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Thaís Diniz Segatto

Influência do nível de bateria de unidades LED sem fio na intensidade de luz emitida e nas características de cimento resinoso dual utilizado para cimentação de retentores reforçados por fibra de vidro

Influence of battery level of cordless LED units on the emitted light and characteristics of a dual cure resin cement used for luting fiber posts.

Dissertação apresentada à Faculdade de Odontologia da Universidade Federal de Uberlândia, para obtenção do Título de Mestre em Odontologia na Área de Clínica Odontológica Integrada.

Uberlândia, 2019

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Orientador: Prof. Dr. Luís Henrique Araújo Raposo
Co-orientador: Prof. Dr. Carlos José Soares

Banca examinadora:
Prof. Dr. Luís Henrique Araújo Raposo
Profa. Dra. Veridiana Resende Novais Simamoto
Prof. Dr. Hugo Lemes Carlo

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UNIVERSIDADE FEDERAL DE UBERLÂNDIA
Coordenação do Programa de Pós-Graduação em Odontologia
Av. Pará, 1720, Bloco 4L, Anexo B, Sala 35 - Bairro Umuarama, Uberlândia-MG, CEP 38400-902
Telefone: (34) 3225-8115/8108 - www.ppgoufu.com - copod@umuarama.ufu.br

ATA

Ata da defesa de DISSERTAÇÃO DE MESTRADO junto ao Programa de Pós-graduação em Odontologia da Faculdade de Odontologia da Universidade Federal de Uberlândia.

Defesa de: Dissertação de Mestrado - COPOD

Data: 25/02/2019

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Título do Trabalho: ***Influência do nível de bateria de unidades LED sem fio na intensidade de luz emitida e nas características de cimento resinoso dual utilizado para cimentação de retentores reforçados por fibra de vidro.***

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As **oito horas** do dia **vinte e cinco de fevereiro de 2019** no Anfiteatro do Bloco 4T, Campus Umuarama, da Universidade Federal de Uberlândia, reuniu-se a Banca Examinadora, designada pelo Colegiado do Programa de Pós-graduação em janeiro de 2019, assim composta: Professores Doutores: Veridiana Resende Novais Simamoto (UFU); Hugo Lemes Carlo (UFJF); e o orientador(a) do(a) candidato(a): **Luís Henrique Araújo Raposo.**

Iniciando os trabalhos o(a) presidente da mesa **Dr. Luís Henrique Araújo Raposo** apresentou a Comissão Examinadora e o candidato(a), agradeceu a presença do público, e concedeu ao Discente a palavra para a exposição do seu trabalho. A duração da apresentação do Discente e o tempo de argüição e resposta foram conforme as normas do Programa.

A seguir o senhor(a) presidente concedeu a palavra, pela ordem sucessivamente, aos (às) examinadore (as), que passaram a argüir o(a) candidato(a). Finalizada a argüição, que se desenvolveu dentro dos termos regimentais, a Banca, em sessão secreta, atribuiu os conceitos finais.

Em face do resultado obtido, a Banca Examinadora considerou o(a) candidato(a) (A)provado(a).

Esta defesa de Dissertação de Mestrado é parte dos requisitos necessários à obtenção do título de Mestre. O competente diploma será expedido após cumprimento dos demais requisitos, conforme as normas do Programa, a legislação pertinente e a regulamentação interna da UFU.

Nada mais havendo a tratar foram encerrados os trabalhos às 10 horas e 35 minutos. Foi lavrada a presente ata que após lida e achada conforme foi assinada eletronicamente pela Banca Examinadora.

Documento assinado eletronicamente por **Luis Henrique Araujo Raposo**, **Professor(a) do Magistério Superior**, em 25/02/2019, às 10:43, conforme horário oficial de Brasília, com fundamento no art. 6º, § 1º, do Decreto nº 8.539, de 8 de outubro de 2015.

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DEDICATÓRIA

Dedico esse trabalho aos meus queridos pais, Lillyan e Junior, minha base e estrutura essencial para todos os momentos de minha vida, minha fonte inesgotável de amor, carinho, força e estímulo.

Meu amor e carinho por vocês são inigualáveis e incondicionais. Sou grata por ter vocês em minha vida e dedico as minhas conquistas a vocês.

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“Nada é por acaso, toda situação guarda um propósito de crescimento!”

(Lucas Mahat)

SUMÁRIO

RESUMO

O objetivo deste estudo foi avaliar a influência do nível de bateria de unidades LED sem fio na intensidade de luz emitida e nas características de um cimento resinoso dual usado para cimentação de retentores reforçados por fibra de vidro. Primeiramente, a intensidade de luz das unidades LED sem fio (Bluephase G2 e Radii Xspert) foram verificadas em diferentes porcentagens de bateria (100%, 50% e 10%). Foram extraídos 60 incisivos bovinos, realizada a instrumentação dos canais radiculares e alívio dos canais. As raízes foram distribuídas aleatoriamente nos grupos experimentais (n=10) para a realização da cimentação dos retentores reforçados por fibra de vidro com cimento resinoso autoadesivo dual de acordo com os diferentes níveis de bateria e unidades LED. Esses espécimes foram seccionados em cortadeira de precisão, resultando em 2 discos para cada terço da raiz para serem submetidos ao teste de resistência de união por micro push-out (RU) e para análise do modo de falha. Foram confeccionados espécimes de cimento resinoso (n=5) para avaliação do grau de conversão (GC), simulando a influência de diferentes níveis de bateria de unidades LED no cimento resinoso nos diferentes terços radiculares. Os resultados de RU e GC foram analisados usando ANOVA 2-way (unidade LED e nível de bateria) com múltiplas repetições (terços radiculares) seguido de teste Tukey HSD (α=0,05). Foi observado que houve diminuição da intensidade de luz das unidades LED com a redução dos níveis de bateria. ANOVA mostrou influência significativa para todos os fatores isolados: unidade LED ($P = 0,003$), nível de bateria ($P < 0,001$) e terços ($P < 0,001$); e também para interação entre unidade e terços radiculares ($P = 0,013$). Não houve diferença para a interação entre unidade e bateria ($P = 0,173$), bateria e terço ($P = 0,954$), unidade, bateria e terço ($P = 0,829$). Os valores de RU foram significativamente menores para o terço apical para todos os grupos, ao contrário do terço cervical que apresentou os maiores valores. Os níveis de bateria 100% e 50% mostraram valores de RU significativamente mais altos do que o nível de bateria 10% para ambos os aparelhos em todos os terços. O modo de falha mais frequentemente observados nos espécimes foi a falha mista. Os resultados do GC não apresentaram diferenças significativas para todos os fatores e para interação entre eles, e a porcentagem do GC ultrapassou 60% para todas as situações. Pode-se concluir que a intensidade de luz emitida pelas unidades LED pode ser afetada pelo baixo nível de bateria das mesmas. Apesar de não ter influenciado no grau de conversão, afetou na resistência de união do cimento resinoso dual usado para fixação de retentores intraradiculares reforçados por fibra de vidro nos diferentes terços radiculares. A unidade LED polywave testada apresentou melhor desempenho para a resistência de união em todas as variáveis.

Palavras chave: cimento resinoso, nível de bateria, retentor reforçado por fibra de vidro, unidades LED sem fio.

ABSTRACT

The aim of this study was to investigate the influence of the battery level of different cordless LED units on the emitted light and characteristics of a dual cure resin cement used for luting fiber reinforced posts**.** First, the light characteristics of the two cordless LED units (Bluephase G2 e Radii Xspert) were checked for all light-curing cycles of the dfferent battery levels. Sixty bovine root canals were endodontically treated, the post spaces were prepared and the roots were randomly divided into the experimental groups (n=10). After surface treatment, the posts were cemented with a self-adhesive dual-cure resin cement, light-cured with different battery levels (100%; 50%; 10%) for the both LED units. Then, roots were sectioned and two disks were obtained from each root third to be submitted to the push-out bond strength test (PBS) and failure pattern analysis. Resin cement specimens (n=5) were obtained to evaluate degree of conversion (DC), simulating the influence of the distinct battery levels and LED units on the resin cement at the different root thirds. The PBS and DC data were analysed using a 2-way ANOVA (light curing unit and battery level) with repeated measures (root canal region) followed by Tukey HSD test (α=.05). The light intensity of the LED units decreased according to reduced battery levels. ANOVA showed significant influence for the all isolated factors: light curing unit ($P = 0.003$), battery level ($P < 0.001$) and root region ($P < 0.001$); and also for interaction between the light curing unit and root region factors ($P = 0.013$). No difference was found for interaction between light curing unit and battery level ($P = 0.173$), battery level and root region ($P = 0.954$), light curing unit, battery level and root region ($P = 0.829$). The root depth reduced significantly the PBS values for all the tested groups. The cervical region showed the highest PBS values while the apical region had the lowest values. The 100% and 50% battery levels showed significant higher PBS values than the 10% level for both LED units. Mixed failure mode was predominantly found for all groups. The DC showed no significant differences for all the factors, with the resin cement exceeding 60% for all the situations. The light characteristics of cordless LED units can be affected by low battery levels, and although the degree of conversion was not affected for this factor, it has influenced the bond strength. Cordless LED units should be kept with 50%-100% battery levels in order to achieve better properties for resin cements, thus reducing the risk of retention loss for glass fiber posts. The polywave light curing unit tested resulted in better performance for the bond strength in all variables.

Key words: battery level, cordless LED unit, fiberglass reinforced, resin cement.

INTRODUÇÃO E REFERENCIAL TEÓRICO

Retentores intraradiculares reforçados por fibra de vidro podem ser fixados usando diferentes cimentos resinosos (Mosharraf & Zare, 2014). Os cimentos resinosos podem ter sua reação de polimerização ativada quimicamente (autopolimerização), por ação de luz (fotopolimerização) ou por dupla ativação (dual) (Tonello et al., 2016). Cimentos resinosos ativados apenas pela luz tem uso limitado em situações em que a energia luminosa não tem condições de alcançar o material polimérico como na cimentação de retentores intraradiculares, por isto nestes casos é indicado o emprego dos cimentos resinosos químicos ou de dupla-ativação (Aguiar et al., 2015).

Na fotoativação, a formação do polímero ocorre após a sensibilização dos fotoiniciadores, sendo a canforoquinona o mais comumente utilizado em polímeros odontológicos, que em conjunto com o co-iniciador (amina alifática aromática) geram radicais livres quando irradiadas por luz na região azul-violeta do espectro visível (comprimento de onda de 470 nm) (Aguiar et al., 2015). A polimerização induzida quimicamente, apresenta-se necessariamente com pelo menos dois reagentes (um ativador e um iniciador) que quando em contato, sofrem reação química e geram radicais livres. O iniciador, peróxido de benzoíla, é responsável pelo início da reação de polimerização pois quando energizado apresenta radical livre que reage com as moléculas dos monômeros para o crescimento da cadeia. A amina terciária, age como ativador pois sua presença no meio reduz a energia térmica necessária para quebrar o iniciador em radicais livres na temperatura ambiente (D'Alpino et al., 2015). Nos materiais que possuem dupla ativação, os dois mecanismos citados previamente estão associados (D'Alpino et al., 2015).

Os dois modos de ativação dos cimentos resinosos duais são suplementares e independentes e iniciam uma dinâmica de formação de radicais livres e conversão de monômeros que naturalmente se sobrepõem durante o processo de polimerização. Idealmente trata-se de um material com tempo de trabalho adequado e capaz de atingir adequado grau de conversão de seus monômeros com menores intensidades ou mesmo na ausência de luz (Tonello et al., 2016). A condição esperada seria que ambas reações se complementassem proporcionando adequada polimerização, mas a fotoativação prematura desses materiais pode dificultar a movimentação dos radicais livres e a posterior reação de polimerização química, restringindo o cimento de obter as propriedades almejadas (Tonello et al., 2016).

Evidências mostram limitações no procedimento de cimentação de retentores reforçados por fibra de vidro que pode levar a falhas na adesão entre os compósitos resinosos, os pinos e a dentina radicular (Maroulakos et al., 2018). Fatores relacionados a morfologia do tecido dentinário, materiais usados para obturação endodôntica e microinfiltrações podem afetar a retenção dos pinos reforçados por fibra à dentina radicular, devendo se ter cuidadosa seleção do tratamento de superfície, irrigantes e adesivos para obter adesão efetiva entre as estruturas envolvidas (Pereira et al., 2014). Além disso, a reação de polimerização do cimento pode ser influenciada por fatores como composição, conteúdo de partículas inorgânicas, tipo de monômero utilizado e fatores extrínsecos como temperatura, luz do fotopolimerizador, tempo de polimerização e densidade de energia (Maroulakos et al., 2018). A fotoativação também pode ser influenciada pelo espectro de emissão da luz, tempo de exposição, fotoiniciadores, co-iniciadores, estabilizadores, inibidores, bem como tipos, proporções dos monômeros e partículas de carga (Wegehaupt et al., 2017).

As unidades fotoativadoras e sua intensidade de luz podem afetar diretamente o tratamento restaurador odontológico (Ribeiro et al., 2016). Diodos emissores de luz (LEDs) têm sido frequentemente empregados como fontes de irradiação em unidades fotoativadoras na prática odontológica para fotoativação de compósitos resinosos. Dentre as principais vantagens, estão a menor emissão de calor e custo de manutenção, combinado com maior durabilidade (Weig et al., 2018). A fonte de luz ideal deve ter nível de irradiação capaz de ativar adequadamente o material resinoso no menor tempo possível (Magalhães et al, 2015). Também é considerado importante a forma de distribuição do feixe de energia pela ponteira, já que a irradiância e potência da luz das unidades LED dependem do modelo/fornecedor, pois a distribuição de energia não é necessariamente uniforme em toda a área do feixe (Filho et al., 2018).

Devido às limitações da primeira e segunda geração de LEDs, uma terceira geração foi desenvolvida, conhecida como unidades *polywave*, as quais emitem luz com comprimento de onda variando entre 380 e 515 nm (luz ultravioleta a azul) ao contrário das unidades LED de segunda geração que emitem luz com comprimento de onda mais estreito, entre 410 e 470 nm, (luz azul) (Micheud et al., 2014). As unidades *polywave* são capazes de fornecer melhor fotoativação quando são usados compósitos resinosos que contem fotoiniciadores alternativos, como por exemplo fotoiniciadores a base de germânio (Ivocerin) ou Lucerin-TPO (Oliveira et al., 2017). Mesmo assim, a quantidade de energia entregue para as camadas mais distantes do composto resinoso pode ser influenciada por fatores como reatividade do fotoiniciador, dispersão de luz e falta de uniformidade do feixe de luz emitido (Oliveira et al., 2017)

Teoricamente, quanto maior a intensidade da luz e/ou maior o tempo de exposição, maior a densidade de energia será entregue ao material resinoso e consequentemente maior será o grau de conversão do mesmo (Wegehaupt et al., 2017). A profundidade ou espessura das restaurações pode limitar a conversão em regiões afastadas da superfície onde a luz é emitida (Anathana et al., 2017), fato esse que pode acontecer durante a cimentação de retentores intraradiculares reforçados por fibra de vidro. Existe correlação entre a intensidade de luz eficaz disponível para a fotopolimerização de monômeros de resina e a distância entre a fonte de luz da unidade fotoativadora e a superfície do material, segundo a qual a intensidade da luz pode diminuir proporcionalmente ao quadrado da distância da área de emissão (Anathana et al., 2017). Isso significa que, quando se dobra a distância entre a fonte de luz da unidade fotoativadora e a superfície do material, o resultado é a redução da intensidade da luz a um quarto da intensidade original (Wegehaupt et al., 2017).

No tratamento restaurador com retentores reforçados por fibra de vidro, a localização da fonte de luz emitida pela unidade fotoativadora fica muitas vezes posicionada distante do cimento resinoso (Pereira et al., 2016). Levando em consideração que o aumento da distância entre a fonte de luz e a superfície do material promove redução na densidade de energia, podendo afetar o grau de conversão do compósito, e que o nível de bateria de unidades LED sem fio pode afetar a intensidade de luz emitida pelas mesmas (Pereira et al., 2016), é valido investigar se o tratamento restaurador com retentores reforçados por fibra de vidro pode ser afetado por esses fatores.

PROPOSIÇÃO

Objetivo geral:

Avaliar a influência do nível de bateria de unidades LED sem fio na intensidade de luz emitida pelas mesmas e resistência de união e grau de conversão de um cimento resinoso autoadesivo de dupla ativação empregado na cimentação de retentores intraradiculares reforçados por fibra de vidro à dentina radicular.

Objetivos específicos:

1. Avaliar as características de luz emitidas pelas unidades LEDs sem fio *poliwave* (Bluephase G2, Ivoclar Vivadent) e *monowave* (Radii Xspert, SDI) em diferentes níveis de bateria (100%, 50% e 10%);

2. Analisar o efeito dos diferentes níveis de bateria das unidades LED sem fio na resistência de união de retentores reforçados por fibra de vidro à dentina radicular cimentados com cimento resinoso autoadesivo de dupla ativação nos diferentes terços radiculares (cervical, médio e apical);

3. Analisar os modos de falha ocorridos entre o retentor, cimento resinoso e dentina radicular nos espécimes após o teste de resistência de união;

4. Determinar o grau de conversão do cimento resinoso autoadesivo de dupla ativação utilizado de acordo com as unidades LED, diferentes níveis de bateria e terços radiculares.

CAPÍTULO 1

Influence of battery level of cordless LED units on the emitted light and characteristics of a dual cure resin cement used for luting fiber posts.

Authors: Segatto TD, Braga SS, Lopes CC, Tavares LN, Novais VR, Soares PV, Soares CJ, Raposo LH

Thaís Diniz Segatto, DDS, Master student, Department of Occlusion, Fixed Prosthodontics and Dental Materials, School of Dentistry, Federal University of Uberlândia, Uberlândia, Brazil

Stella Sueli Lourenço Braga, DDS, MSc, PhD student, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Uberlândia, Brazil

Camila de Carvalho Almança Lopes, DDS, MSc, PhD student, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Uberlândia, Brazil

Lucas do Nascimento Tavares, DDS, MSc, PhD student, Department of Occlusion, Fixed Prosthodontics and Dental Materials, School of Dentistry, Federal University of Uberlândia, Uberlândia, Brazil

Veridiana Resende Novais, DS, MSc, PhD, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Uberlândia, Brazil

Paulo Vinícius Soares, DDS, MSc, PhD, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Uberlândia, Brazil

Carlos José Soares, DDS, MSc, PhD, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Uberlândia, Brazil

*Luís Henrique Araújo Raposo, DDS, MSc, PhD, Department of Occlusion, Fixed Prosthodontics and Dental Materials, School of Dentistry, Federal University of Uberlandia, Uberlândia, Brazil

Corresponding authors:
Av. Pará, 1720, Bloco 4L, Campus Umuarama Uberlândia - MG, Brazil - CEP: 38400-902 E-mail: thais.segatto@ufu.br
Av. Pará, 1720, Bloco 4L, Campus Umuarama Uberlândia - MG, Brazil - CEP: 38400-902 E-mail: stellabraga@ufu.br
Av. Pará, 1720, Bloco 4L, Sala 4LA42, Campus Umuarama Uberlândia - MG, Brazil - CEP: 38400-902 E-mail: camilacalopes@ufu.br
Av. Pará, 1720, Bloco 4L, Sala 4LA32, Campus Umuarama Uberlândia - MG, Brazil - CEP: 38400-902 E-mail: lucasnascimentotavares@hotmail.com
Av. Pará, 1720, Bloco 4L, Sala 4LA32, Campus Umuarama Uberlândia - MG, Brazil - CEP: 38400-902 E-mail: paulovsoares@yahoo.com.br
Av. Pará, 1720, Bloco 4L, Sala 4LA32, Campus Umuarama Uberlândia - MG, Brazil - CEP: 38400-902 E-mail: carlosjsoares@ufu.br
*Av. Pará, 1720, Bloco 4L, Sala 4LA42, Campus Umuarama Uberlândia - MG, Brazil - CEP: 38400-902 E-mail: raposo@ufu.br

CLINICAL RELEVANCE

Cordless LED units should be maintained with 50-100% battery levels in order to ensure adequate light output reaching dual cure resin cements while luting glass fiber posts, thus reducing the risk of retention loss.

SUMMARY

Objective: The aim of this study was to investigate the influence of the battery level of different cordless LED units on the emitted light and characteristics of a dual cure resin cement used for luting fiber reinforced posts. **Materials and Methods**: First, the light characteristics of the two cordless LED units (Bluephase G2 e Radii Xspert) were checked for all light-curing cycles of the dfferent battery levels. Sixty bovine root canals were endodontically treated, the post spaces were prepared and the roots were randomly divided into the experimental groups according to the LED units and distinct battery levels (n=10).

After surface treatment, the posts were cemented with a self-adhesive dual-cure resin cement, light-cured with different battery levels (100%; 50%; 10%) for the both LED units. Then, roots were sectioned and two disks were obtained from each root third to be submitted to the push-out bond strength test (PBS) and failure pattern analysis. Resin cement specimens (n=5) were obtained to evaluate degree of conversion (DC), simulating the influence of the distinct battery levels and LED units on the resin cement at the different root thirds. The PBS and DC data were analysed using a 2-way ANOVA (light curing unit and battery level) with repeated measures (root canal region) followed by Tukey HSD test (α=.05). **Results:** The light intensity of the LED units decreased according to reduced battery levels. ANOVA showed significant influence for the all isolated factors: light curing unit (P = 0.003), battery level (P < 0.001) and root region (P < 0.001); and also for interaction between the light curing unit and root region factors (P = 0.013). No difference was found for interaction between light curing unit and battery level (P = 0.173), battery level and root region (P = 0.954), light curing unit, battery level and root region (P = 0.829). The root depth reduced significantly the PBS values for all the tested groups. The cervical region showed the highest PBS values while the apical region had the lowest values. The 100% and 50% battery levels showed significant higher PBS values than the 10% level for both LED units. Mixed failure mode was predominantly found for all groups. The DC showed no significant differences for all the factors, with the resin cement exceeding 60% for all the situations. **Conclusion:** The light characteristics of cordless LED units can be affected by low battery levels, and although the degree of conversion was not affected for this factor, it has influenced the bond strength. Cordless LED units should be kept with 50%-100% battery levels in order to achieve better properties for resin cements, thus reducing the risk of retention loss for glass fiber posts. The polywave light curing unit tested resulted in better performance for the bond strength in all variables.

Key words: battery level, cordless LED unit, glass fiber post, resin cement.

INTRODUCTION

The successful rehabilitation of endodontically treated teeth requires the evaluation of factors such as reminiscent tooth structure, periodontal condition of the supporting tissues and restoration aesthetics[1].Fiber Posts are often indicated in full crown restorations of non-vital teeth in order to promote retention and stability for the future restoration.[2] High success rates are reported for cases treated using glass fiber posts, due to characteristics such as elastic modulus (about 20 GPa) that is similar to the root dentin elastic modulus (about 18 GPa).[1] Thus, when using glass fiber posts, uniform stress distribution is observed along the root, reducing the risk of unfavorable root fractures.[2,3] Also, glass fiber posts have aesthetic advantages, improved corrosion resistance, biocompatibility, , besides favoring a shorter clinical time when compared to metallic retainers.[4,5]

The retention of glass fiber posts with resin-based materials depends on the degree of conversion of the resin cement selected. The most used resin cement for cementing fiberglass posts is polymerized by blue visible light irradiation (photoinitiation, wavelength between 400-500 nm) associated with chemical initiation (dual-cured).[10,12] Incomplete polymerization of the resin cement compromises the mechanical properties of the composite material, what means that a clear understanding of the polymerization process and light characteristics of the curing unit is important.[1,10,11] Inadequate polymerization of the resin cement may lead to premature displacement of the retainers.[1,7,13]

When more intense light energy is used to activate a composite material, more photons are able to reach the photoinitiators (e.g.: camphorquinone) within the resin, which are activated and raised to the excited state. In this state, the photoinitiator molecule collides with an aliphatic amine, and a free radical is formed.[14] Then, the latter reacts with the carbon to break a carbon double bond (C=C) of a monomer molecule, thus initiating polymerization, converting monomers into polymers.[14] As seen, the light irradiation of the LED and the energy reached by the resin cement used for luting the glass fiber posts may be related to the loss of retention.[10,15]

This fact can occur when the energy dose from the light source is not sufficient to promote adequate polymerization of the resin cement inside the root canal. This situation may become even more critical in some regions of the root canal, particularly in the deeper thirds, since they are located far from the light source.[12,15,16] Even when using dual-cure resin cements, which are activated chemically and by light in order to favor polymerization in areas in which light is attenuated, the reduction in the irradiance reaching the deeper portions of the root canal can result in reduced degree of conversion for the resin cements, possibly impairing the properties of these materials.[10,16]

Due to the limitations of the first and second generation LED, a third generation was developed (known as "polywave"), which emit light with a wavelength ranging from 380 to 515 nm (ultraviolet to blue) unlike the second-generation LED, which emit light with a narrower wavelength (410-470 nm). The polywave units are able to provide better photoactivation when resin composites containing alternative photoinitiators are used. Even so, the amount of energy delivered to the furthest layers of the resinous compound can be influenced by factors such as photoinitiator reactivity, dispersion of light and lack of uniformity of the beam of light emitted.[17]

In clinical practice, light-curing units and their light output intensities can vary significantly, and there may be pronounced differences for newer lights, such as argon ion lasers and light-emitting diodes (LEDs), which have been continuously improved to achieve higher irradiation intensities.[14] Lithium-ion (Li-Ion) and lithium polymer (LiPO) batteries are the most common power sources used by the current cordless LED units, and little is known about the influence of the battery on the performance of these equipment while discharging.[14] Among so many variables, a cordless LED unit with low battery level can even provide inadequate light source when compared to a LED unit connected directly to the power source or with high battery level.[14] Therefore, this factor must be taken into consideration in order to ensure longevity to the restorative treatments using glass fiber posts cemented with resin cements.[14]

Thus, the aim of the present study was to investigate the Influence of the battery level of different cordless LED units on the characteristics of the emitted light and properties of a dual cure resin cement used for luting glass fiber posts. The following null hypotheses were tested: 1) the battery level of the different cordless LED units would not influence the light emitted, and; 2) the bond strength and degree of conversion of the resin cement would not be affected by the different LED units neither by the distinct battery levels in all root regions.

MATERIALS AND METHODS

Light characterization

Two cordless LED units were evaluated, a monowave LED (Radii Xspert, SDI, Australia, Bayswater, Victoria) and a polywave LED (Bluephase G2, Ivoclar Vivadent, Schaan, Liechtenstein). To determine the emitted light intensity corresponding to each battery level of the cordless LED units, the devices were fully charged following the manufacturer's recommendations (24 h) and used until full discharging (high mode). The maximum number of cycles with the battery charged at 100% has been defined, and thus the cycles for the 50% and 10% levels were determined. The devices were successively used and after every 50 activations (20 sec, each cycle) 3 light measurement were performed using a USB4000 spectrometer (NIST, MARC, Bluelight Analytics Inc., Halifax, Canada), in order to measure the intensity of light relative to the different battery levels for each LED unit. The irradiance (mW/cm^2) and the absolute irradiance (mW/cm^2/nm) of the LED units were individually checked in all battery levels (100%, 50% and 10%). For assuring the consistency of the data, three complete charge-discharge cycles with readings were performed for the two cordless LED units, after which the average of the three data blocks obtained in each cycle for the units in the distinct battery levels (100%, 50% and 10%) was calculated, as shown in the scheme (Fig. 1).

Micro push-out bond strength test (PBS)

For the bond strength test, sixty bovine teeth recently extracted were obtained (n=10), which had their coronary portion sectioned with double-sided diamond disc, resulting in 15.0 mm roots. To standardization of the specimens, roots in which a K #20 file (Dentsply Maillefer, Bensheim, Switzerland, Germany) was inserted tight to the root canal were selected as the specimen standard. Then, the root canals were prepared with manual K-files #20-35 up to 1.0 mm of the total length for formation of the apical stop and #40-50, backing 2.0 mm from the total working length. The root canals were irrigated with 2.5% sodium hypochlorite (Biopharma, Uberlândia, MG, Brazil) alternately at each instrument change. Finally, the canals were irrigated with saline solution and dried with absorbent paper points. The root canals were not sealed in order to avoid possible influences of the sealing cement and gutta-percha in the interaction between resin cement and root dentin.[12,15]

Post spaces (10.0 mm) were prepared using #2-4 Largo burs, followed by the drill recommended by the manufacturer for the glass fiber post system, obtaining adequate space for cementation of the posts (Exacto Conical Translucent, #2, Ângelus, Londrina, PR, Brazil). The roots were then randomly distributed between the 6 experimental groups (n=10), according to the LED unit (Bluephase G2 and Radii Expert) and the battery level during photoactivation (100%, 50% and 10%).

The glass fiber posts used have double-tapper geometry, with 1.6 mm diameter in the cervical portion and 0.9 mm in the apical portion (Exacto, #2, Ângelus, Londrina, PR, Brazil). The posts were cleaned with 70% alcohol solution and received surface treatment with 37% hydrogen peroxide for 1 min, being subsequently washed with air/water streams for 1 min followed by air-drying for 30 s.[3] Thereafter, a layer of silane coupling agent (Gluma Ceramic Primer, Heraeus Kulzer, Mitsui Chemicals Group, São Paulo, SP, Brazil) was applied actively for 30 s and volatilized for 60 s, followed by application of a layer of an universal adhesive system (Gluma to Bond Universal, Heraeus Kulzer, Mitsui Chemicals Group) and photoactivation for 20 seconds.

The glass fiber posts were then cemented with a self-adhesive dual-cure resin cement (Maxcem Elite, Kerr, Orange, CA, USA). The resin cement was mixed and inserted into the root canal using automix tips for better deposition of the cement along the canal, and then the posts were brought into position and kept under controlled digital pressure. The excess resin cement was removed after 1 min of the manipulation. The initial polymerization of the resin cement was allowed for 5 min and photoactivation was then performed for 40 s with the tip of the light source from the LED unit positioned perpendicular to the long axis of the post. After that, the specimens were stored at 100% humidity at 37 °C for 24 h prior to testing. The specimens were stored in distilled water to prevent tooth structure dehydration.[18]

Following storage, the specimens were fixed in acrylic plates with cyanoacrylate-based adhesive (Superbonder, Loctite, Itapeví, SP, Brazil) and Godiva (Exata, Nova DFL, Rio de Janeiro, RJ, Brazil) to be sectioned perpendicularly to the long axis of the root using a double-sided diamond disc of 0.5 mm (4" X 0.12 X 0.12, Extec, Enfield, CT, USA) mounted on a precision cutter (Isomet 1000, Buehler, Lake Bluff, IL, USA) using water cooling. Thus, two 1.0 mm thick discs were obtained for each root third of the specimens (cervical, middle and apical), which were individually stored in Eppendorf tubes. Then, the discs from the different root thirds were tested for micro push-out bond strenght in a universal testing machine (DL 2000, EMIC, São José dos Pinhais, PR, Brasil) to perform the micro push-out test. The discs were positioned so that the loading applicator tip coincided with the hole in the metal base and the compression loading in the apex/crown direction was performed on the surface of the post at a crosshead speed of 0.5 mm/min, until failure occurred. For each root third a tip corresponding to the diameter of the post was used (cervical third - 1.2 mm; middle third - 1.0 mm; apical third - 0.7 mm).

Push-out bond strength values in MPa were calculated by dividing the peak force by the bonded surface area (α) of the post segment. The bonded surface was calculated using the following formula:

$$a = 2\pi[(R + r) \div 2]h$$

Where, $\pi = 3.14$, R is the radius of the post in the cervical region, *r* is the radius of the post in the apical region, and *h* is the thickness of each layer in millimeters (calculated by digital caliper with 0.01 mm precision- 500-197-200, Mitutoyo, Tokyo, Japan). Push-out bond strength was calculated using the following formula:

$$\sigma = \frac{f}{a}$$

Where f is the load required for sample rupture, and a is the bonded surface (mm^2). Finally, the average of the results were discs referring to the same root third of each sample

Failure mode

After the micro push-out test, the failure mode of all specimens was analyzed in a measuring microscope (STM, Olympus Optical Co, Tokyo, Japan) at 25× magnification. According to the failures the specimens were categorized in the following failure modes using an adapted criterion[12]: I- Adhesive failure between dentin and resin cement; II- adhesive failure between resin cement and post; III- cohesive failure within resin cement; IV- Cohesive failure within dentin; V- mixed failure.

Representative specimens of each group were selected for scanning electron microscopy (n=3). The discs were cleaned in ultrasonic bath, dried and fixed on aluminum stub with double-sided carbon tape (Electron Microscopy Sciences, Washington, DC, USA). Then, the specimens received metallic coverage with a layer of gold/palladium (Denton Vacuum Desk II Sputtering, Denton Vacuum, Cherry Hill, NJ, USA). The specimens were placed in the scanning electron microscope (SEM- Carl Zeiss Microscopy GmbH 07745 JENA, ALEMANHA) in high vacuum mode with 20 kV voltage acceleration, working distance of 10 mm e spotsize ranging from 25 pA to 100 pA, for illustrating the failure modes.

Degree of conversion

For assessing the degree of conversion, specimens of the resin cement (n=5) simulating the incidence of light in the different root canal thirds (cervical, middle and apical) were obtained according to the battery levels of the two cordless LED units (100%, 50% and 10%). The specimens were obtained from custom Teflon matrices developed by the authors for this study (Fig. 2) which have a central hole with corresponding diameter to the glass fiber post used (Exacto #2, Ângelus – cervical diameter 1.6 mm; apical diameter 0.9 mm; taper 1 = 0.03 mm; taper 2 = 0.1 mm; Total length = 17.0 mm), and a lateral rectangular channel 1.0 mm x 0.5 mm for insertion of the resin cement. The resin cement was manipulated using automix tips and inserted in the lateral channel and then a glass fiber post was inserted in the central hole with a Millar strip interposed, assuring isolation between the resin cement and the post in the simulated root canal of the matrix.

The excess cement was removed after 1 min of the manipulation and after 5 min, photoactivation of the resin cement specimens was performed with the cordless LED units (Bluephase G2 and Radii Xspert) at the different battery levels (100%, 50% and 10%). The resin cement block simulating the cement layer in the different root thirds was separated from the post and Teflon matrix and wet ground using sandpapers, so they could present smooth surface to be tested for the degree of conversion. Then the resin cement specimens were stored sheltered from light at 100% relative humidity at 37 °C for 24 h. The degree of conversion of the resin cement specimens (n=5) was accessed in a Fourier transform infrared spectroscopy (ATR- FTIR) unit (Tensor 27, Bruker, Ettlingen, Germany) in the regions corresponding to the different root thirds (cervical, middle and apical; Fig. 2) according to the different battery levels of the cordless LED units (100%, 50% and 10%).

The remaining unconverted carbon double bonds were calculated by comparing the percentage of aliphatic C = C (1638 cm^{-1}) and 1716 absorbed between the polymerized and unpolymerized specimens of the resin cement Maxcem Elite (Glycerol phosphate dimethacryalte GPDM, Co-monomers, Proprietary self-curing redox activator, Camphorquinone, Stabilizer, Barium glass fillers, Fluoroalumino silicate glass filler, silica).[19,20] For methacrylate monomers without aromatic ring, a peak about 1710-1730 cm^{-1} associated with the carbonyl group C = O is used as reference.[21] The spectra of the polymerized and unpolymerized specimens were obtained using 128 scans at a resolution of 4 cm

$^{-1}$ in the range of 400 to 6000 cm $^{-1}$. Spectra of interest were obtained using the software provided with the FTIR equipment (Omnic 6,1, Nicolet Instrument Corp., Madison, WI, EUA) expanding in the region of interest between 1560 and 1670 cm $^{-1}$. The degree of conversion was calculated using the standard baseline technique and a peak area comparison at 1639 cm $^{-1}$ (C = C aliphatic) and the peak of the internal standard at 1716 cm $^{-1}$. Then, the degree of conversion (GC) was calculated by the following equation:

$$GC\% = 100 - \left[\frac{\frac{Abs.C = C(1.635cm^{-1})}{Abs.C = 0(1.716cm^{-1})} polymer}{\frac{Abs.C = C(1.636cm^{-1})}{Abs.C = 0(1.716cm^{-1})} monomer} \right] \times 100$$

Statistical Analysis

The micro push-out (PBS) and degree of conversion (DC) data were statistically analyzed using a 2-way analysis of variance (light curing unit and battery level) with repeated measures (root canal region) followed by the Tukey test post-hoc multiple comparison test. The significance level was set at 5%. All statistical analyses were performed using Sigma Plot 12.1 (Stata Corp, College Station, TX).

RESULTS

Light characterization

The battery level of the cordless LED units (Bluephase G2 and Radii Xspert) affected the light intensity emitted by the both devices. The irradiance has decreased as the battery level reduced. The difference between the light intensity versus the battery level was greater for the Bluephase G2 unit when compared to Radii Xspert. The results are shown in Figs. 3 and 4. The figures show a decrease in the intensity of light at the end of the 20 seconds period defined in the LED unit, followed by a new activation cycle with light intensity increase until complete 40 seconds (2 activation cycles).

Micro Pushout Bond Strength (PBS)

The bond strength values (MPa) (mean and standard deviation) in terms of the light curing unit, battery level and root canal region for the experimental groups, are shown in Fig 5. Two-way ANOVA showed significant influence for the all isolated factors: light curing unit ($P = 0.003$), battery level ($P < 0.001$) and root canal region ($P < 0.001$); and also for interaction between the light curing unit and root region factors ($P = 0.013$). No significant differences were found for interaction between light curing unit and battery level ($P = 0.173$), battery level and root canal region ($P = 0.954$), light curing unit, battery level and root canal region ($P = 0.829$). The root depth reduced significantly the bond strength values for all the tested groups. The cervical region showed the highest PBS values while the apical region had the lowest values. The 100% and 50% battery levels showed significant higher PBS values than the 10% level for both light curing units, independent of the third root. The specimens photoactivated with the Bluephase G2 exhibited higher PBS values than the Radii Xspert specimens in all root thirds at 100% and 50% battery level. At 10% battery level, the specimens photoactivated with both LED units had similar PBS performance.

Failure mode distribution

The most frequent failure mode found for the specimens after the PBS test was the mixed mode (Type V) as shown in Fig. 6. The second failure mode most prevalent was the adhesive failure between resin cement and post (Type II). No cohesive failures in dentin were detected (Type IV). Fig. 7 illustrate the failure modes found in the present study (Types I, II, III and V). Besides the faults, it was possible to observe bubbles and cracks.

Degree of conversion

The degree of conversion (%) (mean and standard deviation) in terms of the light curing unit, battery level and root canal region for the experimental groups, are shown in Fig. 8. ANOVA showed no significant differences for all isolated factors battery level ($P = 0.134$), light curing unit ($P = 0.279$), root canal region (P = 0.336); and also for interaction between the light curing unit and root canal region factors (P = 0.379), light curing unit and battery level (P = 0.133), battery level and root canal region (P = 0.430), light curing unit, battery level and root canal region (P = 0.799). The degree of conversion of the resin cement used exceeded 60% for all the situations investigated.

DISCUSSION

According to the results observed in the present study, the first null hypothesis was rejected because the battery level of the cordless LED units affected the light characteristics of the devices evaluated. There was a progressive decrease in the irradiance as the battery levels of the Bluephase G2 and Radii Xspert cordless LED units were reduced.

A previous study concluded that the light intensity significantly affects the physical and biological properties of resin-based materials.[22] Another showed that light curing units with higher irradiances can result in better properties for resin cements used in post cementation.[12] These facts may be helpful to explain the recurrent problem of retention loss observed for glass fiber posts.[12] Thus, clinicians should be aware on this aspect, since the adequate reaction of the photoinitiator in the resin cement is directly proportional to the light intensity received by the material.[7,12,23]

The second null hypothesis was also rejected since the different battery levels of the both LED units influenced the bond strength values of the resin cement for all the root thirds, with the polywave LED unit presenting better performance than the monowave unit. Several studies have shown significant differences in the bond strength values for glass fiber posts fixed with resin cements in the different root thirds, commonly, with higher in the coronal third and lower in the middle and apical thirds,[5,8,12,16,23,25] what is in agreement with our findings. It is important to emphasize that in the present study, the posts were loaded with different tips in the micro pushout test, using a tip corresponding to the diameter of the post in each root third (cervical third - 1.2 mm; middle third - 1.0 mm; apical third - 0.7 mm) to avoid the applicator tip to exert loading on the dentine. The use of loading tips with standardized size for all root thirds can lead to inconsistent bond strength results when inadvertently loading dentin, since cohesive fractures may occur in this substrate.

This fact can also be explained by the decrease in light transmission from the light output through the root canal, especially at the end of the middle third to the apical region.[16] By its turn, the increased light penetration in the coronal region may explain the higher bond strength values commonly verified for this region in this study and by other investigations.[5,16] Even in the case of using dual-cure resin cements, the chemical reaction alone may not be able to ensure adequate properties to the material in regions that receive reduced light intensity from the curing unit.[5,16] A relationship between the effective light intensity available for photoactivating resin-based materials can be made by relating the distance between the tip of the curing unit and the surface of the material.[26] According to this, the light intensity can decrease in proportion to the square of the distance, i.e. by folding the distance between the output tip and the surface of the material, results in a reduction of light intensity to a quarter of the original intensity.[26] Thus, one can assume that an increase in the distance between the light output and the surface of the resin-based material will result in reduced light intensity and, consequently, in decreased energy density.[26]

Camphorquinone has an absorption peak near 470 nm, and the polymerization in the deeper layers of the resin material can be influenced by factors like reactivity of the photoinitiator, light scattering and a lack of uniformity of the light beam emitted by the light curing unit.[17] It is possible that the use of high radiant emission for a longer period of time may have led to higher bond strength values in the present study, as suggested by other investigation.[17] Additionally, a previous study showed that polywave LED units have promoted higher degree of conversion for composites resin than monowave LED units,[21] and despite this difference was not verified in the present investigation for DC, significant differences were observed between the LED units for PBS values. The more favorable results found for the polywave LED unit may be explained by its emission spectrum, which is similar to the absorption spectrum of the photoinitiator, since the peak emission of the polywave unit was 461 nm, while the emission peak of the monowave unit was at 447 nm.[21]

Another possible explanation for the lower bond strength found in deeper root regions is that in the apical third fewer dentinal tubules are present and the dentine is more irregular when compared to the dentine of the cervical third, which has a higher tubule density, thus influencing the adhesive properties of the resin-based materials.[11,16] In addition, factors such as root canal configuration, post type,[23] restrict access and visualization,[9,12] humidity control,[8] irrigation methods,[3] cleaning of the dentinal walls in the apical region,[9] and cement layer thickness,[4] can also affect the retention of the glass fiber posts.[12,16,25]

The adhesive strategy used in the fixation of glass fiber posts can also influence the bond strength values, as demonstrated in previous studies.[12] Some investigations showed increased bond strength for glass fiber posts treated with 32-37% hydrogen peroxide prior to the application of silane coupling agents.[12,21,24] However, simple procedures that require less clinical time and have greater technical convenience should be indicated.[7,23] Approaches that require multiple steps as when using resin cements that depend on the previous application of adhesive systems and etchants can be more critic.[7,18]

Although adhesion to deeper portions of root dentin may be compromised, results from other studies have demonstrated that increasing the depth of the preparation during the canal relief, may contribute to improved retention of the glass fiber posts.[12] Higher bond strength values were observed in the apical area of the root canals for glass fiber posts when using self-adhesive resin cement, and the authors justified that this result is dependent on the type of resin cement used in the different experimental models.[11] In the specific case of Maxcem Elite, this material showed lower bond strength possibly due to its monomer component GPDM.[20] The GPDM monomer plays the self-etching role, but the acid component of this resin cement may be insufficient to trigger adequate chemical bonding with the dental substrates, consequently, not achieving greater bond strength.[20] MDP containing resin cements, would then present different results compared to the cement used in the present investigation.

The most frequent failure mode found for the specimens after bond strength test in this study was the mixed failure (Type V), corroborating with other studies that performed bond strength tests in root canals using bovine teeth.[5,11,23] The second failure mode most frequently verified was the adhesive failure (Types II and III), what is in agreement with other investigations.[8,12,20,24] Most adhesive failures may be possibly related to the type of teeth used in the present study, because the use of bovine teeth may result in increased thickness of the resin cement layer, because of the larger root canal diameter. The polymerization shrinkage of the resin cement is increased in thicker layers, what can contribute to a greater number of adhesive failures.[25,26] In this way, the mixed failure is considered more favorable when compared to the adhesive failure, since it demonstrates better interaction between dentin-cement-post.[12]

SEM images showed crack propagation around the structures adjacent to the glass fiber post in the mixed failures, that is, resin cement and dentin, corroborating with the results of other studies.[12] Structural discontinuities and bubbles are often observed in the apical third, which are most evident at the resin-cement/dentin interface,[26] with similar findings verified in the present study, mainly in the apical third. This fact can be explained by the way the resin cement is inserted, what can lead to the formation of bubbles. The insertion of the resin cement using automix tips tends to produce more homogeneous cement layers with less bubbles.[25,26]

Fourier transform infrared spectroscopy is an accurate method used to measure the degree of conversion of resin-based materials, allowing to calculate the percentage of double carbon bonds that transform into single bonds (monomer-polymer conversion).[16] Despite the second null hypothesis was rejected, no significant differences were verified for the DC between the experimental groups. The results found in this study may have been influenced by the storage time used for the DC specimens (24 h), which probably allowed for extended chemical polymerization of the resin cement. Even though, the design used for the specimens analyzed is compatible to what happens in clinical practice. The dual-cure resin cements begin their polymerization from the chemical reaction (benzoyl peroxide + aromatic tertiary amines) and by the presence of light (camphorquinone + aliphatic amines).[16] Therefore, the use of translucent glass fiber posts may be helpful to improve light transmission along the root canal during photoactivation.[1] Results of a previous study support this assertion, since when using translucent glass fiber posts, the degree of conversion of the resin cement was not significantly different between the root thirds.[16]

These results may also be explained by the good performance of the chemical portion of the resin cement used, since even without the presence of light activation this material showed acceptable degree of conversion in other investigations.[19] Some dual-cure resin cements have limited chemical polymerization when exposed to light immediately, what can restrict materials to achieve proper mechanical properties.[15] Manufacturers generally recommend the immediate photoactivation of dual-cure resin cements. However, some authors recommended a waiting period before photoactivating these materials in order to avoid the interference of light activation in the polymerization kinetics of the chemical activation components.[13] Thus, the chemical reaction mechanism proceeds slowly and is expected to ensure polymerization, especially in areas where the light from curing units is unable to reach. This waiting period can help polymerization chains to build a network, especially on the surface of the cement, thus acting as an insulating layer preventing the diffusion of unreacted monomers and by-products from the deeper layers.[13] Thus, waiting time for the chemical reaction is a significant variable that can affect polymerization as the reaction by light only can limit the depth of polymerization and conversion at sites away from the light source.[29]

The limited number of studies evaluating the battery levels of cordless LED units made comparisons of the results more difficult. The literature is still limited about the interference of photoactivation modes in the properties of resin cements when luting glass fiber posts.[11] No previous studies reporting the battery level factor and its influence during the cementation of glass fiber posts were found. But, based on the findings of this study, it can be admitted that decreased battery levels in cordless LED units result in reduced light irradiance. This fact can intensify light attenuation during the glass fiber post cementation procedures, interfering in the bond strength of the resin cements used in the fixation of these retainers.

Another limitation is that a single resin cement was tested. Since resin cements differ in their chemical and mechanical properties, caution should be taken when transferring the results of the present study to other resin-based materials available. Further studies evaluating different resin cements for the cementation of glass fiber posts under distinct conditions of photoactivation with different LED units would be of benefit. This study highlights important information on the cementation of glass fiber posts, showing that bond strength of resin cements to root dentin can be affected by different battery levels of LED units.

CONCLUSION

The light characteristics of cordless LED units can be affected by low battery levels, and although the degree of conversion was not affected for this factor, it has influenced the bond strength. Cordless LED units should be kept with 50%-100% battery levels in order to achieve better properties for resin cements, thus reducing the risk of retention loss for glass fiber posts. The polywave light curing unit tested resulted in better performance for the bond strength in all variables.

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ANEXOS

Figures.

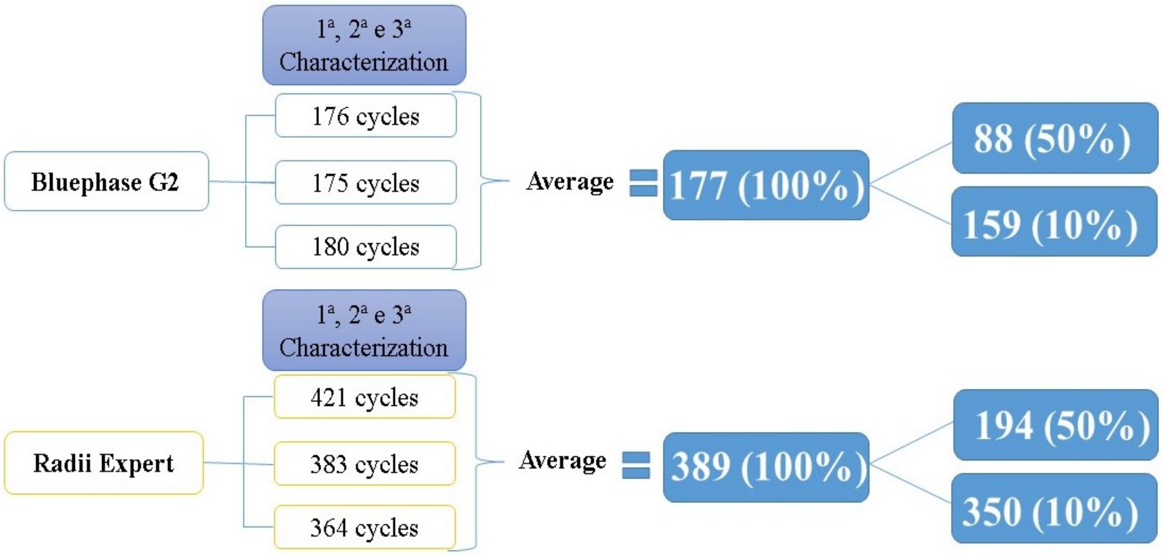
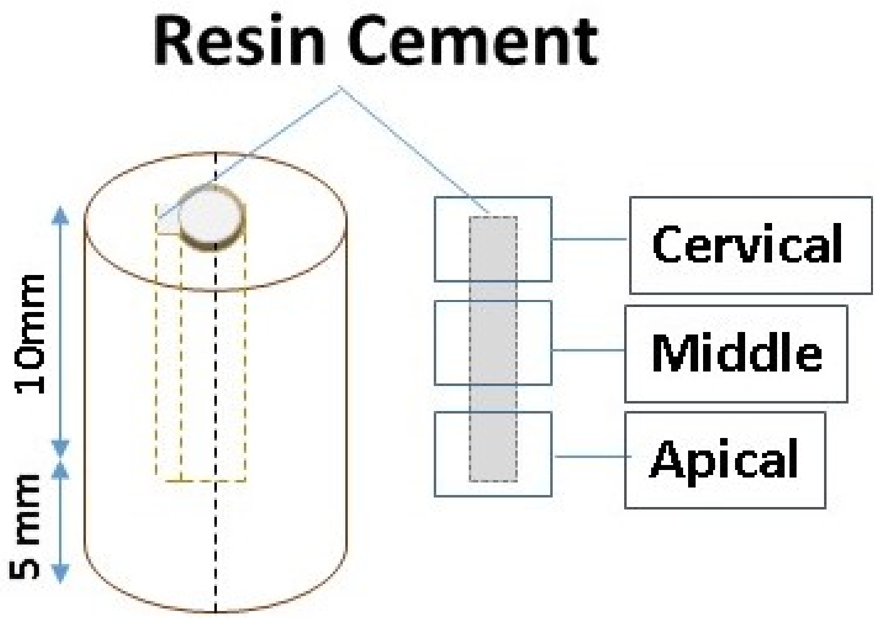


Fig. 1. Scheme for light characterization of each LED unit at the distinct battery levels (100%, 50% and 10%).



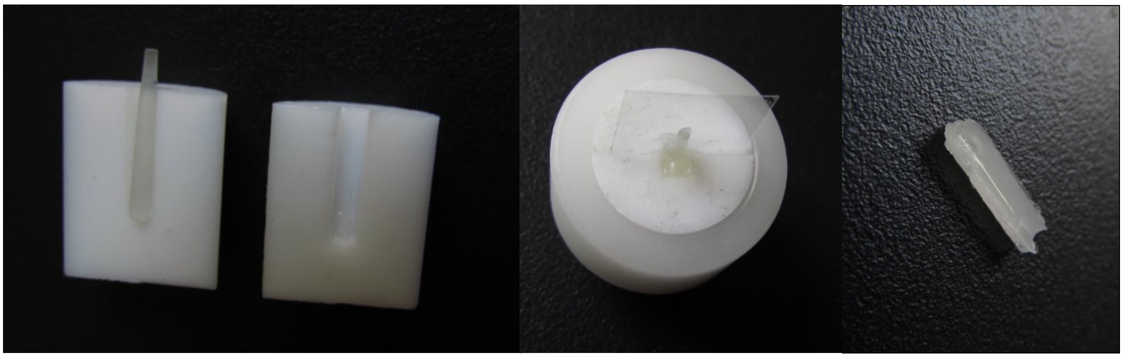


Fig. 2 – Schematic representation and picture of the custom Teflon matrix with the obtained resin cement block and the respective regions corresponding to the root third in which the readings for degree of conversion were performed.

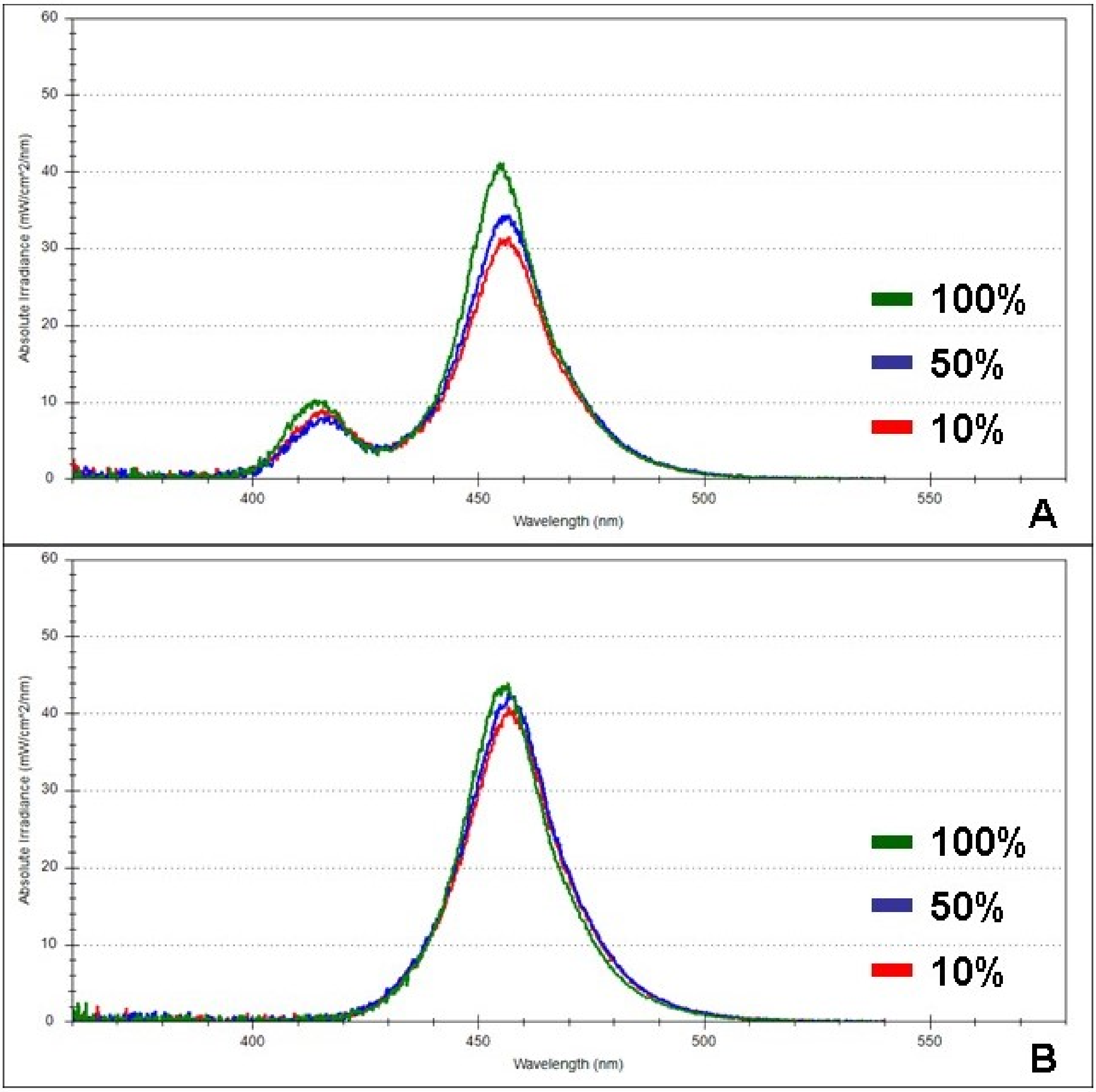


Fig 3. A: Absolute irradiance (mW/cm^2/nm) of the Bluephase G2 cordless LED unit in the three battery levels (100%; 50%; 10%); B: Absolute irradiance (mW/cm^2/nm) of the Radii Xspert cordless LED unit in the three battery levels (100%; 50%; 10%).

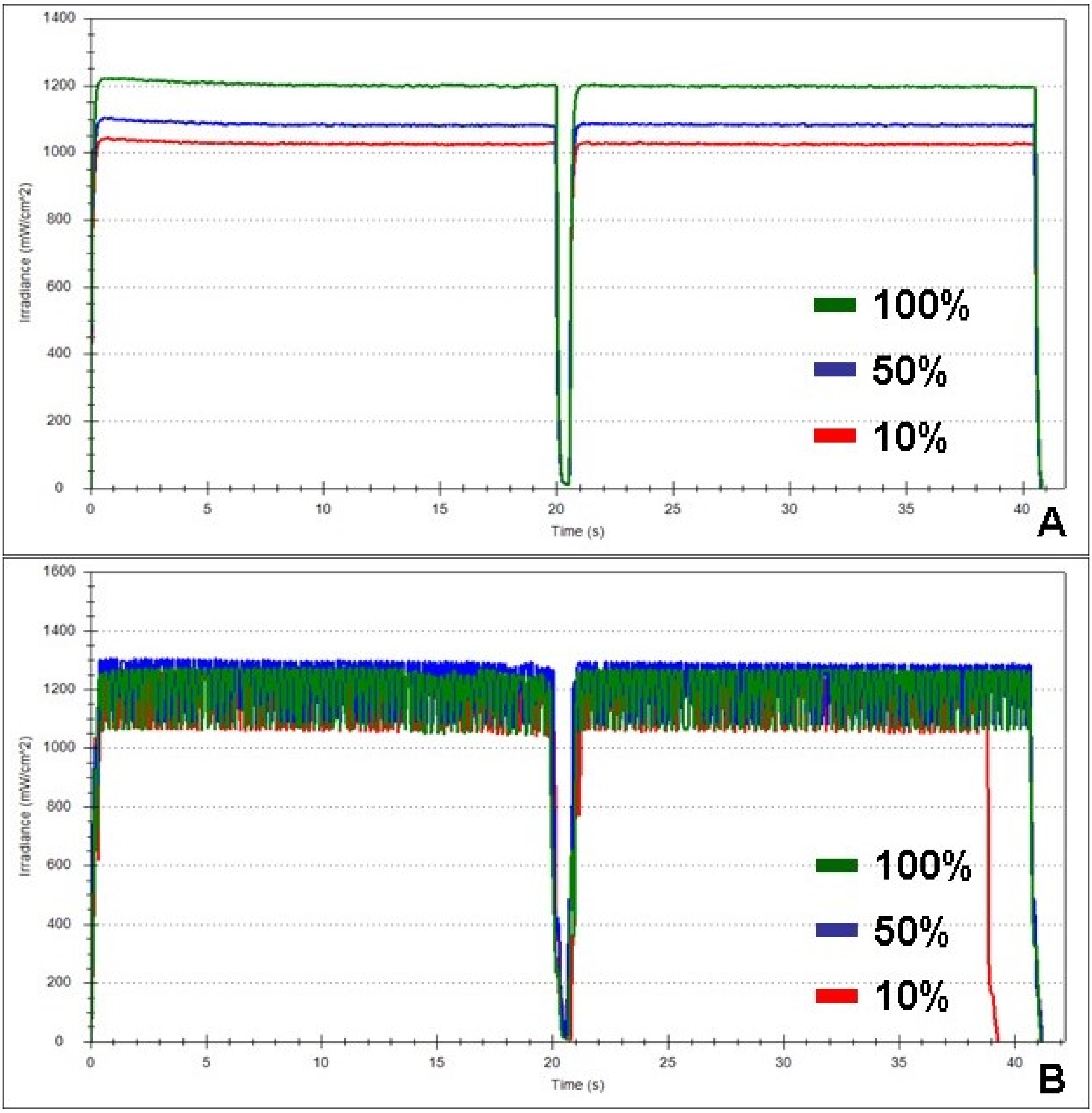


Fig 4. A: Irradiance (mW/cm^2) of the Bluephase G2 cordless LED unit in the three battery level (100%; 50%; 10%); B: Irradiance (mW/cm^2) of the Radii Xspert cordless LED unit in the three battery levels (100%; 50%; 10%).

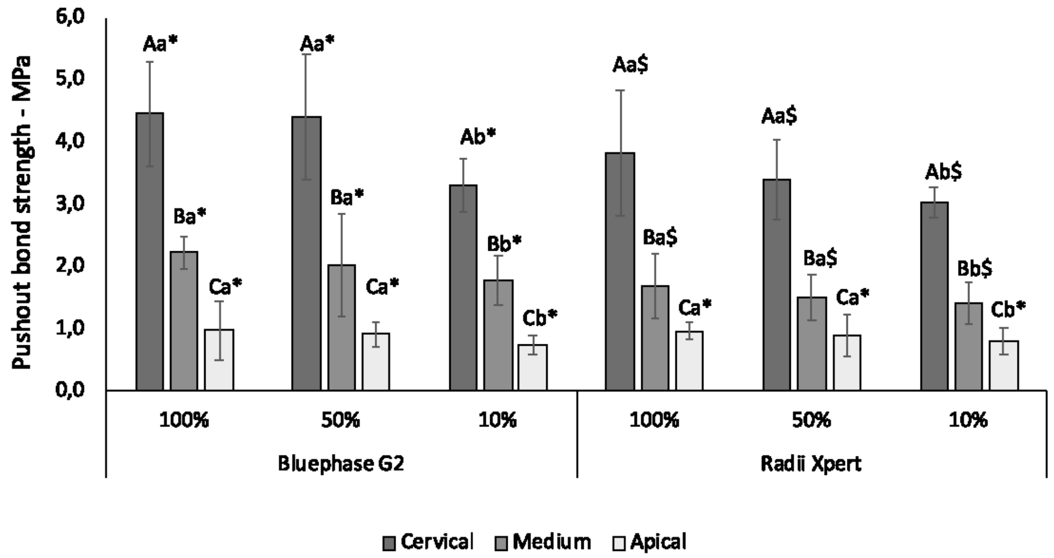


Fig 5. Push-out bond strength values in MPa (mean and standard deviation) according to light curing unit, battery level and root canal region. Mean values followed by different letters differ among them by the Tukey test ($P < 0.05$). Uppercase letters used for comparing the root region; lowercase letters used for comparing battery level for each LED unit and; symbols used for comparing LED unit for each battery level.

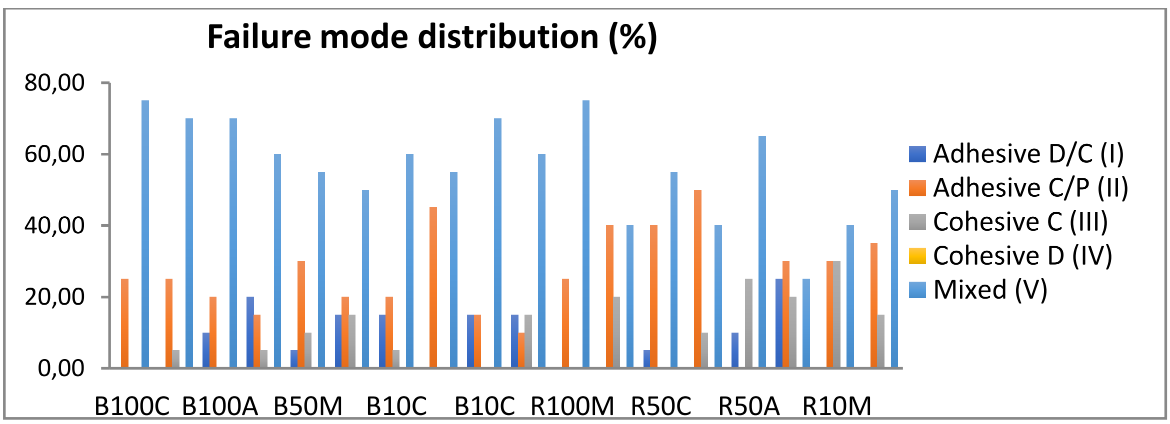


Fig 6- Failure mode distribution for the experimental groups.

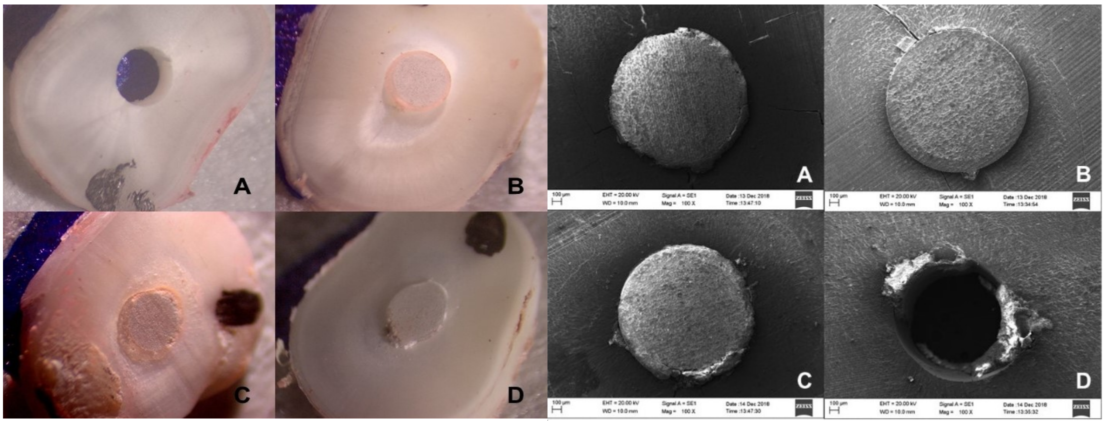


Fig. 7. Optical microscopy images (0.63× magnification) and SEM images (100× magnification) of the failures modes: A- optical microscopy shows absence of cement inside the root canal and SEM image shows remaining cement in the post, adhesive failure between dentin and resin cement (Type I); B- no cement is observed in the post surface, adhesive failure between resin cement and post (Type II); C- cement is observed in both dentin and post, cohesive failure within resin cement (Type III); D- some parts of the post present cement and others not, as in dentine, mixed failure (Type V).

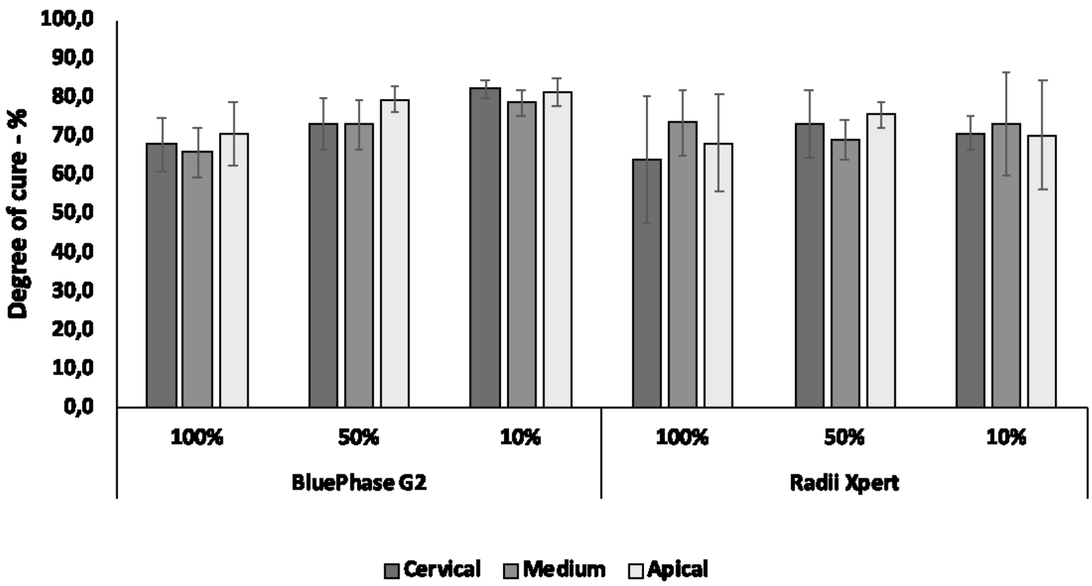


Fig. 8. Degree of conversion (%) (mean and standard deviation) according to LED unit, battery level and root region. Statistical analysis showed no significant differences for all isolated factors battery level, light curing unit, root canal region; and also for interaction between then. The degree of conversion of the resin cement used exceeded 60% for all the situations.

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