º generation	700	350	22,0	10s
3	XL 3000	Quartz Tungsten Halogen (QTH)	450	250	33,0	10s
4	Bluephase 16i	LED 2º generation	1100	540	8,0	20s
5	Ultra-Lume LED5	LED 3º generation	700	350	16,0	20s
6	XL 3000	QTH	450	250	24,0	20s
7	Bluephase 16i	LED 2º generation	1100	540	5,0	30s
8	Ultra-Lume LED5	LED 3º generation	700	350	10,0	30s
9	XL 3000	QTH	450	250	15,0	30s

Bluephase 16i – Vivadent - Bürs-Austria A-6706; Ultralume LED 5 – Ultradent - South Jordan, USA; XL 3000 – 3M/ESPE – Grafenau Germany 28352.

* Irradiance was measured with the Demetron Radiometer - Demetron Research Corp., Model 100, Serial 105415 **(Kerr Corporation - Orange, CA 92867)**

** Irradiance calculated the incidence radiant (light intensity), considering this incidence released at the light curing tip.

Table 2: Microleakage means.

Light Curing Unit	**Light Curing Time**		
	10 seconds	20 seconds	30 seconds
LED2º generation	0.0681(0.03)Aa	0.0572(0.03)Aa	0.0524(0.02)Aa
LED3º generation	0.0420(0.02)Aa	0.0467(0.01)Aa	0.0470(0.02)Aa
Conventional	0.0432(0.01)Aa	0.0509(0.01)Aa	0.0678(0.03)Aa

Means values with the same letter were not statistically different (p>0,05) (same lower case letter were not statistically different for comparison among the light curing unit, and same upper case letter were not statistically different for comparison among different light curing time).

FIGURE

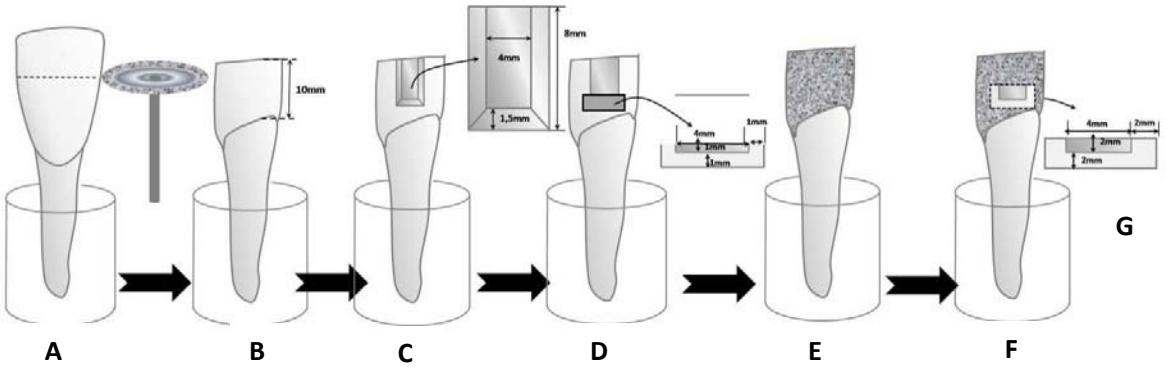

Figure 1 – Tooth sectioning with double-faced diamond disc (A); 10 mm above the amelodentinal proximal junction; after cutting, the tooth surface was planned with sandpapers #600, suffering a wear 1.0 mm (B); Class II slot preparations concluded, 1.0 mm above the amelodentinal proximal junction – proximal and occlusal-gingival directions (C); Before dye immersion, adhesive tape was placed 1mm around the area was infiltrated (D); Application of two layers nail varnish (E); After removal of the adhesive tape for further dye immersion (F); Dental block + restoration for trituration (G).

Capítulo 2

TITLE:

Effect of light polymerization time, mode and thermal and mechanical load cycling on microleakage in resin composite dental restorations.

ABSTRACT

The aim of this *"in vitro"* study was to quantify the microleakage in restorations made with Adper Single Bond 2 adhesive (3M- Dental Products) and composite resin (Filtek Z350- 3M Dental Products) with or without thermal and mechanical loading cycling. One hundred and eighty cavities were prepared on proximal surfaces of bovine teeth, and were randomly divided according to the light curing time (20, 40 or 60 seconds) and light curing modes (QTH - 450 mW/cm^2, LED 2nd generation - 1100 mW/cm^2 or LED 3rd generation - 700 mW/cm^2) of a composite resin. Afther the restorative procedures and the thermal and mechanical loading cycling for the select groups, the samples were immersed in methylene blue for 12 hours. The samples were ground and the powder was prepared for analysis in an absorbance spectrophotometer. All results were statistically analyzed by the ANOVA and Tukey 5% test. Results showed that the increase in polymerization time and the unit used were factors not capable of interfering in the process of marginal microleakage in Class II cavities. Considering the units used for photoactivation, there were also no statistical differences among the groups. Thermal and mechanical loading cycling showed effect on microleakage in groups LED 3 e QTH.

CLINICAL SIGNIFICANCE

In deeper cavities, it is essential to supervise proximal restorations periodically to monitor their clinical success. An increase in polymerization time and the use of light curing units with higher irradiances did not reduce the cervical microleakage of the restorations. Simulated mechanical and thermal stresses increased microleakage in Class II restorations.

INTRODUCTION

With the increasing demand for esthetics, new adhesive restorative materials have been developed. These materials are able to achieve the natural color and characteristics of teeth.[1] Among these materials, resin composite has presented undeniable technological development. Despite the great improvement in its structural characteristics, some undesirable factors are still observed, such as the coefficient of thermal expansion differing from that of the dental structure, superficial pigmentation, wearing off, and polymerization shrinkage.[2]

Appropriate polymerization is essential in order to produce a restoration with ideal properties[3] and maintain the integrity of the restoration interface.[4-6] Composite polymerization occurs due to the presence of photoinitiators capable of absorbing light at certain wavelengths.[7] In the case of resins, the most commonly used photoinitiator is camphoroquinone that absorbs energy with an absorbance peak at 468nm.[8] The formation of free radicals during the excitation of camphoroquinone, takes place by the conversion of molecule monomers in a polymeric network, causing molecular approach and this leads to material shrinkage. [9]The larger the number of resinous monomers converted in polymers, the better the physical-mechanical properties of the material formed; [10-12] however, the greater this degree of conversion, the greater the volumetric shrinkage.[13] This may cause strong tension at the tooth/restoration interface and possibly bond breakage causing gaps in the restorative material, and disruption between the

dental structure [14] and material in the cavity.[15] The result of stress due to shrinkage may contribute to the failure of resin composite restorations, breaking the bond between the tooth/restoration, microleakage, postoperative sensitivity and secondary caries. [10;16-20]

Polymerization shrinkage stress is not only associated with the adhesive system and the restorative material. It also depends on the cavity configuration (C Factor), material insertion technique, resin composition, particle size, distance from the curing light source, intensity and light polymerization modes. [21-22] The stresses resulting from all of these factors are responsible for the clinical success of the restoration as far as microleakage is concerned.

Deficient polymerization can occur in deeper cavities due to the dispersion of light energy as a result of the distance between the tip of the light curing appliance and the first resin composite increment.[23-25] In a deep Class II cavity the interface between the tooth structure and the first increment of resin composite may be not well polymerized. The exposure of this interface to the oral environment can result in restoration fracture, as well as solubility of the resin composite and adhesive, leading to microleakage and secondary caries.[26]

Even after controlling the effects of the polymerization shrinkage, marginal microleakage may occur through the rupture caused by chemical, thermal or mechanical stress at the adhesive interface.[27-28] Laboratory studies have mostly used the microleakage test to evaluate the marginal integrity and the sealing ability of restorative materials.[29] Mechanical and thermal cycling tests have also been proposed to evaluate the stress at the bond interface in order to simulate the "*in vivo*" situation.[27; 30]

The aim of this "*in vitro*" study was to measure microleakage of Class II dental restorations made with an adhesive system and a dental composite, with different light polymerization times and light curing modes of the resin, submitted to the thermal and mechanical load cycling, or not. Thus, the tested hypotheses were:

1- increasing the photoactivation time of the resin composite would reduce the microleakage at the cervical interface of the restoration; 2- using light curing units with high irradiance would reduce the microleakage at the cervical interface, mainly after thermal and mechanical load cycling; 3- and the thermal and mechanical load cycling would increasing the microleakage in Class II cavities.

METHODS AND MATERIALS

One hundred and eighty bovine incisors were cleaned with a periodontal curette to remove organic debris, and polished with a rubber cup and pumice paste under water. Afterwards, they were stored in distilled water until they were used.

To enable the compressing tip of the mechanical cycling machine to touch the proximal surfaces of the teeth - the faces on which the restorations were made - the root portions of the bovine teeth were embedded in polystyrene resin (Piraglass, Piracicaba, SP, Brazil). The teeth had part of their roots embedded in cold cure polystyrene resin, and afterwards, were split obliquely, 10 mm from the amelodentinal proximal junction (Figure 1A and B) using a double-faced diamond disc (KG Sorensen, Barueri, SP, Brazil). After cutting, they were finished with water abrasive papers #600, to obtain a smooth, flat incisal surface.

Specimen Preparation: Cavities were made using a diamond tip #3146 (KG Sorensen, Barueri, SP, Brazil) coupled to a cavity preparation unit on the flattest proximal surface, simulating Class II, measuring 8 mm, 4mm wide and 1.5mm deep (Figure 1C), under irrigation with air/water jet. The burs were replaced after every five preparations. The cavities were randomly restored, following the manufacturers' instructions. The preparations were etched for 15 seconds using 35% phosphoric acid (3M ESPE Products, St Paul, MN, USA), then washed for 15 seconds and gently air dried to prevent excessive dentin drying. The adhesive system Adper Single Bond 2 (3M ESPE Products, St Paul, MN, USA) was applied in two consecutive coats, lightly air dried for 10 seconds and light-polymerized (XL

3000 –3M – Espe) for 10 seconds. The nanofilled resin composite Filtek Z350 (3M ESPE Products, St Paul, MN, USA) was inserted in four horizontal increments 2mm thick each. The first two increments were polymerized according to each experimental group (Table 1) and the last two increments were polymerized for 20 seconds. After 24 hours storage at 37ºC, the restorations were finished and polished with aluminum oxide disks Sof-Lex Pop-on (3M ESPE, St Paul, MN, USA), in decreasing order of granulation.

Thermal and Mechanical Load Cycling: The groups that were submitted to aging tests were thermal cycled 1000 times (5 ± 2^{o} C and 55 ± 2^{o} C) with a dwell time of 30 seconds each at each temperature, and transfer interval of 5 seconds. After these procedures samples were submitted to mechanical cycling.

The samples were coupled to the mechanical cycling machine (ERIOS International, São Paulo, SP, Brazil) and submitted to 200 thousand load cycles, frequency of 1 cycle/second to receive an incidence of an intermittent vertical load of 80N on the restoration. During the test, the samples were submerged in distilled water at 37ºC.

Dye Immersion: After these procedures, the entire sample (except the interface between the restoration and the tooth) was protected with two layers of fast setting cyanoacrylate-based adhesive Superbonder (Loctite Adesivos, LTDA). Before dye immersion, a 1mm strip of adhesive tape was placed around the area that was infiltrated (Figure 1D) and two layers of nail varnish (Figure 1E) were applied. Then the specimens were totally immersed in 2% neutral methylene blue solution for 12 hours. After this period, the blocks were removed from dye solution, washed under running water and dried. The nail varnish was removed with the use of a periodontal curette and the dye on the restoration was worn off 0.05 mm from the surface, controlled by a caliper.

Sample Trituration: To take a reading of the infiltrated dye color, specimens (dental block + restoration) (Figure 1F and G) were initially split up and weighed.

After the weighing the specimens were triturated in a hard fabric grinder in order to obtain a powder composed of tooth/restoration and then weighed again. If the difference between the initial and final weight was higher than 10%, the specimen would be discarded. In this study, no specimen was discarded.

Dissolution: After trituration, the powder obtained from each sample was separately immersed in a test tube containing 4 ml of absolute alcohol PA, for 24 hours, to dissolve the dye that leaked through the tooth/restoration interface. The solution obtained was centrifuged at 3000 rpm for 3 minutes, so that the powder and other elements decanted. The supernatant of the centrifuged solution was submitted to quantitative analysis of the dye present in the solution by a Spectrophotometry (DU 65, Beckman) unit through absorbance reading.

The absorbance reading was taken in an adjusted unit at a wavelength of 668 nm, corresponding to the maximum absorbance of methylene blue dye. Prior to the readings, the Spectrophotometry unit had been adjusted by spectral reading with pattern solutions at the concentrations of 0.1; 0.2; 0.3; 0.5; 1; 2; 4; 6 µg / ml, to obtain the maximum spectral absorbance wavelength. Readings of the solutions were made using the wavelength value to find the maximum value of spectral absorbance. Through the ABS-concentration system, one obtains the r^2 value (0.9998) and the equation of the line (y = a + bx). The following regression was obtained: Absorbance = 0.2716 x (dye Concentration) - 0.0075. From this regression, dye concentration could be calculated. A graph of lines in a Cartesian system of axes was drawn, using the values of dye concentration in micrograms per milliliters on the axis of the abscissas and the optical density obtained on the axes of the ordinates. The linear regression was obtained from Y as a function of X to determine the equation of the line, from which the concentration of dye was calculated.

RESULTS

The exploratory analysis of the data indicated that they met the presuppositions of a parametric analysis. They were also submitted to three-way ANOVA and Tukey's Test at a 5% level of significance. According to the proposed method and to the factors studied, the results obtained are presented in Tables 2 and 3. According to the data obtained, it was possible to note that there was interaction between cycling X photoactivation (p=0.0094) and between polymerization X time (p=0.0106). There was no triple interaction among the studied factors cycling X photoactivation X time (p=0.3812).

The results showed that the increase in photoactivation time did not decrease the microleakage in all experimental groups (Table 2). For LED 3, the 60 seconds groups showed higher microleakage means than the 20 second groups. For LED 2 and QTH, there were no differences between the 20, 40 and 60 -second groups. When light curing units were compared (Table 3), QTH showed lower microleakage means than LED 2, when photoactivated for 20 seconds, without thermal and mechanical load cycling. For 60 seconds, QTH showed lower microleakage means than LED 3, for the groups with or without thermal and mechanical load cycling. The groups submitted to thermal and mechanical load cycling showed higher microleakage than those that were not submitted to thermal and mechanical load cycling in Groups LED 3 and QTH, for all photoactivation times.

DISCUSSION

The distance of the light guide tip from the resin composite was capable to affect the polymerization of the composite resin. [24-25] Therefore, it was expected that the increase of the radiation exposure (using higher photoactivation time or irradiance) would improve the degree of conversion of the resin composite, and

consequently the bond strength of the resin composite in the deeper walls of cavity. However, based on the results, the first and second hypotheses were almost completely rejected. Microleakage mean values were equal or increased with the increase of the photoactivation time or with the use of light curing units with high irradiance. Two factors must be considered to explain these results: how polymerization stress takes place and what is the quality of the polymer formed after photoactivation.

The first factor points to the fact that the top surface presents higher polymerization rates than the bottom surface of the increment, when photoactivation was performed with the light tip positioned distant from the top surface of the sample.[24-25; 31] Aguiar and others[25] showed that, even with a distance of 8mm from the tip of the light curing unit, the top surface receives adequate irradiant energy. Therefore, on the top surface, high irradiance photoactivation initiated a multitude of growth centers of polymers with higher cross-linking density. [32-33] Low irradiant energy reached the bottom surface, [24-25;31;34] which was in contact with the cervical wall, decreasing the degree of conversion and cross-linking density.[34] The high polymerization rate on the top surface and the lower polymerization rate on the bottom surface leads to higher bond strength around the walls and lower bond strength on the cervical walls. More polymers bonding the resin composite and the adhesive will be formed around the walls that are nearer to the light curing tip than on the cervical wall.

Adapting Versluis and others[35] theory, it is possible that better polymerization on the top creates a stress flow of polymerization shrinkage from the cervical walls to the walls around the cavity - buccal and lingual-, which opens a gap in the cervical interface.

Groups with lower radiant exposure (less photoactivation time or irradiance) tend to decrease stress created by the polymerization shrinkage. This was performed with the LED 3 group for 20 seconds of photoactivation time compared

with 60 seconds of photoactivation time, and in the QTH group compared with LED 2 (20 seconds without thermal and loading cycling) and LED 3 (60 seconds with or without thermal and loading cycling). In this situation, less polymer centers will grow on the top surface, leading to a more linear polymeric structure in all the composite resin increment. Thus, lower irradiance will delay the increase of viscosity of the composite resin during polymerization, [36-37] allowing more conversion of monomers[37] and decreasing polymerization stress.

The quality of the polymer formed after photoactivation may also affect the microleakage means. Linear polymers with low cross-linking density present low elastic modulus, and it is related to the capacity of relieve the stress from polymerization shrinkage.[38] Therefore, in the groups mentioned above, the lower cross-linking may be associated with lower microleakage.

The third hypothesis studied was partially accepted. Thermal and mechanical load cycling significantly increased microleakage for groups LED 3 and QTH, in all photoactivation time studied. Thermal cycling induces stress and degradation to the interface due to the difference between tooth structures and restorative materials.[39-40] Mechanical load cycling produces stress in the composite resin, and it is transmitted to the bonding interface.[41] Interfaces less affected by microleakage - before the aging test- were more susceptible to thermal and load cycling.

However, for LED 2, there was no statistical difference between groups with or without thermal and mechanical load cycling. LED 2 presents the characteristics of having a narrow spectral range with a peak around 470nm, which matches the optimum absorption wavelength for the activation of the camphorquinone photoinitiator, [42-43] and high irradiance (1100 mW/cm^2). Therefore, high polymerization rates were developed in early photoactivation, mainly on the top surface, and they are associated with low conversion in the deeper layer of the increment due to the limited diffusion of polymerization at earlier stages.[36-37] As a

result, the bottom surface of the increment probably showed lower polymerization with the adhesive applied in the cervical wall, opening a gap in this interface, before any aging test. In this situation, thermal and mechanical load cycling did not show a significant effect of greater deterioration on the interface with gaps opened. Therefore, after the aging test, the LED 2 group did not show any statistical difference from the other light curing unit groups.

CONCLUSION

Within the limits of this study, it could be concluded:

1) Increase the photo-activation time of the resin composite did not reduce the microleakage of the cervical interface of the restoration, regardless the light curing modes used;

2) Use of light curing units with high irradiance did not reduce the microleakage of the cervical interface;

3) Thermal and mechanical loading cycling increased the microleakage in groups LED 3 e QTH.

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TABLE

Table1: Experimental Groups

Groups	Light Curing Unit	Polymerization mode	Irradiance* (mW/cm^2)	Irradiance** (mW/cm^2) at 6mm	Light Curing Time
1/10	Bluephase 16i	Light-emitted diodes (LED) 2º generation	1100	600	20s
2/11	Ultra-Lume LED5	LED 3º generation	700	400	20s
3/12	XL 3000	Quartz Tungsten Halogen (QTH)	450	280	20s
4/13	Bluephase 16i	LED 2º generation	1100	600	40s
5/14	Ultra-Lume LED5	LED 3º generation	700	400	40s
6/15	XL 3000	QTH	450	280	40s
7/16	Bluephase 16i	LED 2º generation	1100	600	60s
8/17	Ultra-Lume LED5	LED 3º generation	700	400	60s
9/18	XL 3000	QTH	450	280	60s

Groups 10 to 18 were submit to thermal and mechanical loading cycling (**Bluephase 16i – Vivadent - Bürs-Austria A-6706; Ultralume LED 5 – Ultradent - South Jordan, USA; XL 3000 – 3M/ESPE – Grafenau Germany 28352).**

* Irradiance was measured with the Demetron Radiometer - Demetron Research Corp., Model 100, Serial 105415 **(Kerr Corporation - Orange, CA 92867)**

**Irradiance calculated the incidence radiant (light intensity), considering this incidence released at the light curing tip.

Table 2: Microleakage means.

Light Curing Unit	Light Curing Time	Mechanical and Thermal Cycling	
		(-)	(+)
LED 2	20s	0.060 (0.03) Aa	0.064(0.05) Aa
	40s	0.050 (0.01) Aa	0.053 (0.02) Aa
	60s	0.046 (0.02) Aa	0.065 (0.04) Aa
LED 3	20s	0.036 (0.01) Bb	0.053 (0.02) Ab
	40s	0.047 (0.01) Bab	0.075 (0.04) Aab
	60s	0.055 (0.02) Ba	0.090 (0.05) Aa
QTH	20s	0.031 (0.01) Ba	0.079 (0.06) Aa
	40s	0.039 (0.01) Ba	0.056 (0.02) Aa
	60s	0.035 (0.01) Ba	0.048 (0.01) Aa

Means values with the same letter were not statistically different (p>0,05) (same lower case letter were not statistically different for comparison among the light curing unit, and same upper case letter were not statistically different for comparison among mechanical and thermal cycling).

Table 3: Microleakage means.

Light Curing Time	Light Curing Unit	Mechanical and Thermal Cycling	
		(-)	(+)
20s	LED 2	0.060 (0.03) a	0.064 (0.05) a
	LED 3	0.036 (0.01) ab	0.053 (0.02) a
	QTH	0.031 (0.01) b	0.079 (0.06) a
40s	LED 2	0.050 (0,01) a	0.053 (0.02) a
	LED 3	0.047 (0.01) a	0.075 (0.04) a
	QTH	0.034 (0.01) a	0.056 (0.02) a
60s	LED 2	0.046 (0.02) ab	0.065 (0.04)ab
	LED 3	0.055 (0.02) a	0.090 (0.05)a
	QTH	0.035 (0.01) b	0.049 (0.01)b

Means values with the same letter were not statistically different (p>0,05) (same lower case letter were not statistically different for comparison among the light curing unit).

FIGURE

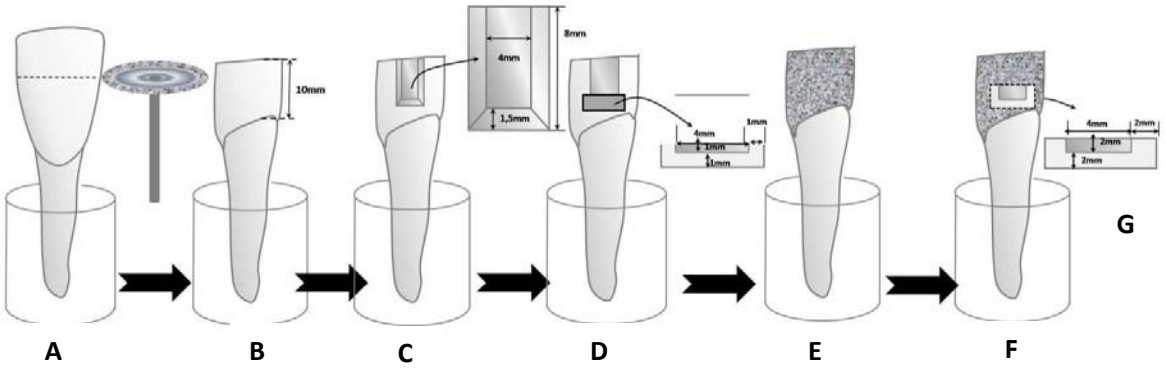

Figure 1 – Tooth sectioning with double-faced diamond disc (A); 10 mm above the amelodentinal proximal junction; after cutting, the tooth surface was planned with sandpapers #600, suffering a wear 1.0 mm (B); Class II slot preparations concluded, 1.0 mm above the amelodentinal proximal junction – proximal and occlusal-gingival directions (C); Before dye immersion, adhesive tape was placed 1mm around the area was infiltrated (D); Application of two layers nail varnish (E); After removal of the adhesive tape for further dye immersion (F); Dental block + restoration for trituration (G).

4 - CONSIDERAÇÕES GERAIS

O insucesso nas margens de restaurações Classe II em resina composta é um evento freqüente na prática clínica. Sensibilidade, manchamentos e lesões de cárie recorrentes são fatores que contribuem para a substituição destas restaurações adesivas. Estudos têm demonstrado divergências sobre a influência das fontes de luz ou das técnicas de fotoativação utilizadas para polimerização de materiais adesivos para o selamento marginal das restaurações.

A contração volumétrica é uma das características da resina composta durante o processo de polimerização. Durante a fotoativação, os monômeros são convertidos em uma rede de polímeros, reduzindo os espaços intermoleculares, e aumentando a contração. A maior parte das tensões de contração ocorre durante o período inicial de polimerização, e vai diminuindo com o tempo (Choi *et al.,* 2004). Se a tensão de polimerização gerada em uma cavidade exceder a resistência da união, falhas na interface dente-restauração são produzidas (Davidson *et al.*, 1984; Choi *et al.*, 2004; Nikolaenko *et al.*, 2004). Se uma pequena falha da adesão ocorrer durante a contração de polimerização, a retenção da resina composta pode não ser significativamente afetada, mas o vedamento da restauração possivelmente será prejudicado, causando conseqüências clínicas indesejáveis. A formação de fendas marginais pode possibilitar a entrada de fluidos orais e produtos bacterianos (Carvalho *et al.*, 1996).

A infiltração marginal é um fator importante para o desempenho clínico de materiais restauradores e tem sido utilizada em diversos estudos laboratoriais, como um indicador da manutenção destes materiais (Taylor & Lingh, 1992). Os efeitos clínicos da infiltração marginal são a penetração de bactérias e seus subprodutos na interface dente-restauração, contribuindo para a formação de cáries secundárias e sensibilidade pós-operatória. Neste estudo, foi utilizado o corante azul de metileno 2% para a avaliação da técnica de infiltração marginal.

Todo o corante infiltrado foi medido pela técnica espectrofotometria. Desta forma, foi possível quantificar a infiltração marginal dos espécimes.

No Capítulo 1 verificou-se que o aumento do tempo de fotoativação ou o tipo de aparelho utilizado não foram fatores capazes de interferir no processo de infiltração marginal de cavidades Classe II. Mesmo quando o tempo de polimerização foi dobrado ou triplicado em relação ao que recomenda o fabricante do sistema adesivo, os valores de infiltração marginal não foram reduzidos. Isto se pode dever ao fato de que quando se fotoativa um incremento de resina composta de 2mm de espessura a luz emitida pelo aparelho fotopolimerizador pode atravessar essa camada de resina e atingir também o adesivo que está localizado logo abaixo, sendo assim uma maior quantidade de luz irá atingir a camada de adesivo. A baixa espessura da camada híbrida formada do sistema adesivo Single Bond (Arrais & Giannini, 2002) pode ser um dos fatores que explicam a igualdade entre os grupos experimentais.

Os sistemas adesivos atingem rapidamente a fase de vitrificação, dificultando a polimerização dos monômeros residuais e a evaporação dos solventes após este período (Nunes *et al.,* 2005). Desta forma, o período de polimerização após a fase de vitrificação não traria nenhum efeito benéfico para o grau de conversão do sistema adesivo e conseqüentemente para a interface adesiva. Calcula-se que o tempo necessário para se atingir esta fase seria menor que 10 segundos (Nunes *et al.,* 2005).

Tipos de aparelhos fotopolimerizadores, suas intensidades e seu tempo de polimerização são importantes fatores que influenciam o grau de conversão de um material adesivo. Sabe-se que maior intensidade de luz pode resultar em adequado grau de conversão (Knezevic *et al.* 2007). Porém a alta intensidade também pode aumentar a contração de polimerização (Demoli *et al*, 2004). Isto foi encontrado no Capítulo 2, onde se pode observar que a infiltração marginal foi igual ou maior, com o aumento do tempo de fotoativação ou com o uso de aparelhos de alta irradiância.

A fotoativação, com emissão de altas intensidades de luz, parece promover um rápido aumento da viscosidade, limitando o escoamento e interferindo na acomodação do material às paredes cavitárias (Goracci *et al*., 1996). Tem sido demonstrado experimentalmente que uma lenta reação de polimerização das resinas compostas, pode causar menos danos à interface da restauração, aumentar o escoamento e diminuir o estresse de contração de polimerização (Mehl *et al*., 1997). Isto pode ser obtido através da baixa intensidade de luz, sem, no entanto, haver comprometimento da polimerização do material (Unterbrink & Muessner, 1995; Mehl *et al*., 1997).

Os grupos com menor tempo fotoativação ou irradiância tendem a diminuir a tensão criada pela contração de polimerização. Isto foi observado no grupo LED 3, com 20 segundos de fotoativação, em comparação com 60 segundos, e no grupo QTH comparado com LED 2 (20 segundos sem ciclagem térmica e mecânica) e LED 3 (60 segundos com ou sem ciclagens). Nesta situação observa-se a formação de uma estrutura mais linear em todo o compósito, assim, uma menor irradiância irá diminuir a velocidade de reação de polimerização da resina composta (Atai & Watts, 2008; Pfeifer *et al*., 2008), permitindo uma maior conversão dos monômeros (Pfeifer *et al*., 2008) e diminuindo a contração de polimerização.

Ciclagens térmicas e mecânicas são usadas em estudos "*in vitro"* para simular o processo natural de envelhecimento das restaurações dentro da cavidade bucal (Cardoso *et al.,* 2002; Mitsui *et al*., 2006). Nikaido *et al*., 2002, relataram que a fadiga cíclica pode diminuir a resistência da união dente/resina. Segundo os autores, a deformação da restauração é um possível acontecimento durante os ciclos térmicos e mecânicos, a qual pode criar micro separações entre as paredes cavitárias e o sistema adesivo. No Capítulo 2 onde os grupos foram submetidos ou não aos testes de ciclagens térmica e mecânica, pôde-se observar que aumentaram significativamente a microinfiltração para os grupos LED 3 e QTH, para todos os tempos estudados.

Para o grupo LED 2, não houve diferença estatística entre os grupos com ou sem ciclagem térmica e mecânica. LED 2 apresenta a característica de ter uma estreita faixa espectral com pico em torno de 470nm, que corresponde a máxima absorção de onda para a ativação do fotoiniciador canforoquinona (Emani & Söderholm, 2003; Tsai & Meyers & Walsh, 2003). Além disso, o LED 2 apresentou maior irradiância (1100 mW/cm^2), quando comparado ao LED 3 (800 mW/cm^2). Assim, altas taxas de polimerização foram desenvolvidas no início da fotoativação. Essas altas taxas estão associadas à baixa conversão devido a difusão limitada da polimerização no seu estágio inicial (Atai & Watts, 2008; Pfeifer *et al.*, 2008). Como resultado, a superfície inferior do incremento provavelmente apresentou menor polimerização, abrindo uma lacuna nesta interface, antes de qualquer teste de envelhecimento. Nesta situação, as ciclagens térmica e mecânica não mostraram um efeito significativo.

Diante da coletânea e análise dos resultados obtidos neste estudo composto por dois capítulos, pôde se observar que as fontes de luz e tempos de fotoativação, podem interferir diretamente na infiltração marginal de restaurações adesivas.

5- CONCLUSÃO GERAL

De acordo com as condições experimentais e com base nos resultados obtidos neste estudo pôde-se concluir que:

1) o aumento do tempo de fotoativação do sistema adesivo não mostrou melhores resultados no processo de infiltração marginal no sistema adesivo estudado, independente da fonte luz utilizada;

2) o aumento do tempo de fotoativação da resina composta não mostrou melhores resultados no processo de infiltração marginal, independente da fonte luz utilizada;

3) a utilização de aparelhos fotopolimerizadores com maiores intensidades de luz e especificidade não mostraram melhores resultados quando comparados ao de menor intensidade, quando utilizados para fotoativação dos primeiros incrementos de resina composta;

4) houve aumento da infiltração marginal após os testes de ciclagem mecânica e térmica nos grupos LED 3ª geração e halógena, quando estes foram utilizados para fotoativação dos primeiros incrementos de resina composta.

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APÊNDICE

Anexo 1: MATERIAIS E MÉTODOS

PREPARO DOS DENTES:

Para a presente pesquisa, foram utilizados 270 dentes incisivos bovinos (Figura 1). Os dentes foram submetidos à raspagem manual com cureta periodontal para remoção de debris orgânicos, e polidos com taça de borracha e pasta de pedra pomes com água. Caso o dente apresentasse alguma rugosidade nas superfícies proximais, estas sofreram um pequeno acabamento com lixas d'água n° 600. Em seguida, esses dentes foram armazenados em água destilada até o momento da sua utilização.

Figura 1: Dentes bovinos

Os dentes foram seccionados transversalmente, a uma distância de 10 mm da junção amelo-cementário proximal, com o auxílio de discos diamantados dupla face (Figura 2A), e polidos com lixa d'água n° 600 para se obter assim uma superfície plana incisal (Figura 2B). Através de uma ponta diamantada # 3146 acoplada à máquina de preparo cavitário, foram realizados preparos, simulando

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Classe II, com 8 mm de altura, 4mm de largura e 1,5mm de profundidade, sob irrigação com spray de ar/água (Figura 3 A e B). A cada 5 preparos, a ponta foi substituída por outra nova.

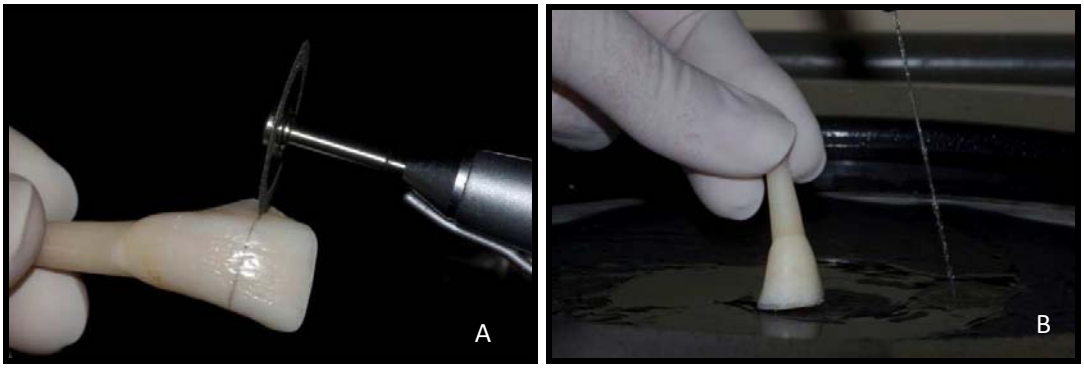

Figura 2: A- Seccionamento dos dentes, a uma distância de 10mm da junção amelo-cementária proximal, com o auxílio de discos diamantados dupla face. B- Desgaste de 1mm com lixa d'água n° 600 para a planificação da superfície.

Figura 3: A- Esquema das dimensões do preparo cavitário. B- Confecção da cavidade.

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INCLUSÃO DAS RAÍZES

A fim de se proporcionar o posicionamento dos dentes na máquina de ciclagem mecânica, a porção radicular dos dentes bovinos foi incluída em resina de poliestireno (*Piraglass, Piracicaba, SP, Brasil*). As raízes foram posicionadas próximas a um dos lados no interior de tubos de PVC de 2,1cm de diâmetro e, aproximadamente 2 cm de altura, os quais se encontravam fixados em uma placa de cera utilidade (Figura 4A), de modo que a superfície proximal de cada dente ficasse voltada ao centro do tubo. Esse posicionamento teve como objetivo possibilitar que a ponta compressora da máquina de ciclagem mecânica estabelecesse contato com a superfície proximal dos dentes, ou seja, na face onde serão realizadas as restaurações em resina composta. A resina de poliestireno foi preparada na proporção de 2% (100g de resina para 2g de catalisador) e vertida no interior dos tubos de PVC. Após um período de aproximadamente seis horas, a resina já se encontrava polimerizada e os dentes puderam ser removidos dos anéis de PVC (Figura 4B).

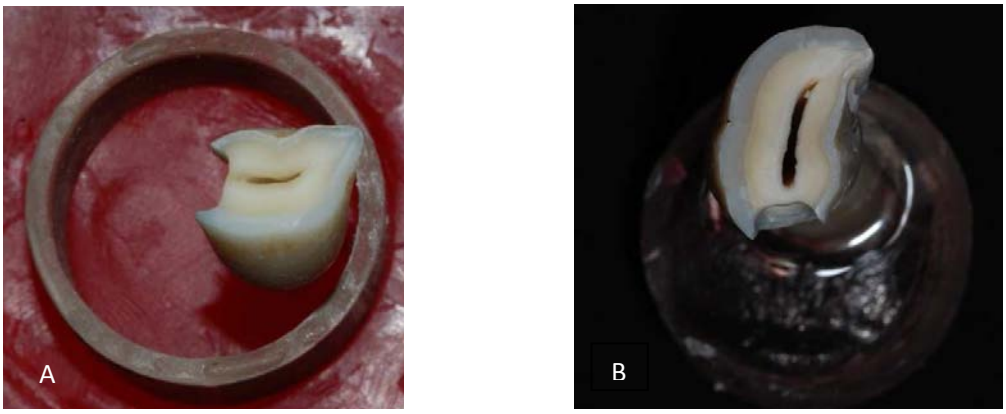

Figura 4: Inclusão dos dentes em resina de poliestireno.

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RESTAURAÇÕES:

Cada cavidade foi lavada abundantemente com água e secada com papel absorvente, evitando o ressecamento da dentina. Em seguida as cavidades foram restauradas em seqüência aleatorizada, seguindo as instruções dos fabricantes, detalhadas através do seguinte protocolo:

A cavidade foi condicionada por 15 segundos com ácido fosfórico a 35% (ESPE, St Paul, MN, USA**)** (Figura 5 A e B), seguida de lavagem por 15 segundos (Figura 5C) e secagem com papel absorvente, evitando ressecar a dentina. ADPER SINGLE BOND 2 (Figura 6 A, B e C) foi aplicado em duas camadas e a última polimerizada de acordo com os grupos experimentais para o Capítulo 1 (fotoativador **Bluephase 16i** –Vivadent com irradiância de luz de 1100 mW/cm^2, **Ultra-Lume LED 5** –Utradent com intensidade de luz de 700 mW/cm^2, ou **XL 3000** –3M - Espe com irradiância de luz de 450 mW/cm^2 - Figura 7 A,B e C) e por 10 segundos (**XL 3000** –3M - Espe com irradiância de luz de 450 mW/cm^2) para o Capítulo 2. A resina composta Filtek Z350 foi inserida em quatro incrementos horizontais, tendo cada 2mm de espessura (Figura 8). Cada incremento foi polimerizado por 20 segundos para o Capítulo 1 (**XL 3000** –3M - Espe com intensidade de luz de 450 mW/cm^2) e de acordo com cada grupo experimental para o Capítulo 2 fotoativador **Bluephase 16i** –Vivadent com irradiância de luz de 1100 mW/cm^2, **Ultra-Lume LED 5** –Utradent com irradiância de luz de 700 mW/cm^2, ou **XL 3000** –3M - Espe com irradiância de luz de 450 mW/cm^2 (Figura 7 A,B e C).

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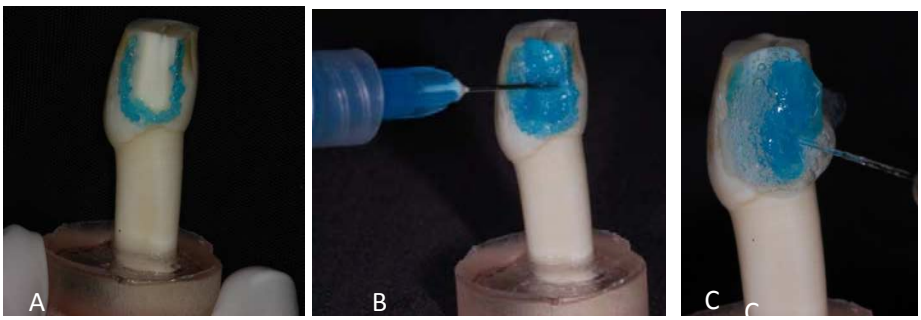

Figura 5: A- Condicionamento ácido em esmalte. B- Condicionamento ácido em dentina. C- Lavagem por 15 segundos.

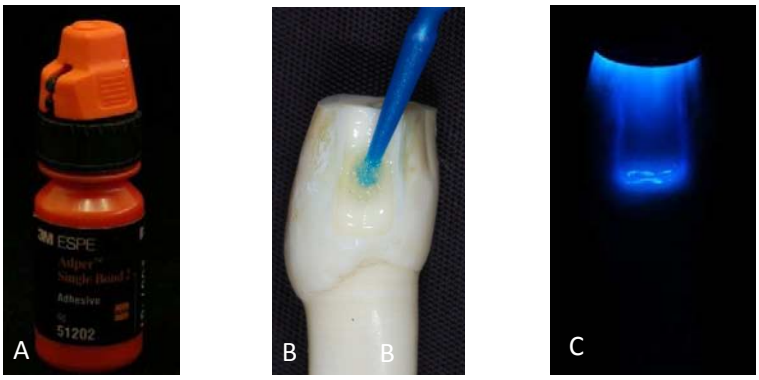

Figura 6: A- Adesivo Adper Single Bond 2. B- Aplicação do adesivo. C- Fotopolierização do adesivo.

Figura 7: A- Bluephase 16i **–Vivadent com intensidade de luz de 1600 mW/cm^2.** B- Ultra-Lume LED 5 **–Ultradent com intensidade de luz de 800 mW/cm^2.** C- XL 3000 **–3M - Espe com intensidade de luz de 500 mW/cm^2**.

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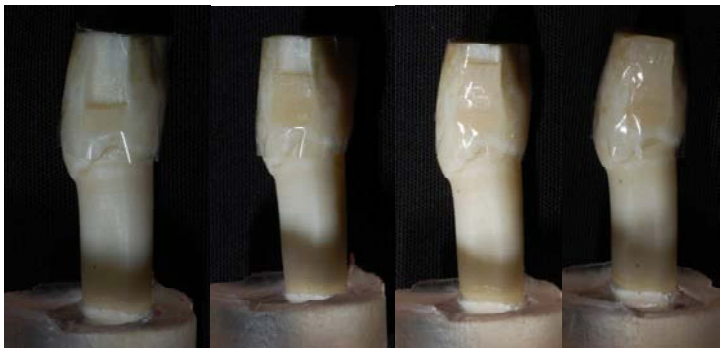

Figura 8: Resina composta inserida em 4 incrementos de 2mm.

Após 24 horas, foram realizados acabamento e polimento das restaurações, utilizando-se disco de óxido de alumínio Sof-Lex, em ordem decrescente de granulação.

CICLAGEM TÉRMICA E CICLAGEM MECÂNICA:

Para o Capítulo 1, os grupos foram termociclados com 1000 ciclos de temperatura em água destilada entre 5 ± 2 °C e 55 ± 2 °C, com 1 minuto de permanência em cada banho, e 5 segundos de intervalo de transferência. Após estes procedimentos, as amostras foram submetidas à ciclagem mecânica.

Para o Capítulo 2, os grupos foram termociclados com 1000 ciclos de temperatura em água destilada entre 5 ± 2 °C e 55 ± 2 °C, com 30 segundos de permanência em cada banho, e 5 segundos de intervalo de transferência. Após estes procedimentos, as amostras foram submetidas à ciclagem mecânica.

Após a termociclagem, as amostras foram submetidas a 200 mil ciclos mecânicos, com freqüência de 1 ciclo/segundo e força axial intermitente de 80N. Durante o teste, as amostras estiveram imersas em água destilada a 37ºC.

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IMERSÃO EM CORANTE:

Após estes procedimentos, toda a amostra, com exceção da interface entre a restauração e o dente, foi protegida com duas camadas de adesivo a base de cianoacrilato de presa rápida Superbonder (Loctite Adesivos, LTDA). Antes da imersão em corante foi colocada uma fita crepe adesiva ao redor da área a ser infiltrada (Figura 9A) em seguida foi aplicada duas camadas de esmalte para unha (Figura 9B e C). Então, os espécimes foram totalmente imersos em solução neutra de azul de metileno a 2% (Figura 9D). Decorrido este período, os blocos foram removidos da solução corante, lavados em água corrente, secados e o bloco dental removido do cilindro de poliestireno. Com o objetivo de remover o corante depositado na superfície da restauração, o corpo de prova sofreu um desgaste superficial de 0,05 mm, controlado por um paquímetro.

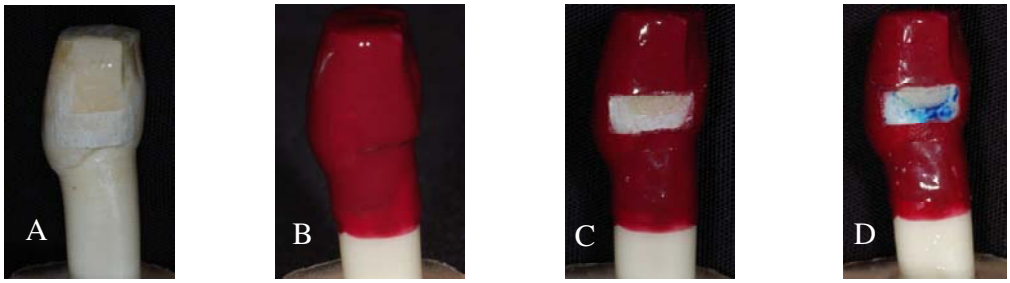

Figura 9: A- Colocação da fita crepe adesiva. B- Aplicação de duas camadas de esmalte para unhas. C- Após remoção da fita adesiva. D- Após imersão em corante.

TRITURAÇÃO DAS AMOSTRAS:

Para preparar a leitura de corante infiltrado, os corpos de prova (bloco dental + restauração) foram pesados e triturados em moinho para tecidos duros, com o objetivo de obter um pó composto pelo conjunto dente/restauração e

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pesado novamente. Se a diferença entre os pesos, inicial e final, fossem maior que 10%, a amostra seria descartada. Neste estudo nenhuma amostra foi descartada.

DISSOLUÇÃO:

Após a trituração, o pó obtido de cada amostra foi imerso, separadamente, em tubo de ensaio, contendo 4 ml de álcool absoluto PA, por 24 horas, para dissolver o corante infiltrado na interface dente/restauração. A seguir, a solução obtida pelas amostras trituradas foi centrifugada em centrífuga regulada para 3000 rpm por 3 minutos, para que o pó e eventuais impurezas fossem decantados. O sobrenadante da solução centrifugada foi submetido à análise quantitativa de corante presente na solução pelo aparelho de espectrofotometria por meio da leitura de absorbância.

Para a leitura da absorbância, o aparelho foi ajustado com um comprimento de onda adequado para o corante azul de metileno correspondente a absorbância máxima de corante. Para tanto, anteriormente às leituras, o aparelho de espectrofotometria foi calibrado, realizando-se uma varredura espectral, utilizando-se de soluções padrão nas concentrações de 0,1; 0,2; 0,3; 0.5; 1; 2; 4; 6 μg / ml, para se obter o comprimento de onda de máxima absorbância espectral. Utilizando-se desse valor de comprimento de onda, foi realizada a leitura das soluções obtendo-se o valor máximo de absorbância espectral. Através do sistema ABS-Concentração, obtém o valor de r^2 e a equação da reta.

Foi traçado um gráfico de linhas em um sistema de eixos cartesianos, utilizando os valores de concentração de corante em microgramas por mililitros no eixo das abscissas e a densidade óptica obtida nos eixos das ordenadas. Foi obtida a regressão linear de y em função de x e determinada a equação da reta, a partir da qual pôde ser calculada a concentração de corante.

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