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BEATRIZ DE CÁSSIA ROMANO

DETERMINAÇÃO DA PROFUNDIDADE DE CURA DE COMPÓSITOS RESTAURADORES

DEPTH OF CURE DETERMINATION OF THE RESTORATIVE COMPOSITES

PIRACICABA

2022

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Dissertação apresentada à Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas como parte dos requisitos exigidos para obtenção do título de Mestra em Materiais Dentários.

Dissertation presented to the Piracicaba Dental School of the University of Campinas in partial fulfillment of the requirements for the degree of Master in Dental Materials.

Orientador: Prof. Dr. Marcelo Giannini

ESTE EXEMPLAR CORRESPONDE À VERSÃO FINAL DA DISSERTAÇÃO DEFENDIDA PELA ALUNA BEATRIZ DE CÁSSIA ROMANO E ORIENTADA PELO PROF. DR. MARCELO GIANNINI.

PIRACICABA
2022

Agência(s) de fomento e nº(s) de processo(s): CNPq, nº 130481/2020-3

Ficha catalográfica
Universidade Estadual de Campinas
Biblioteca da Faculdade de Odontologia de Piracicaba
Marilene Girello - CRB 8/6159

R662d
Romano, Beatriz de Cássia, 1996-
Determinação da profundidade de cura de compósitos restauradores /Beatriz de Cássia Romano. – Piracicaba, SP : [s.n.], 2022.

Orientador: Marcelo Giannini.
Dissertação (mestrado) – Universidade Estadual de Campinas, Faculdadede Odontologia de Piracicaba.

1. Resinas compostas. 2. Dureza. 3. Solventes. I. Giannini, Marcelo, 1969-. II. Universidade Estadual de Campinas. Faculdade de Odontologia dePiracicaba. III. Título.

Informações para Biblioteca Digital

Título em outro idioma: Depth of cure determination of the restorative composites
Palavras-chave em inglês:
Composite resins
Hardness
Solvents
Área de concentração: Materiais Dentários
Titulação: Mestra em Materiais Dentários
Banca examinadora:
Marcelo Giannini [Orientador]
André Luís Faria e Silva
Míriam Lacalle Turbino
Data de defesa: 29-04-2022
Programa de Pós-Graduação: Materiais Dentários

Identificação e informações acadêmicas do(a) aluno(a)
- ORCID do autor: https://orcid.org/0000-0002-2574-6269
- Currículo Lattes do autor: http://lattes.cnpq.br/5570264214587071

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Faculdade de Odontologia de Piracicaba

A Comissão Julgadora dos trabalhos de Defesa de Dissertação de Mestrado, em sessão pública realizada em 29 de abril de 2022, considerou a candidata BEATRIZ DE CÁSSIA ROMANO aprovada.

PROF. DR. MARCELO GIANNINI

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A Ata da defesa, assinada pelos membros da Comissão Examinadora, consta no SIGA/Sistema de Fluxo de Dissertação/Tese e na Secretaria do Programa da Unidade.

DEDICATÓRIA

Dedico este trabalho à minha família, **Marilena Paccagnan Perili Romano, Alexandre Romano** e **Lucas Romano**, por serem exemplo de vida, pelo apoio incondicional em minhas decisões e por não pouparem esforços em minha formação educacional e profissional.

AGRADECIMENTOS

Agradeço à **Deus**, por guiar cada passo e iluminar o meu caminho.

Ao meu orientador **Prof. Dr. Marcelo Giannini** pelo exemplo de profissional e competência. Agradeço pela oportunidade de orientação e trabalho, pelos conhecimentos transmitidos e oportunidades concedidas.

Ao meu coorientador, **Prof. Dr. Jorge Rodrigo Soto-Montero**, pelo suporte nesse trabalho e por todo o ensinamento e disponibilidade com minha formação profissional.

Ao **Prof. Dr. Elliot Watanabe Kitajima** da Escola Superior de Agricultura Luiz de Queiroz (ESALQ) e ao técnico **Renato Barbosa Saralori** por possibilitarem o uso do microscópio óptico, propiciando imagens de alta resolução para o enriquecimento deste trabalho.

À Universidade Estadual de Campinas – UNICAMP, na pessoa do magnífico Reitor **Prof. Dr. Antônio José de Almeida Meirelles** e à Faculdade de Odontologia de Piracicaba, por meio do diretor **Prof. Dr. Francisco Haiter Neto**.

À coordenadora geral dos cursos de Pós-graduação da FOP-UNICAMP, **Profa. Dra. Karina Gonzales Silvério Ruiz.**

Ao **Conselho Nacional de Desenvolvimento Científico e Tecnológico - CNPQ** pela concessão de bolsa de estudos durante o mestrado (processo nº 130481/2020-3).

Ao **Programa de Pós-Graduação em Materiais Dentários** da Faculdade de Odontologia de Piracicaba e a todos os docentes da área, **Prof. Dr. Américo Bortolazzo Correr, Prof. Dr. Luís Roberto Marcondes Martins, Prof. Dr. Lourenço Correr Sobrinho, Prof. Dr. Mario Alexandre Coelho Sinhoreti, Prof. Dr. Mário Fernando De Góes, Prof. Dr. Rafael Leonardo Xediek Consani, Prof. Dra. Regina Maria Puppin Rontani** e **Prof. Dr. Simonides Consani,** por todo aprendizado e pelo crescimento profissional que me propiciaram durante o mestrado.

Aos membros da banca de qualificação, **Prof. Dr. Adriano Fonseca de Lima, Prof. Dr. Carlos Alberto Kenji Shimokawa** e **Prof. Dr. Rafael Leonardo Xediek Consani**, por aceitarem o convite, dispenderem o tempo para leitura e correção, e contribuir para o enriquecimento do trabalho.

Ao engenheiro mecânico **Marcos Blanco Cangiani** e à secretária **Selma Aparecida Barbosa de Sousa Segalla**, do laboratório da Área de Materiais Dentários, da Faculdade de Odontologia de Piracicaba, pela disposição e auxilio na condução das etapas laboratoriais.

Aos amigos e colegas adquiridos durante o programa de pós-graduação, especialmente aos de orientação, **Amanda Endres Willers, Beatriz Ometto Sahadi, Daniela Bandeira, Daniel Kenji Funabashi, Eduardo Fernandes de Castro, Jorge Rodrigo Soto-Montero, Mayara dos Santos Noronha e Vitaliano Araújo Neto**, pelos momentos de descontração e ensinamentos.

Aos amigos, **Maria Júlia Assis Vicentin, Thomas Barbin, Letícia Tank Oliveira, Willian Peter Boelcke**, companheiros desde a graduação que dividiram experiências e bons momentos durante essa caminhada.

À toda minha **família** pela dedicação, amor e carinho que sempre tiveram comigo, por sempre estarem presente na minha formação pessoal e me apoiarem para alcançar meus sonhos.

Agradeço a todos aqueles que de certa forma contribuíram com meu desenvolvimento pessoal e profissional. Àqueles que sempre me apoiaram incentivaram e desejaram o bem.

Meus sinceros agradecimentos.

RESUMO

O objetivo deste estudo foi avaliar a influência de métodos para determinação da profundidade de cura (PC) e tempos de fotoativação de compósitos restauradores. Foram avaliados dois compósitos convencionais (Filtek Universal, 3M Oral Care e Beautifil II, Shofu Corp.) e dois compósitos do tipo *bulk-fill* (Filtek One Bulk Fill, 3M Oral Care e Beautifil Bulk Restorative, Shofu Corp.). A partir de matrizes de polivinilsiloxano de 6 mm de diâmetro interno e 12 mm de altura, foram confeccionadas amostras cilíndricas as quais foram fotoativadas de acordo com o tempo de exposição recomendado pelo fabricante ou o dobro do tempo recomendado. Foram obtidos 320 espécimes, sendo 40 para cada compósito em cada tempo, os quais foram analisados pelo método da ISO 4049 e pelo método de dissolução em solvente. No método da ISO 4049, a resina não polimerizada foi removida manualmente com espátula plástica e para o cálculo da PC o comprimento remanescente da amostra foi mensurado com paquímetro digital. No método da dissolução em solventes, após remoção da resina não polimerizada, as amostras foram distribuídas entre os solventes: acetona, etanol, clorofórmio e tetrahidrofurano (n=10) e armazenadas por 48 horas. Após a remoção do solvente, a área afetada pelo solvente e a PC foram mensurados com o software ImageJ e em seguida, a microdureza longitudinal foi determinada. Os dados obtidos da área afetada pelo solvente foram analisados estatisticamente por modelos lineares generalizados, com intervalo de confiança de 95%. Na análise dos dados da PC e de microdureza, utilizou-se modelos lineares generalizados (3 fatores: solvente, região do espécime e tempo de exposição) e teste de Kruskal Wallis seguido do método de Dunn para múltipla comparação com o Controle (ISO 4049). O método ISO 4049 pode apresentar diferenças em comparação com a técnica de dissolução com solventes nas áreas laterais ou centrais das amostras, mas depende do tipo de compósito. A microdureza para todos os compósitos foi reduzida com o aumento da profundidade, mais precisamente a partir de 3 a 4 mm para as resinas convencionais e 4 a 5 para as do tipo *bulk-fill*, dependendo do tempo de cura. O aumento do tempo de fotoativação pode aumentar a PC e manter os valores microdureza em maior profundidade. A área afetada pelo solvente dependeu do tipo de compósito e solvente. Os resultados sugerem que os materiais apresentam comportamentos distintos frente às metodologias utilizadas e o cálculo da PC pode variar de acordo com a técnica de medição empregada.

Palavras-Chave: Profundidade de cura, Resina composta, Fotoativação, Dureza, Solventes.

ABSTRACT

The aim of this study was to evaluate the influence of methodologies on the determination of the depth of cure (DOC) and light activation time of restorative composites. Two conventional composites (Beautifil II, Shofu Corp. and Filtek Universal, 3M Oral Care) and two *bulk-fill* composites (Beautifil Bulk Restorative, Shofu Corp. and Filtek One Bulk Fill, 3M Oral Care) were evaluated. Cylindrical samples were fabricated from polyvinylsiloxane matrices with an internal diameter of 6 mm and a height of 12 mm, which were light-cured according to the exposure time recommended by the manufacturer or twice the recommended time. A total of 320 samples were obtained, 40 for each composite at each time, which were analyzed by the ISO 4049 method and by the solvent dissolution method. In the ISO 4049 method, the unpolymerized resin was manually removed with a plastic spatula and for the DOC calculation, the remaining length of the sample was measured with a digital caliper. In the solvent dissolution method, after removing the unpolymerized resin, the samples were distributed among acetone, ethanol, chloroform and tetrahydrofuran (n=10) and stored for 48 hours. After solvent removal, the solvent-affected area (AAOS) and DOC were measured with ImageJ software and the microhardness (KNH) was also analyzed in depth. Data obtained from the AAOS were statistically analyzed by generalized linear models, with a confidence interval of 95%. In the analysis of DOC and KNH data, generalized linear models were used (3 factors: solvent, sample region and exposure time) and Kruskal Wallis test followed by Dunn's method for multiple comparison with the control group (ISO 4049). The ISO 4049 method showed differences compared with solvent-dissolution technique. KNH for all the RBRMs was reduced following a depth increase. The results suggest that increasing the light curing time extended the DOC and KHN. The affected area by solvent is material and solvent dependent. There are differences in the properties of the tested RBRMs and calculation of the depth of cure can vary according to the measurement technique used.

Key words: Depth of cure, Resin composites, Light curing, Hardness, Solvents.

SUMÁRIO

1. INTRODUÇÃO

Restaurações com emprego de resina composta ou compósitos são rotineiramente realizadas para repor os tecidos dentários perdidos por cárie, desgaste ou fratura e nos casos em que haja exigência estética (Spencer et al., 2010; Liu Y et al., 2011). A introdução desses materiais poliméricos associados aos sistemas adesivos tem permitido a realização de procedimentos restauradores de forma mais conservadora (Ferracane, 2011). Inicialmente, as resinas compostas eram ativadas quimicamente (Craig, 1981) e demonstravam razoável estética e durabilidade, embora ainda houvesse a necessidade de busca por um material que permitisse um tempo de trabalho prolongado, tempo de presa reduzido, estabilidade de cor e resistência ao desgaste (Paffenberger G, 1974; Rueggeberg et al., 2010; Ferracane, 2011). O desenvolvimento de compósitos ativados por luz facilitou o tempo de trabalho do material e melhorou a qualidade das restaurações, ainda que outros obstáculos intrínsecos ao material e ao procedimento tenham surgido, como a contração e tensão de polimerização, além da subpolimerização dos compósitos (Tirtha et al., 1982; Rueggeberg, 1999; Stansbury, 2000).

Em algumas situações clínicas como as cavidades profundas em dentes posteriores, grande volume de compósito é necessário para restauração. O material deve ser aplicado em camadas (ou incrementos), cujas espessuras dependem do tipo de compósito. Essas camadas são fotoativadas e dependendo do posicionamento intraoral do aparelho fotoativador, a luz pode não atingir o material com energia suficiente para que ocorra a polimerização uniforme e eficiente, que compromete as propriedades físicas do material e consequentemente, a durabilidade das restaurações (Price e Felix, 2009; Rueggeberg, 2011). A ineficiência da polimerização está associada às consequências clinicas como abertura de fendas, sensibilidade pós-operatória, pigmentação marginal (Price et al., 2014), infiltração bacteriana nas margens da restauração e possibilidade de desenvolvimento de cárie recorrente (Ferracane, 2013).

Com a finalidade de minimizar esses efeitos negativos, convencionou-se que os compósitos tradicionais deveriam ser aplicados em incrementos com espessura entre 1,5 a 2 mm (Lutz et al., 1986; Pollack, 1987; Hilton e Ferracane, 1999). Dessa forma, seria possível assegurar um maior grau de conversão monomérico da resina e também controlar a tensão de polimerização, que é reflexo da contração do volume de material e do fator C cavitário (Van Dijken, 2010; Borges et al., 2014). A inserção incremental tradicional dos compósitos na espessura de até 2 mm tem sido utilizada por mais de 30 anos, entretanto novos materiais podem ser inseridos em incrementos maiores que simplificam e reduzem o tempo clínico para os dentistas confeccionarem uma restauração dental.

Modificações na composição das resinas restauradoras têm gerado as resinas do tipo *bulk-fill*. De acordo com os fabricantes, esses materiais permitem aplicação de incrementos maiores e igual que 4 mm de espessura, com grau de conversão uniforme, menor contração volumétrica e tensão de polimerização. Desde o seu lançamento até os dias de hoje, as resinas do tipo *bulk-fill* têm passado por um desenvolvimento e aprimoramento substancial (Pfeifer et al., 2017). As novas formulações desses materiais restauradores a base de resina e que possibilitam o aumento da espessura do incremento do compósito compreendem modificações nas propriedades ópticas para aumentar a translucidez inicial do material, alterações na matriz monomérica para modular a reação de polimerização, adição de fotoiniciadores mais reativos e diferentes tipos de partículas de carga, como pré-polímeros e fibras de vidro capazes de absorver as tensões, além de aumentar a resistência mecânica. Porém existe um grande questionamento acerca da efetividade da polimerização nas camadas mais profundas da restauração com espessura maior das camadas de compósitos

Após o processo de fotoativação, embora a superfície da resina mais próxima da luz polimerize prontamente dando a aparência de que todo o material foi completamente polimerizado, nem sempre ocorre o mesmo nas camadas mais profundas da restauração. A extensão em profundidade que os monômeros são convertidos em polímero é chamada de profundidade de cura ou polimerização, geralmente referindo-se à espessura total que a resina composta é "adequadamente" polimerizada. Durante a passagem da luz no interior do compósito ocorre absorção e dispersão, que são influenciados por inúmeros fatores, entre eles a quantidade, tamanho e tipo de partículas de carga (Shortall et al., 2008), a opacidade do material (Ferracane et al., 1986; Moore et al., 2008), tipo e concentração do fotoiniciador (Leprince et al., 2012), a semelhança do índice de refração entre a matriz resinosa e as partículas de carga (Shortall et al., 2008), fonte fotoativadora e duração da irradiação. Ainda em relação à efetiva polimerização das resinas compostas, tem sido demonstrado que os tempos de fotoativação diferentes podem resultar em diferentes propriedades (Bennett and Watts, 2004; Hogg et al., 2016), sendo que os tempos de exposição recomendados pelos fabricantes para seus materiais podem não ser suficientes para a polimerização adequada, fator fundamental para o procedimento restaurador (Rueggeberg et al., 2009; Busemann et al., 2011; Tarle et al., 2015).

Os problemas relacionados a polimerização insuficiente ou inadequada podem ser tornar ainda mais preocupantes quando o cirurgião-dentista faz restaurações em cavidades com profundidade maior que 4 mm e usando compósitos do tipo *bulk-fill* (Wang et al., 2019; Ferracane, 2011). O acesso limitado do aparelho fotoativador em restaurações posteriores devido ao formato dele e da angulação da ponteira (Soares et al., 2017; Price, 2017; André et al., 2018), o aumento da distância entre a fonte de luz e o material podem influenciam o grau de conversão na base da restauração (Fronza et al, 2015; Shimokawa et al, 2018). Como alternativa, tem sido proposto que a extensão do tempo de exposição à luz pode compensar alguns dos fatores que influenciam negativamente a polimerização do material em profundidade (Lynch et al, 2014; Price et al; 2014; Tarle et al, 2015; Shimokawa et al, 2017; Romano et al, 2020).

Afim de que a polimerização dos materiais resinosos seja adequada nas restaurações, diversos métodos diretos e indiretos têm sido relatados para avaliar a profundidade de cura para garantir a aplicabilidade e longevidade clínica do material restaurador. Para certificar que todo o incremento polimerize de forma eficiente, a Organização Internacional de Normalização introduziu, em 1988, através da ISO 4049, um método para definir a espessura máxima do incremento de resina (Flury et al., 2012). A profundidade de cura é definida como 50% do comprimento total do compósito polimerizado, após a remoção das camadas amolecidas com espátula plástica. Para essa avaliação, a técnica constitui em condensar o material em um molde, fotoativar com 1 milímetro de distância da superfície da resina e após a ativação, a resina não polimerizada e amolecida deve ser removida, mensurando o comprimento da amostra restante e dividindo por dois. (ISO 4049, 2009).

Embora esse teste tenha sido padronizado para que haja uma normatização a fim de avaliar os materiais de maneira homogênea, a técnica para determinação da profundidade de cura quantifica subjetivamente, uma vez que a força aplicada para remoção do material não polimerizado e exercida sobre a amostra pode variar dependendo do operador. Além disso, não é possível identificar visualmente com essa técnica o que está bem polimerizado ou não. Assim, estudos recentes têm criticado a sua aplicabilidade pela dificuldade em padronizar o método da raspagem manual (Leprince et al., 2012) e por sobrestimar a profundidade de cura em comparação com outros testes (Price et al., 2005; Flury et al., 2012; Reis et al., 2017).

A dureza é um método indireto para avaliar o grau de conversão (Rueggeberg et al., 2009; Yan et al., 2010). Quando a razão de dureza obtida entre o topo e base é de até 80%, a conversão é considerada adequada (Bouschlicher et al., 2004). Assim, mensurar a microdureza tem sido provado como bom indicador pra avaliar a profundidade de cura quando comparado ao método da ISO (Reis et al., 2017).

A eficiência de cura também pode ser avaliada pelo método de dissolução em acetona. A imersão de amostras de compósitos após fotoativação em solventes orgânicos se baseia na dissolução da camada pobremente polimerizada ou não polimerizada (Price et al., 2016). A região afetada pelo solvente é facilmente identificada e duas zonas distintas podem ser observadas: uma zona porosa com característica “branco-opaco” na parte mais inferior e uma camada intermediária “borrachoide” entre a fase vítrea e a zona porosa.

O método de dissolução em acetona tem sido considerado para avaliação da profundidade de cura das resinas compostas restauradoras (Corciolani et al., 2008; Price et al., 2016; Romano et al, 2020). Entretanto, existem vários tipos de solventes orgânicos que também podem ser utilizados, mas nunca foram testados. O interesse pela avaliação desses outros solventes baseia-se na interação deles com as diferentes composições monoméricas, que precisam ser avaliadas para escolher o melhor solvente para essa técnica de determinação da profundidade de cura. Portanto, este trabalho tem como objetivo geral avaliar a influência de quatro solventes orgânicos na determinação da profundidade de cura de compósitos restauradores convencionais e do tipo *bulk-fill*.

2. **ARTIGO:** Depth of cure determination of the restorative composites

Artigo submetido ao periódico: Journal of Oral Rehabilitation

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ABSTRACT

The aim of this study was to evaluate the influence of methodologies on the determination of the depth of cure (DOC) and light activation time of restorative composites. Two conventional composites (Beautifil II, Shofu Corp. and Filtek Universal, 3M Oral Care) and two *bulk-fill* composites (Beautifil Bulk Restorative, Shofu Corp. and Filtek One Bulk Fill, 3M Oral Care) were evaluated. Cylindrical samples were fabricated from polyvinylsiloxane matrices with an internal diameter of 6 mm and a height of 12 mm, which were light-cured according to the exposure time recommended by the manufacturer or twice the recommended time. A total of 320 samples were obtained, 40 for each composite at each time, which were analyzed by the ISO 4049 method and by the solvent dissolution method. In the ISO 4049 method, the unpolymerized resin was manually removed with a plastic spatula and for the DOC calculation, the remaining length of the sample was measured with a digital caliper. In the solvent dissolution method, after removing the unpolymerized resin, the samples were distributed among acetone, ethanol, chloroform and tetrahydrofuran (n=10) and stored for 48 hours. After solvent removal, the solvent-affected area (AAOS) and DOC were measured with ImageJ software and the microhardness (KNH) was also analyzed in depth. Data obtained from the AAOS were statistically analyzed by generalized linear models, with a confidence interval of 95%. In the analysis of DOC and KNH data, generalized linear models were used (3 factors: solvent, sample region and exposure time) and Kruskal Wallis test followed by Dunn's method for multiple comparison with the control group (ISO 4049). The ISO 4049 method showed differences compared with solvent-dissolution technique. KNH for all the RBRMs was reduced following a depth increase. The results suggest that increasing the light curing time extended the DOC and KHN. The affected area by solvent is material and solvent dependent. There are differences in the properties of the tested RBRMs and calculation of the depth of cure can vary according to the measurement technique used.

Key words: Depth of cure. Resin composites. Light curing. Hardness. Solvents.

Introduction

Resin-based restorative materials (RBRM) have changed in their composition and mode of use that allow the construction of restorations with good mechanical and esthetic properties. Classically, restorations have been placed in increments of 1.5 to 2 mm thick due to the limited depth of cure (DOC) and to reduce the effects of polymerization shrinkage stress of composites. As an alternative to this incremental technique, new bulk-filling ones were introduced claiming build-up protocol with 4 until 6 mm increment thickness in attempt to facilitate the clinical procedures by reducing the clinical time, the risk of incorporating air bubbles or contaminations between the increments (Miletic et al., 2017; Soto-Montero et al., 2022).

In general, *bulk-fill* composites present specific monomers, new initiating systems and/or filler particles. Most *bulk-fill* composites are translucent materials to allow better light passage through them and increase the DOC (Pires et al., 1993; Barghi et al., 1994; Alrahlah et al., 2014). Although, some reports have suggested that bulk-filling technique is an acceptable technique, (Finan et al., 2013; Alrahlah et al., 2014) others have suggested that bulk-filling curing may produce undercured RBRMs, mainly at depth areas of restoration (Flury et al., 2012; Tiba et al., 2013; Tsujimoto et al., 2017). The ideal DOC is dependent on the radiant energy delivered from light-curing unit and the monomeric conversion of *bulk-fill* composite (Rueggeberg et al., 1994).

The extension in depth of the reaction in relation to the amount of resin monomers converted into the polymeric network following the polymerization reaction characterizes the DOC (Leprince et al., 2012), which means that the resin matrix switches from a glassy to a rubbery state and is an indicator of the maximum thickness of composite that can be used (Price et al., 2014). One method used to determine the polymerization efficiency of resin composites is to measure the DOC as described ISO 4049 standard that is based on measurement with a caliper of the thickness of the RBRM that remains after removal of uncured soft material with a plastic spatula. The procedure of scraping off the uncured resin-based material has been considered difficult to standardize (Flury et al., 2012) and the suitability of the ISO 4049 test to determine the DOC for bulk fill RBCs has been questioned, because it has been reported to overestimate the true DOC (Fronza et al., 2015; Leprince et al., 2012, Price et al., 2016; Romano et al., 2020).

Since hardness measurement has been shown to be a practical method to indirectly determine degree of conversion for a given resin composite (Bouschlicher et al., 2004; Yan et al., 2010; Rueggeberg et al., 2009; Ferracane et al, 2017), hardness profiles can be used to alternatively measure DOC. The bottom-to-top hardness ratio that is at least 80% is considered to be acceptable (Bouschlicher et al., 2004) and more reliable than ISO 4049 test (AlQahtani et al., 2015). Another readily accessible method suggested dissolution in acetone, involving removal of the unreacted monomers and has been preferred by some researchers (Ferracane et al., 2017; Price et al., 2016; Romano et al., 2020). It has been reported that the use of organic solvents allows the operator to differentiate between the properly cured, unaffected composite and a ‘frosty’ region of inadequately polymerized, solvent-affected material (Price et al., 2016) that might correspond to a ‘glassy-rubbery transition’ in the resin matrix (Leprince et al., 2012). Besides the action of organic solvents evidencing the DOC, a standard protocol for application of the solvent-immersion method has not been established and basically the acetone has been used (Price et al, 2016).

The literature is inconsistent regarding the determination of DOC and some results are contradictory (Leprince et al., 2012; Van Ende et al., 2017). How this depth should be determined or what range in variation is considered acceptable, is still a matter of debate. The necessity to establish a reliable method becomes important as a result of studies reporting that some materials might fail to reach the DOC indicated by the manufacturer (Price et al., 2016; Soto-Montero et al., 2020). The purposes of this study were to analyze the influence of organic solvents and light-activation time on measurement method of the DOC of conventional and *bulk-fill* commercial RBRM. The null hypotheses were that: 1- for the same composite, methods of measurement of DOC do not produce different results; 2- extending the light-activation time does not significantly increase the DOC of conventional and *bulk-fill* composites and 3- the type of organic solvent does not influence the resin dissolution and consequently the DOC.

Materials and Methods

Four commercially available RBRM were tested, two restorative products were considered as conventional composites, indicated for increments of ≤ 2 mm thickness: Filtek Universal Restorative (FU) (3M Oral Care, St Paul, MN, USA); and Beautifil II (BE) (Shofu Dental Corp.). The two other materials were classified as high-viscosity, *bulk-fill* materials intended for use in increments of 4–5 mm thickness: Filtek One Bulk Fill (FO) (3M Oral Care, St Paul, MN, USA); and Beautifil Bulk (BB) (Shofu Dental Corp.). The specifications for all four RBCs tested are presented in Table 1.

Forty cylindrical samples were fabricated for each composite and exposure time (four composites and two curing times) using a polyvinyl siloxane impression material mold (Variotime, Kulzer GmbH, Hanau, Germany) of 6 mm diameter and 12 mm height dimensions. Composites were placed into the mold using a single increment, and a 50-µm thick transparent polyester film (K Dent; Quimidrol, Joinville, SC, Brazil) was placed at the bottom and top surfaces of the composite-filled mold. The assembly was lightly pressed between two glass slides to remove the excess of composite. The mold was placed over a white filter-paper (Whatman, Little Chalfont, Bucks, UK) background.

The LED light-curing unit (Elipar DeepCure, 3M Oral Care, St Paul, MN, USA) was fixed in a clamp and the light tip was positioned perpendicular to the aperture of silicone mold touching the upper glass slide (1-mm thick) with the light tip completely covering the sample. The light-curing unit delivered a spectral peak emission at 450 nm (blue). For 10, 20 and 40 seconds the energy densities were 12.77, 25.54 and 51.08 J/cm^2, respectively. Also, the radiant emittance was 1,277 mW/cm^2 and the incident irradiance on the composite surface was 1,203 mW/cm^2. Composite was light-activated following the exposure time recommended by the manufacturer (T1) or double that recommended (T2). Light-curing times for each composite are presented in Table 1.

Table 1. Classifications, brand names, compositions, exposure time (s), shades, and lot numbers of the tested composites.

Classification (Recommended thickness)	Composite /Manufacturer	Composition	Exposure time		Shade	Filler content by % weight (by volume)	Lot number
			T1	T2			
Conventional (Increment of ≤ 2 mm)	Filtek Universal Restorative /3M Oral Care	AUDMA, UDMA, DDDMA, Silane Treated Ceramic, Silane Treated Silica, Silane Treated Zirconia, Ytterbium Fluoride, Titanium Oxide, Water.	10 s	20 s	A3	76.5% (58.4%)	1936000305
	Beautifil II /Shofu Dental Corp.	Bis-GMA, TEGDMA, AluminoFluoro-Borosilicate Glass, Aluminum Oxide, Camphorquinone, Others.	10 s	20 s	A3	83% (69%)	052082
Bulk-fill (Increments of 4-6 mm)	Filtek One Bulk Fill Restorative /3M Oral Care	AUDMA, DDDMA, UDMA Silane Treated Ceramic, Silane Treated Silica, Ytterbium Fluoride, Silane Treated Zirconia, Water.	20 s	40 s	A3	76.5% (58.5%)	NC88136
	Beautifil-Bulk Restorative /Shofu Dental Corp.	Bis-GMA, UDMA, Bis-MPEPP, TEGDMA, Fluoro-Alumino-Silicate Glass, Reaction Initiator, Others.	10 s	20 s	A	87% (74.5%)	012045

Abbreviation: AUDMA- aromatic urethane dimethacrylate; Bis-GMA- bisphenolglycidyl methacrylate; DDDMA- 1,12-dodecane-dimethacrylate; TEGDMA- triethylene glycol dimethacrylate; UDMA- urethane dimethacrylate; T1- light-activation time recommended by the manufacturer; T2, extended light-curing time by 100% to double the exposure.

Measurement of DOC by manual scrapping

Composite samples were removed from the molds and DOC was immediately evaluated using the manual scraping technique (ISO 4049). A plastic spatula (Fuji spatula; GC Corp., Tokyo Japan) was used to remove all the softened composite, until the first sign of resistance to removal them was identified by a single operator. After all softened material had been completely removed, the height of the cylinder was measured to the nearest 0.01 mm using a digital caliper (Pittsburgh 4″ Digital Caliper; Harbor Freight Tools, Calabasas, CA, USA). The measured value was divided by two and recorded in a spreadsheet program (EXCEL 2016; Microsoft, Redmond, WA, USA) as the DOC of the sample using this measuring technique (n = 40).

Measurement of DOC by dissolution technique using organic solvents

The same composite samples used to calculate the DOC by manual scrapping were randomly assigned to immersion in one of the four organic solvents: acetone (ACE), chloroform (CHL), ethanol (ETH) and Tetrahydrofuran (THF) (n = 10). The Table 2 shows the features of each one. The immersed composites were kept in a laboratory fume hood, in the dark, for 48 hours at 22°C (Romano et al, 2020). The composites were removed from the solvent and allowed to air-dry for one hour. Due to the shape of the composite samples after immersion in the solvents, DOC measurements were performed in one of the lateral regions (side) and in the center of the sample (middle).

Table 2. Organic solvents, specifications and manufacturers.

Solvent (Abbreviation)	Molecular formula	Chemical Family	Solubility Parameter $(MPa)^{1/2}$	Manufacturer
2-Propanone or Acetone (ACE)	C_3H_6O	Ketone	19.70	Química Moderna, Barueri, SP, Brazil
Trichloromethane or Chloroform (CHL)	$CHCl_3$	Halogenated hydrocarbon	18.70	Dinâmica, Indaiatuba, SP, Brazil

Ethyl Alcohol or Ethanol (ETH)	C_2H_6O	Alcohol	26.10	Química Moderna, Barueri, SP, Brazil
Tetrahydrofuran (THF)	C_4H_8O	Ether	21.80	Dinâmica, Indaiatuba, SP, Brazil

Measurement of the cured and organic solvent-affected areas

Composite samples were cut into two halves, using a diamond blade that was coupled to a precision cutter machine (IsoMet 1000, Buehler Ltd., Lake Bluff, IL, USA). One of the halves that contained the central region of the sample was used in this part of study. It was reduced to a thickness of 1 mm and used to evaluate the cured and solvent-affected areas. Images of each sample were taken in digital microscope (KH-1300, Hirox Co Ltd, Tokyo, Japan) at x35 magnification to analyze structural integrity and the polymerization pattern of the composites.

The scale bar of the images was used to measure by the line tool (ImageJ 1.8.0 software, National Institutes of Health, USA), which allowed to define the area of each sliced samples. Images were binarised in 8-bit format and the “Default” variation of thresholding was manually adjusted to calculate the percentage of cured and poorly cured areas of the samples (n = 10), after DOC scrapping method.

Microhardness determination

Another half of the samples had their internal area polished with 1200-grit and 2000-grit abrasive papers (Wetordry, 3M, Sorocaba, SP, Brazil) for 1 minute each paper, under water cooling (Aropol, Arotec Ind. e Com. Ltda, Cotia, SP, Brazil) Microhardness tester (Future-Tech FM Corp, Tokyo, Japan) coupled to software (FM-ARS 9000, Future-Tech FM Corp, Tokyo, Japan) was used to obtain Knoop microhardness values (KHN) under a static load of 50 g (0.49 N) for five seconds.

Indentations were performed at 0.5 mm from each external margin (lateral of sample or side) and at the central area of the sample (2 mm from the side measurement or middle). The first microhardness measurement was taken just below the surface or top of the sample and then every 1 mm between measurements up to a depth of 10 mm (n = 10). Images of microhardness mapping for each group was obtained from the KHN values of the samples.

Statistical Analysis

DOC (by manual scrapping and organic solvents) data were analyzed using generalized linear models and Kruskal Wallis followed by Dunn's method for multicomparisons versus the Control (ISO 4049). Factors within the same solvent and time with a significance level of α = 0.05. Solvent-affected area and microhardness analyses were done using generalized linear models to compare different group means and interactions between-subject factors solvent, depth and time and within-subject factor sample region with a significance level of α = 0.05.

Results

DOC measurements

The DOC results obtained with manual scrapping (ISO 4049) and dissolution by organic solvents for all RBRM tested in this study are presented in Tables 3 to 6. Using the manual scrapping technique, double the light-cure time significantly increases DOC.

In general, the increasing the curing time increased the DOC for the dissolution with organic solvents measurements, regardless the resin-based composite used. The exceptions were observed for FU and BE composites, mainly at the middle of the sample. The side region of the samples exhibited lower DOC than those obtained at middle region, regardless the organic solvent used and light-curing time. The ISO 4049 method showed significant differences regarding most groups measured by the dissolution technique with organic solvents for composites FU, BE and BB, regardless of curing time and type of solvent.

Table 3. Depth of cure mean (95% CI) for Filtek Universal composite according to the method of DOC measurement, the type organic solvent used, the evaluated sample region and the light curing time (in mm).

Filtek Universal									
	ACE		CHL		ETH		THF		ISO 4049
	Side	Middle	Side	Middle	Side	Middle	Side	Middle	
T1	2.7 bA (2.5 – 3.0)	3.9 bAB* (3.6 – 4.2)	2.2 bB (2.0 – 2.4)	3.2 bC* (3.0 – 3.5)	2.5 bAB (2.3 – 2.7)	4.3 aA* (4.0 – 4.7)	2.2 bB (2.0 – 2.4)	3.5 bBC* (3.3 – 3.8)	2.7 b (2.7 – 2.8)
T2	3.4 aAB (3.2 – 3.7)	4.8 aA* (4.4 – 5.2)	2.9 aC (2.7 – 3.1)	4.1 aB* (3.8 – 4.4)	3.7 aA (3.4 – 4.0)	4.8 aA* (4.4 – 5.2)	3.0 aBC (2.8 – 3.3)	4.1 aB* (3.8 – 4.5)	3.0 a (2.8 – 3.1)

Lower case letters compare different curing times (T1 and T2) for the same solvent and evaluated sample region (side or middle). Upper case letters compare different solvents for the same time and sample region. (*) Differ from side region for the same solvent and curing time. Gray cells differ from ISO 4049 (Control) within the same time.

Table 4. Depth of cure mean (95% CI) for Beautifil II composite according to the method of DOC measurement, the type organic solvent used, the evaluated sample region and the light curing time (in mm).

Beautifil II									
	ACE		CHL		ETH		THF		ISO 4049
	Side	Middle	Side	Middle	Side	Middle	Side	Middle	
T1	2.2 b (2.0 – 2.4)	3.1 b* (2.8 – 3.4)	2.2 b (2.0 – 2.4)	3.4 a* (3.1 – 3.7)	2.1 b (1.9 – 2.3)	3.6 a* (3.2 – 3.9)	1.9 b (1.7 – 2.1)	3.2 a* (2.9 – 3.6)	2.7 b (2.7 – 2.7)
T2	2.5 a (2.4 – 2.9)	3.8 a* (3.3 – 4.0)	2.6 a (2.5 – 3.0)	3.8 a* (3.2 – 3.9)	2.5 a (2.4 – 2.9)	4.1 a* (3.6 – 4.4)	2.2 a (2.2 – 2.6)	3.7 a* (3.2 – 3.8)	3.1 a (3.0 – 3.1)

There is no statistical difference among organic solvents. Lower case letters compare different curing times (T1 and T2) for the same solvent and evaluated sample region (side or middle). (*) Differ from side region for the same solvent and curing time. Gray cells differ from ISO 4049 (Control) within the same time.

Table 5. Depth of cure mean (95% CI) for Filtek One Bulk Fill composite according to the method of DOC measurement, the type organic solvent used, the evaluated sample region and the light curing time (in mm).

Filtek One Bulk Fill									
	ACE		CHL		ETH		THF		ISO 4049
	Side	Middle	Side	Middle	Side	Middle	Side	Middle	
T1	3.9 bB (3.7 – 4.1)	5.6 bC* (5.2 – 5.9)	3.9 bB (3.6 – 4.1)	6.0 bBC* (5.6 – 6.4)	5.0 bA (4.7 – 5.3)	6.9 bA* (6.5 – 7.4)	4.7 bA (4.4 – 5.0)	6.4 bAB* (6.0 – 6.8)	4.4 b (4.4 – 4.5)
T2	4.8 aBC (4.5 – 5.1)	6.8 aB* (6.4 – 7.2)	4.6 aC (4.3 – 4.8)	6.6 aB* (6.2 – 7.1)	5.5 bA (5.2 – 5.9)	7.8 aA* (7.3 – 8.2)	5.3 aAB (5.0 – 5.6)	7.0 aAB* (6.6 – 7.5)	4.9 a (4.9 – 5.0)

Lower case letters compare different curing times (T1 and T2) for the same solvent and evaluated sample region (side or middle). Upper case letters compare different solvents for the same time and sample region. (*) Differ from side region for the same solvent and curing time. Gray cells differ from ISO 4049 (Control) within the same time.

Table 6. Depth of cure mean (95% CI) for Beautifil Bulk composite according to the method of DOC measurement, the type organic solvent used, the evaluated sample region and the light curing time (in mm).

Beautifil Bulk									
	ACE		CHL		ETH		THF		ISO 4049
	Side	Middle	Side	Middle	Side	Middle	Side	Middle	
T1	3.2 bA (2.9 – 3.5)	5.5 bAB* (5.1 – 6.0)	2.3 bB (2.1 – 2.5)	4.9 bB* (4.5 – 5.3)	3.4 bA (3.1 – 3.6)	6.0 bA* (5.5 – 6.5)	2.9 bA (2.7 – 3.1)	4.9 bB* (4.5 – 5.3)	4.3 b (4.2 – 4.3)
T2	3.9 aAB (3.6- 4.3)	6.3 aAB* (5.7 – 6.8)	2.9 aC (2.6 – 3.1)	5.4 aB* (5.0 – 5.9)	4.5 aA (4.1 – 4.9)	7.1 aA* (6.5 – 7.7)	3.7 aB (3.4 – 4.0)	6.5 aA* (5.9 – 7.0)	4.8 a (4.7 – 4.9)

Lower case letters compare different curing times (T1 and T2) for the same solvent and evaluated sample region (side or middle). Upper case letters compare different solvents for the same time and sample region. (*) Differ from side region for the same solvent and curing time. Gray cells differ from ISO 4049 (Control) within the same time.

In general, FU/CHL and FU/THF showed lower DOC regardless the sample region and light-curing time (Table 3). Also, for FO composite, ACE and CHL yielded lower DOC, regardless the sample region and light-curing time (Table 5). The type of organic solvent used had no influence on DOC for BE (Table 4). For BB, the side region showed the lowest DOC with CHL at both times, while the middle region presented lower DOC with CHL than those obtained with ETH and THF at T2 (Table 6).

Measurement of the organic solvent-affected areas

Table 7 presents information about the affected area by organic solvent. FU and FO showed lower AAOS than those obtained for BE and BB using ACE, regardless the light-curing time. For CHL, ETH and THF at T1, the lowest affected area of the samples was obtained for FO, while for T2 the BB composite presented higher affected area than FU and FO. In general, ETH yielded lower affected area than other organic solvents, except for FU/ACE at T1 and BE/ACE at T2. The increasing of light-curing time reduced the affected area by organic solvent for FU/CHL, FU/ETH, FU/THF and BB/ETH, while for other groups no changes was observed, regardless the organic solvent used.

Table 7. Mean percentage (95% CI) of the area affected by organic solvents for each group, according to the the light curing time.

		ACE	CHL	ETH	THF
T1	FU	22.4 (18.9 – 25.8) b BC	37.9 (32.1 – 43.7) a A	16.9 (14.3 – 19.5) a C	27.7 (23.5 – 32.0) b B
	BE	31.4 (26.6 – 36.2) a A	33.6 (28.5 – 38.8) a A	21.2 (17.9 – 24.4) a B	31.3 (26.6 – 36.1) ab A
	FO	21.5 (18.2 – 24.8) b A	21.4 18.2 – 24.7) b A	11.3 (9.6 – 13.0) b C	15.9 (13.5 – 18.3) c B
	BB	33.3 (28.2 – 38.4) a A	43.2 (36.6 – 49.9) a A	21.8 (18.4 – 25.1) a B	39.7 (33.6 – 45.8) a A
T2	FU	19.9 (16.8 – 22.9) b A	26.5 (22.4 – 30.6) bc A*	11.5 (9.7 – 13.2) c B*	20.6 (17.4 – 23.7) b A*
	BE	27.7 (23.5 – 32.0) a AB	31.0 (26.3 – 35.8) ab A	23.0 (19.5 – 26.5) a B	31.7 (26.8 – 36.5) a A
	FO	17.4 (14.7 – 20.0) b A	21.6 (18.3 – 24.9) c A	10.0 (8.5 – 11.5) c B	16.3 (13.8 – 18.8) b A
	BB	31.2 (26.4 – 36.0) a A	40.1 (34.0 – 46.2) a A	16.6 (14.1 – 19.2) b B*	32.5 (27.5 – 37.4) a A

Lower case letters compare different composites for the same organic solvent and curing time (T1 and T2). Upper case letters compare different solvents for the same composite and curing time. (*) Indicate statistically significant difference when compared to T1 within the same composite and solvent.

The Figure 1 shows a set of representative images of all groups (eight groups: four RBRM and four solvents). The images show an upper area that appears to be properly polymerized and that is prominent in the sample. The lower, whitish area of the sample corresponds to the region attacked by the organic solvent. The representative patterns of the affected area by organic solvent for each RBRM are presented in Figures 2 to 5.

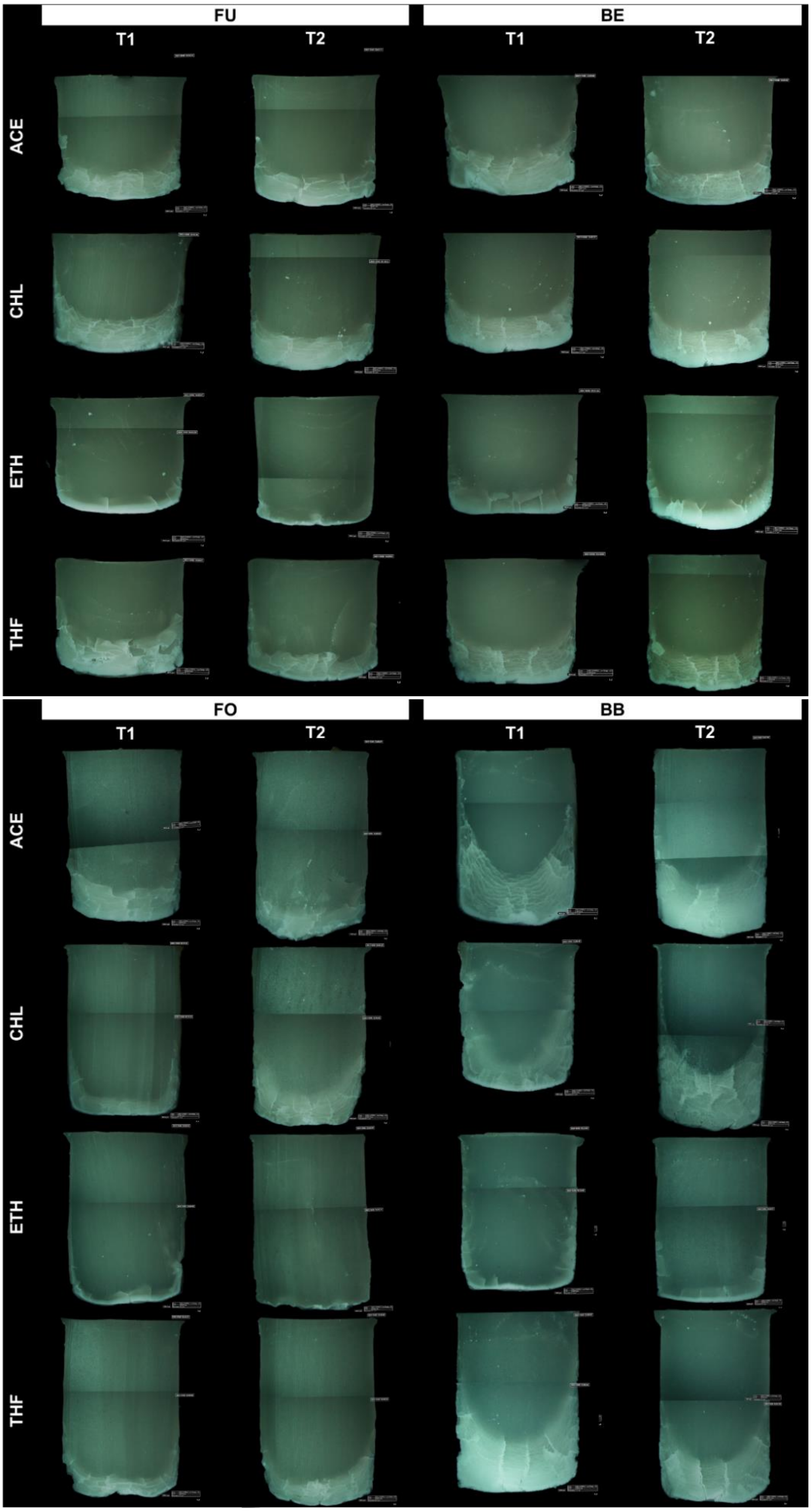

Figure 1. Representative images of composite samples (FU: Filtek Universal, BE: Beautifil II, FO: Filtek One Bulk Fill, Beautifil Bulk: BB) after immersion in the solvents

(ACE: acetone; CHL: chloroform; ETH: ethanol; THF: tetrahydrofuran) (T1: exposure time recommended by the manufacturer; T2: double that recommended). The top region of the samples that was closed to the light source seems properly cured and resistant to the solvent. The basal area of the samples represents the region attacked by the solvent (in white) that seems not fully cured, because of the limited penetration of light into the lower parts of the samples.

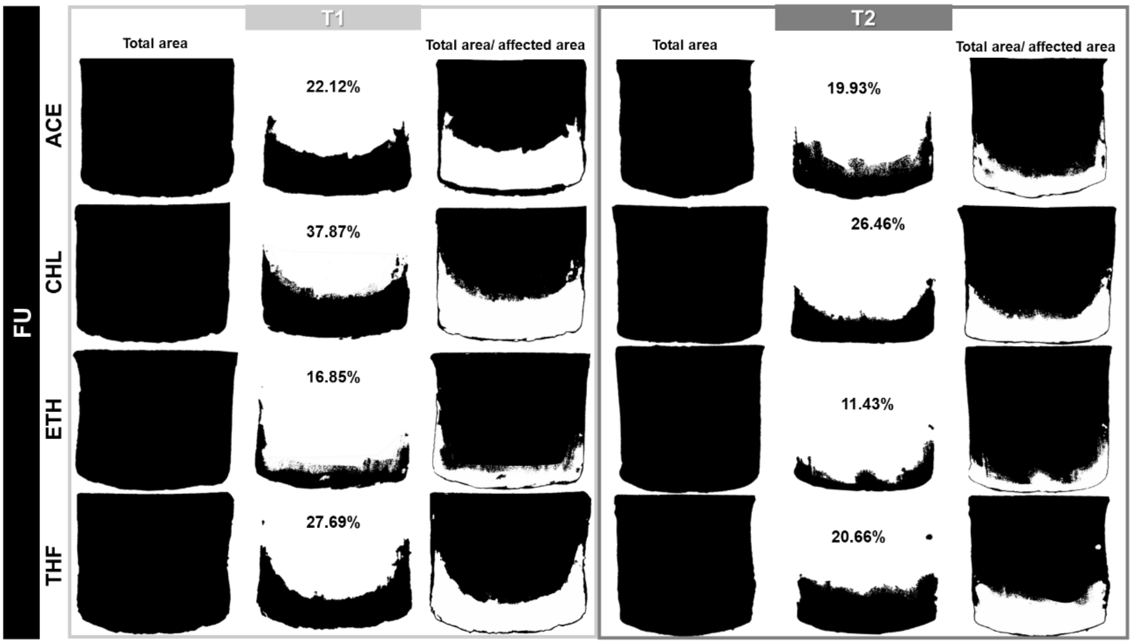

Figure 2. Representative samples of Filtek Universal (FU), according to the organic solvent (ACE: acetone; CHL: chloroform; ETH: ethanol; THF: tetrahydrofuran), curing time (T1: exposure time recommended by the manufacturer; T2: double that recommended). The percentage of the sample area affected by each solvent is presented in the central column that represents the results for each light curing time (T1 and T2).

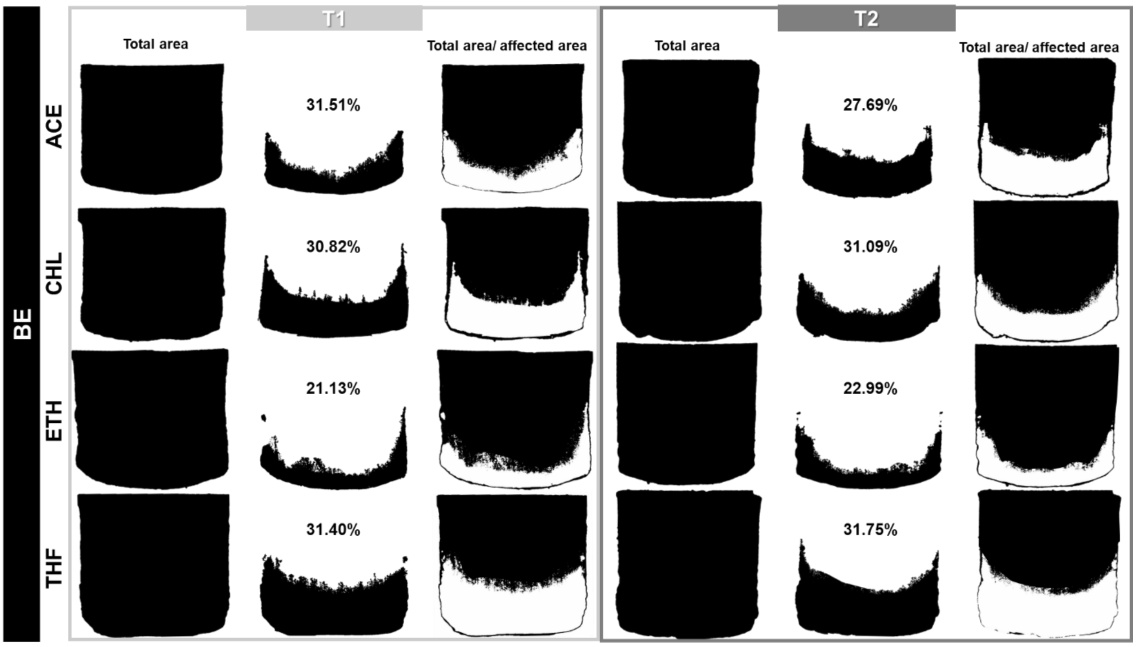

Figure 3. Representative samples of Beautifil II (BE), according to the organic solvent (ACE: acetone; CHL: chloroform; ETH: ethanol; THF: tetrahydrofuran), curing time (T1: exposure time recommended by the manufacturer; T2: double that recommended). The percentage of the sample area affected by each solvent is presented in the central column that represents the results for each light curing time (T1 and T2).

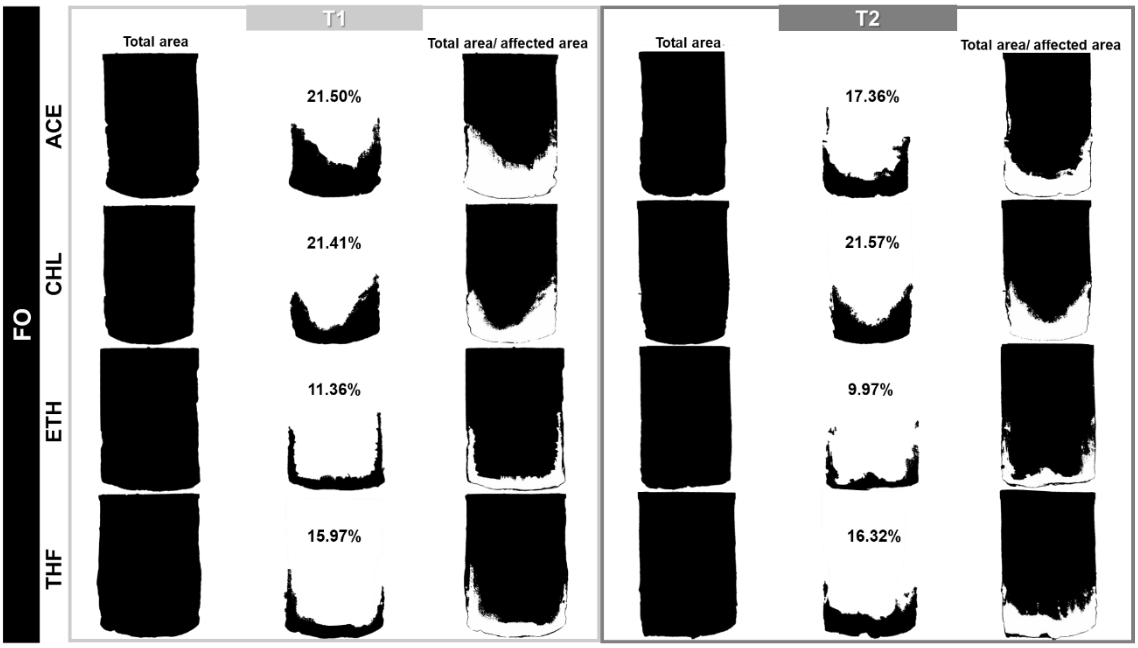

Figure 4. Representative samples of Filtek One Bulk Fill (FO), according to the organic solvent (ACE: acetone; CHL: chloroform; ETH: ethanol; THF: tetrahydrofuran), curing time (T1: exposure time recommended by the manufacturer; T2: double that recommended). The percentage of the sample area affected by each solvent is presented in the central column that represents the results for each light curing time (T1 and T2).

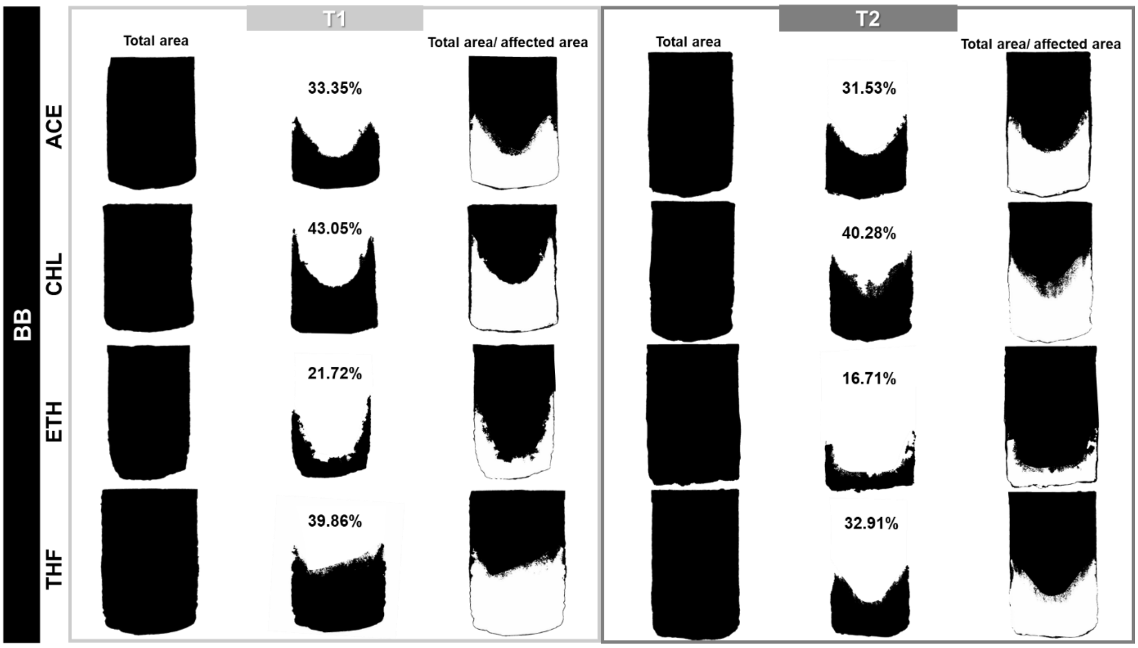

Figure 5. Representative samples of Beautifil Bulk (BB), according to the organic solvent (ACE: acetone; CHL: chloroform; ETH: ethanol; THF: tetrahydrofuran), curing time (T1: exposure time recommended by the manufacturer; T2: double that recommended). The percentage of the sample area affected by each solvent is presented in the central column that represents the results for each light curing time (T1 and T2).

Microhardness determination

The microhardness results for the composites are reported in Tables 8 to 11, according to the type of organic solvent, light curing time, location and depth of evaluation. The hardness mapping for the composites are shown in Figure 6. For some samples, it was not possible to measure the hardness at greater depths due to damage caused by solvents.

In general, for FU at T1, KNH differences in depth started to appear from 3 mm and from 4 mm at T2. The microhardness in the lateral region (or side) and at 5 mm depth for the FU samples treated with CHL showed the lowest KNH. At this same depth, the lateral region differed from the middle one for all organic solvents, but for THF, it already showed this difference at 4 mm depth. Except for ETH, the KNH values at T2 were different from T1 at 5 mm depth (Table 8).

For BE, up to 3 mm in depth the KNH values did not differ regardless the curing time, except for ACE at T1. From 4 mm onwards, the KHN reduced significantly and there was difference among solvents. The side region differed from the middle one at 4 and 5 mm of depth at T1, and at 5 mm at T2, except for ACE. KNH values at T2 differed from T1 at 3 mm for side region with ACE. At 4 mm, the differences were also observed, except for the side region using CHL, and for middle region using ETH and THF. At 5 mm, T2 differed from T1, regardless the organic solvent and sample region (Table 9).

Table 8. Means of FU microhardness (KNH), according to the type of solvent, curing time, location and depth of evaluation.

Filtek Universal (FU)

		ACE		CHL		ETH		THF	
		Side	Middle	Side	Middle	Side	Middle	Side	Middle
T1	TOP	60.6 a A (49.5 – 71.6)	61.6 a A (50.4 – 72.8)	59.2 a A (48.3 – 70.1)	61.2 a A (50.1 – 72.4)	60.1 a A (49.1 – 71.1)	60.5 a A (49.4 – 71.6)	59.0 a A (48.1 – 69.8)	60.7 a A (49.6 – 71.8)
	1 mm	57.5 a A (46.8 – 68.1)	57.8 a A (47.1 – 68.5)	56.1 a A (45.6 – 66.5)	57.1 a A (46.5 – 67.7)	56.1 ab A (45.7 – 66.5)	57.0 a A (46.5 – 67.6)	56.0 a A (45.6 – 66.4)	57.9 ab A (47.2 – 68.6)
	2 mm	52.1 a A (42.3 – 62.0)	53.4 ab A (43.3 – 63.4)	51.4 a A (41.6 – 61.2)	53.9 ab A (43.8 – 64.0)	52.9 ab A (42.9 – 62.9)	54.0 a A (43.9 – 64.2)	50.8 a A (41.1 – 60.5)	53.9 ab A (43.7 – 64.0)
	3 mm	41.1 ab A (32.8 – 49.4)	47.2 ab A (38.0 – 56.3)	41.3 ab A (33.0 – 49.6)	46.8 ab A (37.7 – 55.9)	48.3 ab A (38.9 – 57.6)	50.9 a A (41.2 – 60.6)	38.9 ab A (31.0 – 46.8)	48.6 ab A (39.3 – 58.0)
	4 mm	26.7 b A (20.7 – 32.6)	35.7 b A (28.3 – 43.2)	29.6 b AB (23.2 – 36.1)	36.6 b A (29.0 – 44.1)	36.7 b A (29.1 – 44.4)	42.6 a A (34.1 – 51.1)	26.6 b A (20.6 – 32.6)	39.6 b A (31.5 – 47.6)
	5 mm	7.4 c B (5.1 – 9.7)	16.9 c AB (12.7 – 21.2)	2.5 c C (1.5 – 3.6)	13.5 c B (9.9 – 17.1)	16.9 c A (12.1 – 20.3)	25.3 b A (19.6 – 31.1)	4.7 c BC (3.1 – 6.4)	20.2 c AB (15.3 – 25.0)
	6 mm	-	-	-	-	-	-	-	-
T2	TOP	59.8 a A (48.8 – 70.7)	61.4 a A (50.2 – 72.5)	59.6 a A (48.7 – 70.5)	63.6 a A (52.2 – 75.1)	60.6 a A (49.2 – 71.6)	61.4 a A (50.3 – 72.6)	59.9 a A (48.9 – 70.8)	62.0 a A (50.5- 73.0)
	1 mm	56.7 a A (46.2 – 67.3)	57.3 a A (46.7 – 68.0)	56.4 a A (45.9 – 66.9)	58.6 ab A (47.8 – 69.4)	57.2 a A (46.6 – 67.8)	57.1 a A (46.5 – 67.7)	57.4 a A (46.7 – 68.0)	59.0 a A (48.1 – 69.7)

	2 mm	52.7 ab A	54.2 a A	52.5 a A	55.6 ab A	54.0 a A	53.8 a A	55.1 a A	57.1 a A
		(42.8 – 62.7)	(44.0 – 64.4)	(42. 5 – 62.4)	(45.2 – 66.0)	(43.9 – 64.2)	(43.7 – 63.9)	(44.8 – 65.4)	(46.4 – 67.5)
	3 mm	46.0 ab A	49.3 a A	43.4 ab A	51.2 ab A	49.3 a A	49.9 a A	48.7 a A	52.4 a A
		(37.0 – 55.0)	(39.9 – 58.8)	(34.8 – 52.0)	(41.5 – 61.0)	(39.8- 58.8)	(40.3 – 59.5)	(39.3 – 58.1)	(42.3 – 62.0)
	4 mm	35.8 bc A	42.5 ab A	31.6 b A	42.1 bc A	39.6 ab A	44.9 ab A	40.2 a A*	44.5 ab A
		(28.4 – 43.3)	(34.0 – 51.0)	(24.8 – 38.4)	(33.7 – 50.5)	(31.5 – 47.6)	(36.0 – 53.7)	(32.1 – 48.4)	(35.2 – 52.5)
	5 mm	23.1 c A*	28.2 b A*	12.2 c B*	27.0 c A*	20.5 b A	30.6 b A	23.2 b A*	32.1 b A*
		(17.7 – 28.5)	(22.0- 34.4)	(8.9 – 15.6)	(21.0 – 33.0)	(15.6 – 25.4)	(24.0 – 37.3)	(17.9 – 28.6)	(25.2 – 38.9)
	6 mm		2.1 c A			1.5 c	4.0 c A		2.2 c A
		-	(1.2 – 3.0)	-	-	(0.8 – 2.2)	(2.5 – 5.4)	-	(1.3 – 3.1)

Lower case letters compare different depths for the same solvent, evaluated sample region and curing time. Upper case letters compare different solvents for the same depth, evaluated sample region and curing time. Connective bars indicate significant difference between sample regions (side and middle) within the same solvent and curing time. (*) Differ from T1 within the same solvent, depth and sample region.

Table 9. Means of BE microhardness (KNH), according to the type of solvent, curing time, location and depth of evaluation.

		Beautifil II (BE)							
		ACE		CHL		ETH		THF	
		Side	Middle	Side	Middle	Side	Middle	Side	Middle
T1	TOP	61.1 a A	61.6 a A	60.7 a A	62.1 a A	58.5 a A	61.0 a A	58.9 a A	60.7 a A
		(51.3 – 71.0)	(51.7 – 71.5)	(50.9 – 70.5)	(52.1 – 72.0)	(49.0 – 68.0)	(51.2 – 70.9)	(49.3 – 68.4)	(50.9 – 70.5)
	1 mm	55.9 a A	56.5 a A	58.3 a A	59.6 a A	54.3 a A	58.1 a A	57.1 a A	58.7 a A
		(46.7 – 65.1)	(47.3 – 65.8)	(48.8 – 67.8)	(49.9 – 69.3)	(45.3 – 63.4)	(48.6 – 67.6)	(47.8 – 66.5)	(49.1 – 68.2)
	2 mm	50.5 ab A	51.2 a A	54.9 a A	57.5 a A	49.7 a A	53.9 ab A	53.9 a A	56.2 a A
		(41.9 – 59.0)	(42.6 – 59.9)	(45.8 – 63.9)	(48.1 – 66.9)	(41.2 – 58.1)	(44.9 – 62.8)	(45.0 – 62.9)	(47.0 – 65.5)
	3 mm	35.0 b A	44.9 a A	44.2 a A	53.1 a A	42.0 a A	49.0 ab A	45.8 a A	51.7 a A
		(28.5 – 41.4)	(37.1 – 52.7)	(36.5 – 52.0)	(44.2 – 61.9)	(34.6 – 49.4)	(40.6 – 57.3)	(37.9 – 53.8)	(43.0 – 60.4)
	4 mm	17.6 c B	26.1 b B	26.6 b A	35.0 b AB	23.7 b AB	38.9 b A	23.1 b AB	34.1 b AB
		(13.7 – 21.5)	(20.9 – 31.3)	(21.3 – 31.9)	(28.5 – 41.4)	(18.8 – 28.5)	(31.9 – 45.9)	(18.4 – 27.8)	(27.8 – 40.5)
	5 mm	5.1 d AB	10.6 c B	5.5 c AB	10.8 c B	8.2 c A	19.7 c A	4.1 c B	13.8 c AB
		(3.6 – 6.6)	(8.0 – 13.2)	(3.9 – 7.1)	(8.1 – 13.4)	(6.0 – 10.4)	(15.5 – 23.9)	(2.8 – 5.4)	(10.6 – 17.0)
	6 mm		0.8 d						
		-	(0.4 – 1.2)	-	-	-	-	-	-

T2	TOP	61.1 a A (51.3 – 71.0)	62.4 a A (52.4 – 72.4)	60.3 a A (50.5 – 70.0)	61.6 a A (51.7 – 71.5)	60.7 a A (50.9 – 70.4)	62.3 a A (52.3 – 72.3)	59.0 a A (49.5 – 68.6)	61.4 a A (51.5 – 71.3)
	1 mm	56.7 a A (47.4 – 66.1)	58.6 a A (49.1 – 68.1)	58.2 a A (48-7 – 67.7)	59.8 a A (50.1 – 69.5)	56.7 ab A (47.4 – 66.0)	58.8 a A (49.3 – 68.4)	57.1 a A (47.8 – 66.5)	59.8 ab A (50.1 – 69.5)
	2 mm	53.1 a A (44.2 – 62.0)	55.1 ab A (46.0 – 64.2)	55.6 a A (46.5 – 64.8)	57.9 a A (48.4 – 67.3)	53.4 ab A (44.5 – 62.3)	55.6 a A (46.4 – 64.7)	54.4 a A (45.3 – 63.4)	57.4 ab A (48.0 – 66.8)
	3 mm	45.4 ab A* (37.5 – 53.2)	50.1 ab A (41.7 – 58.6)	51.4 a A (42.7 – 60.0)	54.7 a A (45.6 – 63.8)	48.1 ab A (39.9 – 56.3)	52.8 a A (44.0 – 61.6)	48.8 ab A (40.5 – 57.2)	54.2 ab A (45.2 – 63.2)
	4 mm	31.5 b A* (25.5 – 37.5)	38.8 b A* (31.8 – 45.8)	34.0 b A (27.7 – 40.3)	47.7 a A* (39.6 – 55.9)	39.9 b A* (32.7 – 47.0)	44.6 ab A (36.9 – 52.4)	33.8 b A* (27.5 – 40.1)	42.5 b A (35.0 – 50.0)
	5 mm	16.5 c AB* (12.8 – 20.2)	20.8 c A* (16.4 – 25.2)	16.3 c AB* (12.7 – 20.0)	24.6 b A* (19.6 – 29.5)	20.3 c A* (16.0 – 24.6)	29.8 b A* (24.0 – 35.5)	11.0 c B* (8.3 – 13.7)	24.7 c A* (19.7 – 29.7)
	6 mm	-	-	-	-	-	4.7 c (3.3 – 6.1)	-	-

Lower case letters compare different depths for the same solvent, evaluated sample region and curing time. Upper case letters compare different solvents for the same depth, evaluated sample region and curing time. Connective bars indicate significant difference between sample regions (side and middle) within the same solvent and curing time. (*) Differ from T1 within the same solvent, depth and sample region.

Table 10. Means of FO microhardness (KNH), according to the type of solvent, curing time, location and depth of evaluation.

Filtek One Bulk Fill (FO)

		ACE		CHL		ETH		THF	
		Side	Middle	Side	Middle	Side	Middle	Side	Middle
T1	TOP	60.0 a A	61.2 a A	60.3 a A	61.6 a A	60.8 a A	62.1 a A	60.1 a A	61.3 a A
		(51.3 – 71.2)	(52.3 – 72.4)	(51.6 – 71.5)	(52.7 – 72.9)	(52.0 – 72.0)	(53.2 – 73.5)	(51.4 – 71.2)	(52.4 – 72.5)
	1 mm	56.3 ab A	57.8 ab A	56.1 ab A	58.9 a A	58.2 ab A	60.2 a A	56.7 ab A	58.3 ab A
		(48.0 – 66.9)	(49.4 – 68.7)	(47.8 – 66.7)	(50.3 – 69.8)	(49.7 – 69.1)	(51.5 – 71.3)	(48.4 – 67.4)	(49.8 – 69.2)
	2 mm	53.2 ab A	55.2 ab A	53.9 ab A	56.2 ab A	55.5 ab A	57.4 ab A	53.1 ab A	54.7 abc A
		(45.2 – 63.4)	(47.0 – 65.6)	(45.9 – 64.2)	(47.9 – 66.8)	(47.3 – 66.0)	(49.0 – 68.2)	(45.2 – 63.3)	(46.6 – 65.2)
	3 mm	48.8 abc A	51.5 abc A	48.6 abc A	53.1 ab A	51.6 ab A	54.0 ab A	49.3 ab A	51.3 abc A
		(41.4 – 58.4)	(43.8 – 61.5)	(41.3 – 58.2)	(45.2 – 63.3)	(43.9 – 61.6)	(45.9 – 64.3)	(41.9 – 59.0)	(43.6 – 61.2)
	4 mm	44.7 abc A	47.7 abc A	43.6 abc A	49.3 ab A	47.2 abc A	50.3 ab A	46.1 ab A	47.4 abc A
		(37.8 – 53.7)	(40.4 – 57.1)	(36.8 – 52.5)	(41.9 – 59.0)	(40.0 – 56.6)	(42.7 – 60.1)	(39.0 – 55.3)	(40.2 – 56.8)
	5 mm	39.8 bc A	44.1 abc A	37.5 bcd A	44.2 abc A	42.9 abc A	46.7 ab A	41.9 abc A	44.3 abc A
		(33.5 – 48.1)	(37.3 – 53.1)	(31.5 – 45.5)	(37.3 – 53.1)	(36.2 – 51.6)	(39.5 – 56.0)	(35.4 – 50.6)	(37.5 – 53.3)
	6 mm	33.8 cd A	40.4 bc A	32.4 cd A	38.9 bc A	39.3 bc A	43.0 abc A	37.9 bc A	40.6 bc A
		(28.3 – 41.2)	(34.0 – 48.8)	(27.0 – 39.5)	(32.7 – 47.1)	(33.1 – 47.6)	(36.3 – 51.7)	(31.8 – 45.9)	(34.2 – 49.0)

	7 mm	24.5 d A	35.0 c A	25.3 d A	30.5 cd A	32.3 c A	38.6 bc A	29.7 c A	36.0 cd A
		(20.2 – 30.4)	(29.3 – 42.6)	(20.8 – 31.3)	(25.4 – 37.4)	(26.9 – 39.4)	(32.4 – 46.7)	(24.6 – 36.4)	(30.2 – 43.7)
	8 mm	11.3 e A	21.5 d A	14.9 e A	23.0 d A	15.5 d A	28.0 c A	10.5 d A	24.6 d A
		(9.0 – 14.8)	(17.6 – 26.9)	(12.0 – 19.0)	(18.9 – 28.6)	(12.5 – 19.8)	(23.2 – 34.4)	(8.3 – 13.8)	(20.3 – 30.5)
	9 mm		2.4 e B			4.5 e	5.2 d A		0.8 e C
		-	(1.7 – 3.5)	-	-	(3.3 – 6.3)	(3.9 – 7.2)	-	(0.5 – 1.4)
	10 mm	-	-	-	-	-	-	-	-
T2	TOP	60.5 a A	61.6 a A	60.4 a A	61.6 a A	60.5 a A	62.3 a A	60.4 a A	62.1 a A
		(51.7 – 71.6)	(52.7 – 72.9)	(51.6 – 71.5)	(52.7 – 73.0)	(51.8 – 71.7)	(53.3 – 73.7)	(51.6 – 71.5)	(53.2 – 73.5)
	1 mm	56.9 ab A	58.5 ab A	56.8 a A	58.1 ab A	56.7 a A	58.7 a A	56.9 ab A	59.3 a A
		(48.6 – 67.6)	(50.0 – 69.4)	(48.5 – 67.5)	(49.6 – 69.0)	(48.4 – 67.4)	(50.2 – 69.7)	(48.6 – 67.7)	(50.6 – 70.3)
	2 mm	53.4 ab A	55.6 ab A	53.0 ab A	54.7 ab A	53.8 ab A	55.9 ab A	53.1 ab A	56.0 ab A
		(45.5 – 63.3)	(47.4 – 66.2)	(45.1 – 63.2)	(46.6 – 65.1)	(45.8 – 64.1)	(47.7 – 66.5)	(45.4 – 63.5)	(47.7 – 66.6)
	3 mm	49.4 abc A	52.3 abc A	48.8 ab A	51.2 abc A	51.3 ab A	53.0 ab A	49.5 abc A	52.2 ab A
		(41.9 – 59.1)	(44.5 – 62.4)	(41.4 – 58.4)	(43.5 – 61.1.)	(43.6 – 61.2)	(45.1 – 63.2)	(42.2 – 59.4)	(44.4 – 62.2)
	4 mm	45.0 abcd A	48.3 abc A	45.1 ab A	46.9 abc A	45.8 abc A	49.8 abc A	46.4 abc A	48.8 abc A
		(38.1 – 54.1)	(40.9 – 57.8)	(38.2 – 54.2)	(39.8 – 56.3)	(38.8 – 55.0)	(42.3 – 59.6)	(39.3 – 55.7)	(41.4 – 58.4)
	5 mm	38.7 bcd A	43.6 abcd A	40.5 abc A	43.1 abcd A	42.3 abc A	46.6 abc A	42.2 abc A	45.3 abc A

	(32.5 – 46.8)	(36.8 – 52.4)	(34.1 – 48.9)	(36.4 – 51.9)	(35.7 – 51.0)	(39.4 – 55.9)	(35.7 – 51.0)	(38.3 – 54.3)
6 mm	34.5 cd A	39.7 bcd A	35.1 bc A	39.0 bcd A	37.1 bc A	41.9 abc A	38.0 bcd A	41.5 abc A
	(28.8 – 42.0)	(33.4 – 47.9)	(29.4 – 42.7)	(32.8 – 47.2)	(31.1 – 45.0)	(35.3 – 50.5)	(31.9 – 46.0)	(34.9 – 50.0)
7 mm	29.9 de A	35.1 cd A	27.3 cd A	34.8 cd A	32.4 cd A	37.8 bc A	33.6 cd A	37.8 bc A
	(24.8 – 36.6)	(29.4 – 42.7)	(22.6 – 33.7)	(29.1 – 42.4)	(27.0 – 39.6)	(31.8 – 45.8)	(28.1 – 40.9)	(31.7 – 45.8)
8 mm	19.7 e A*	28.4 d A	18.7 d A	28.7 d A	22.7 d A*	33.7 cd A	24.8 d A*	32.4 c A
	(16.1 – 24.8)	(23.6 – 34.0)	(15.2 – 23.6)	(23.8 – 35.5)	(18.6 – 28.3)	(28.1 – 41.0)	(20.1 – 30.3)	(27.1 – 39.6)
9 mm	5.5 f B	15.6 e A*	8.1 c AB	16.0 e A	12.0 e A*	21.1 d A*	6.3 e B	17.9 d A*
	(4.1 – 7.5)	(12.6 – 19.9)	(6.3 – 10.8)	(12.9 – 20.3)	(9.6 – 15.6)	(17.3 – 26.4)	(4.8 – 8.5)	(14.5 – 22.6)
10 mm	-	-	-	-	-	-	-	-

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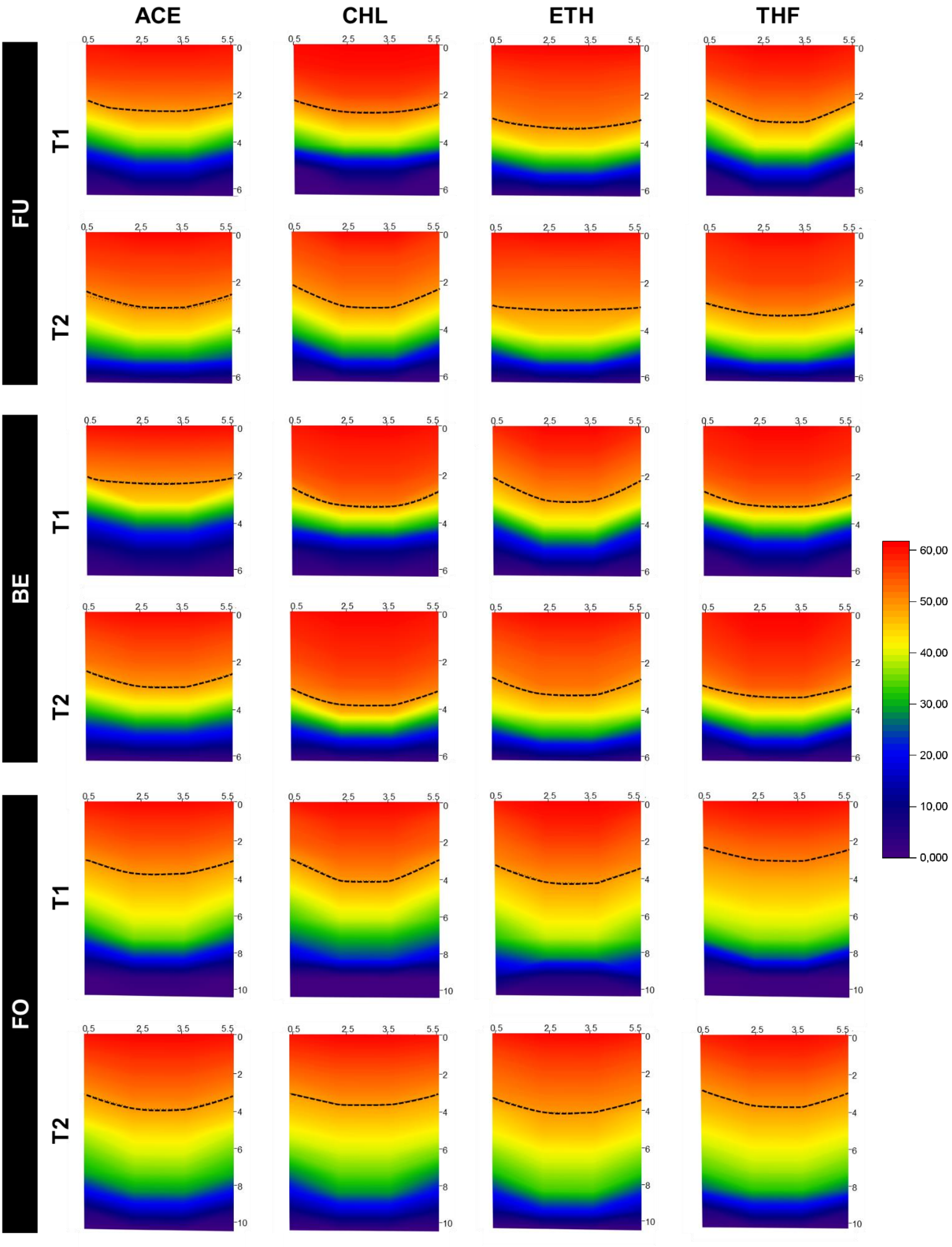
Table 11. Means of BB microhardness (KNH), according to the type of solvent, curing time, location and depth of evaluation.

Beautifil Bulk (BB)

		ACE		CHL		ETH		THF	
		Side	Middle	Side	Middle	Side	Middle	Side	Middle
T1	TOP	59.5 a A	60.4 a A	59.5 a A	60.0 a A	59.4 a A	60.6 a A	58.5 a A	59.6 a A
		(50.0 – 72.0)	(50.8 – 73.0)	(50.0 – 72.0)	(50.4 – 72.6)	(49.9 – 71.9)	(51.0 – 73.3)	(49.2 – 70.2)	(50.1 – 72.1)
	1 mm	55.7 ab A	56.5 a A	55.9 a A	56.9 a A	55.4 ab A	57.1 a A	54.7 ab A	56.5 ab A
		(46.7 – 67.6)	(47.4 – 68.6)	(46.8 – 67.8)	(47.8 – 69.1)	(46.4 – 67.3)	(47.9 – 69.3)	(45.8 – 66.5)	(47.4 – 68.6)
	2 mm	51.8 ab A	53.4 ab A	52.1 a A	52.9 a A	52.1 ab A	53.9 ab A	49.3 ab A	52.1 ab A
		(43.3 – 63.1)	(44.7 - 65.0)	(43.5 – 63.5)	(44.2 – 64.4)	(43.5 – 63.4)	(45.1 – 65.6)	(41.1 – 60.3)	(43.6 – 63.5)
	3 mm	47.4 ab A	49.8 ab A	46.0 ab A	48.6 ab A	47.8 ab A	49.3 ab A	43.7 abc A	46.0 abc A
		(39.5 – 58.1)	(41.5 – 60.8)	(38.2 – 56.4)	(40.5 – 59.4)	(39.8 – 58.6)	(41.8 – 60.2)	(36.3 – 53.8)	(38.2 – 56.4)
	4 mm	42.0 abc A	46.0 ab A	39.0 ab A	44.5 ab A	43.6 abc A	44.8 ab A	36.3 bcd A	41.4 abc A
		(34.7 – 51.7)	(38.2 – 56.4)	(32.2 – 48.2)	(36.9 – 54.6)	(36.1 – 53.6)	(37.2 – 55.0)	(29.9 – 45.2)	(34.2 – 51.0)
	5 mm	36.0 bc A	41.3 ab A	31.1 bc A	39.4 ab A	36.3 bc A	40.5 abc A	28.2 cd A	35.7 bc A
		(29.6 – 44.8)	(34.2 – 50.9)	(25.4 – 39.0)	(32.6 – 48.8)	(29.8 – 45.1)	(33.5 – 50.1)	(22.9 – 35.6)	(29.4 – 44.5)
	6 mm	27.3 c A	34.7 bc A	21.2 c A	32.3 bc A	28.7 cd A	34.9 bc A	22.7 de A	28.4 cd A
		(22.2 – 34.6)	(28.5 – 43.2)	(17.0 – 27.3)	(26.4 – 40.4)	(23.3 – 36.1)	(28.6 – 43.4)	(18.3 – 29.1)	(23.0 – 35.8)
	7 mm	13.4 d AB	23.6 c A	11.2 d B	21.0 c A	19.3 d A	24.8 c A	15.8 e AB	19.2 d A
		(10.4 – 17.7)	(19.0 – 30.1)	(8.7 – 15.1)	(16.8 – 27.0)	(15.4 – 25.0)	(20.0 – 31.6)	(12.4 – 20.7)	(15.3 – 24.9)
	8 mm	3.5 e A	11.5 d A	3.0 e A		5.3 e A	8.1 d A	3.5 f A	9.3 e A
		(2.5 – 5.3)	(8.9 – 15.4)	(2.1 – 4.6)		(3.9 – 7.6)	(6.1 – 11.2)	(2.5 – 5.3)	(7.1 – 12.7)
	9 mm	-	-	-	-	-	-	-	-
	10 mm	-	-	-	-	-	-	-	-

T2	TOP	60.2 a A (50.6 – 72.8)	61.1 a A (51.4 – 73.8)	59.8 a A (50.3 – 72.4)	61.1 a A (51.4 – 73.9)	60.1 a A (50.5 – 72.7)	61.1 a A (51.4 – 73.9)	59.9 a A (50.4 – 72.5)	61.1 a A (51.4 – 73.9)
	1 mm	57.1 ab A (47.9 – 69.2)	58.4 a A (49.0 – 70.7)	56.1 ab A (47.0 – 68.1)	57.6 a A (48.3 – 69.8)	55.8 a A (46.8 – 67.8)	58.0 ab A (48.7 – 70.2)	56.2 ab A (47.0 – 68.1)	57.4 a A (48.2 – 69.6)
	2 mm	52.8 ab A (44.2 – 64.3)	55.2 ab A (46.3 – 67.1)	52.4 ab A (43.8 – 63.9)	54.0 ab A (45.2 – 65.6)	52.2 a A (43.6 – 63.6)	54.3 ab A (45.4 – 66.0)	52.0 ab A (43.4 – 63.3)	53.9 ab A (45.1 – 65.5)
	3 mm	48.2 abc A (40.1 – 58.9)	51.0 ab A (42.6 – 62.2)	48.1 ab A (40.0 – 58.8)	49.2 ab A (41.0 – 60.1)	48.3 ab A (40.2 – 59.1)	50.5 abc A (42.1 – 61.6)	46.9 abc A (39.0 – 57.4)	49.7 ab A (41.4 – 60.6)
	4 mm	44.0 abc A (36.5 – 54.1)	46.9 abc A (39.0 – 57.4)	43.1 abc A (35.7 – 53.0)	45.2 ab A (37.6 – 55.5)	43.8 ab A (36.3 – 53.8)	47.0 abc A (39.1 – 57.5)	42.6 abc A (35.2 – 52.3)	45.5 abc A (37.8 – 55.8)
	5 mm	37.6 bc A (30.9 – 46.6)	42.2 abc A (35.0 – 52.0)	37.4 bc A (30.8 – 46.4)	39.7 abc A (32.8 – 49.1)	38.3 abc A (31.5 – 47.4)	42.9 abc A (35.6 – 52.8)	37.5 bc A (30.6 – 46.2)	40.5 abc A (33.4 – 50.0)
	6 mm	31.4 cd A (25.6 – 39.3)	35.9 bc A (29.5 – 44.7)	29.2 cd A (23.8 – 36.7)	34.0 bc A (27.8 – 42.4)	31.8 bc A (26.0 – 39.8)	37.5 bc A (30.9 – 46.5)	29.9 cd A (24.2 – 37.3)	35.4 bc A (29.1 – 44.1)
	7 mm	19.3 d A* (15.4 – 25.0)	29.8 cd A (24.3 – 37.5)	19.7 d A* (15.7 – 25.4)	25.7 c A (20.8 – 32.6)	24.1 c A (19.4 – 30.7)	31.2 cd A (25.4 – 39.1)	21.4 d A (17.3 – 27.7)	28.3 cd A* (23.0 – 35.7)
	8 mm	6.3 e A* (4.7 – 9.0)	17.7 d AB* (14.1 – 23.0)	7.7 e A* (5.8 – 10.8)	13.8 d B* (10.8 – 18.3)	11.2 d A* (8.6 – 15.1)	24.1 d A* (19.4 – 30.7)	11.3 e A* (8.7 – 15.2)	18.1 d AB* (14.4 – 23.5)
	9 mm	0.6 f B (0.4 – 1.2)	5.4 e A (4.0 – 7.7)	0.8 f B (0.5 – 1.5)	1.0 e B (0.7 – 1.8)	3.8 e A (2.7 – 5.7)	6.4 e A (4.8 – 9.0)	-	-
	10 mm	-	-	-	-	-	-	-	-

Lower case letters compare different depth for the same solvent, evaluated sample region and curing time. Upper case letters compare different solvents for the same depth, evaluated sample region and curing time. Connective bars indicate significant difference between sample regions (side and middle) within the same solvent and curing time. (*) Differ from T1 within the same solvent, depth and sample region.


ACE
CHL
ETH
THF
FU
BE
FO
T1
T2
0.5
2.5
3.5
5.5
0
2
4
6
8
10
60,00
50,00
40,00
30,00
20,00
10,00
0,000

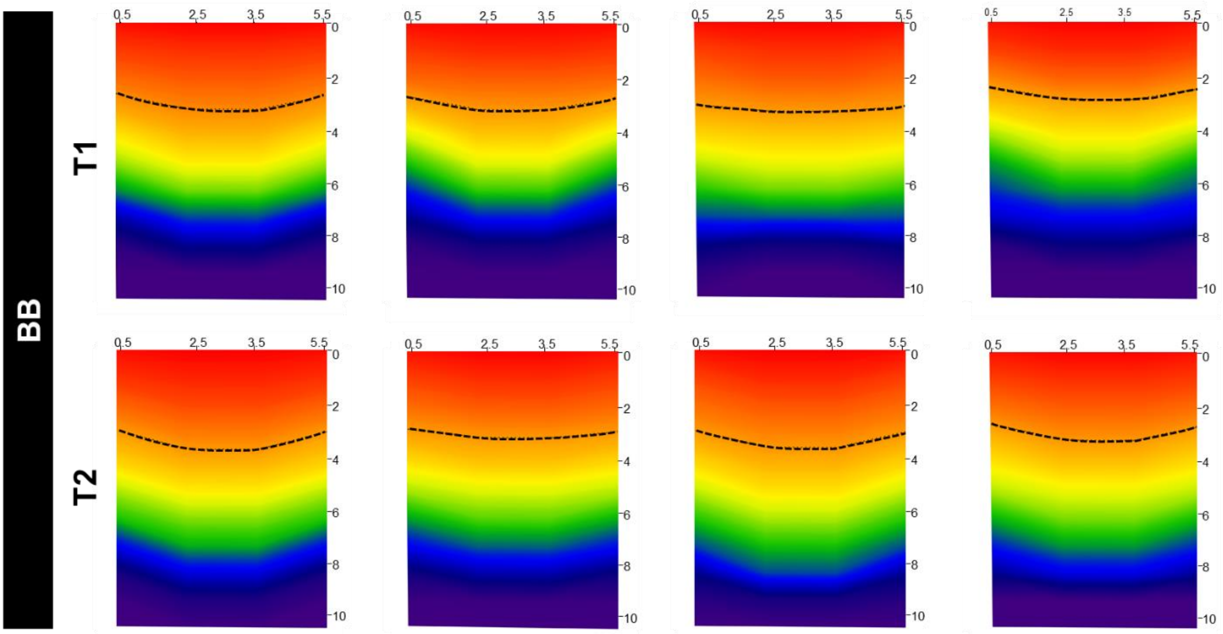


Figure 6. Microhardness map of each RBRM. The 80% of maximum hardness is represented by the dashed line.

For FO at T1, the KHN values from the side region for ACE and CHL were not significantly different up to 4 mm and for ETH and THF, were up to 5 mm of depth. From the middle region, except for ETH, the KNH differences in depth started to appear from 5 mm. The side region showed the lowest KHN at 8 mm for all the organic solvents used at T1. From a depth of 5 mm on the side of the sample, a statistical difference was observed at T2 in relation to the top of the sample for ACE, while for the other solvents from 6 mm. For the central region of the samples, a significant difference in relation to the top of them was observed with ACE and CHL from 6 mm in depth, while for ETH and THF from 7 mm in depth. The KHN difference between side and middle was noted from the 7 and 8 mm depth. CHL was the only solvent used that did not yield significant difference between T1 and T2 (Table 10).

For BB at T1, the decreasing in KNH values started at 3 mm from the side region and at 4 mm from the middle for THF. At T2, all organic solvents did not yield different KNH values up to 5 mm from the middle region. The KHN comparison between side and middle regions at T1 were significantly different at 7 and 8 mm for ACE, at 6 to 8 mm for CHL, and, at 8 mm for THF, while no significant difference was observed for ETH. At T2, these differences can be seen at 7 to 9 mm for ACE, at 8 mm for CHL and THF and at 8 and 9 mm for ETH. Comparing the light-curing times, it can be seen that the double of exposure time resulted in KNH values significantly different at 7 mm from the side region for ACE and CHL and from middle region for THF, while at 8 mm the difference obtained was regardless the organic solvent used and evaluated sample region (Table 11).

In general, the microhardness obtained close to 2 mm depth for all tested conventional RBRM reached more than 80% of the maximum hardness value. The extension of the light-curing time produces the same bottom-to-top ratio at 3 mm at the middle of the sample. For *bulk-fill* resins, the side region of FO generally obtains the value of 80% at 3 mm of depth, while for the central region of the sample varied between 3 to 4 mm, depending on the solvent used, thus twice the light-curing time did not produce in higher depth. For BB at T1, the value of 80% was reached approximately at 3 mm in the central region of the samples, while for the side region this ratio was obtained below 2 mm depth, regardless the solvent used. For T2, the extension of curing time did not increase significantly this ratio at greater depths.

Discussion

Although many studies have compared the DOC of RBRMs using different methods to determine if the manufacturer’s recommendation of curing up to depth of 4 to 5 mm is correct (Finan et al., 2013; Van Ende et al., 2017; Reis et al., 2017; Algamaiah et al., 2020; Kaisarly et al., 2021), the literature is still inconsistent and contradictory regarding the determination of DOC (Van Ende et al., 2017). The findings of the present study indicated that the first research hypothesis, stating that different methods of measuring DOC do not produce different results, was rejected, because there were differences in the DOC of RBRMs measured by manual scrapping and with organic solvent methods for at least one of the sample regions or curing light times evaluated,

For both conventional RBRMs, the DOC recommended by the manufacturers exceeded the 2 mm, regardless the measurement method. The scrapping method has presented significant differences, as reported by other studies about the tendency to under- or overestimate the DOC measurement (Flury et al., 2012). ISO 4049 has failed to detect the poorly polymerized area of transition, i.e., between the glassy and rubbery states, as can be observed clearly in the DOC measurements with the organic solvents (Leprince et al., 2012). Still, the DOC of RBRMs generally increased when the light exposure time was increased. Likewise, the second hypothesis was also rejected.

FU showed a DOC of 2.7 mm following the ISO 4049 and using the curing time recommended by manufacturer (T1). Extending 100% the curing time (T2), the DOC was 3.0 mm. For the dissolution technique using solvents at T1, the DOC was at least 2.2 mm at the side with CHL and THF, but it differed from ISO 4049 method. At the middle, the DOC value was at least 3.2 mm with CHL that did not differ from the scrapping method. For T2, ISO 4049 only differed at side region with ETH, but at the middle region of the sample, the measurements differed regardless the organic solvent used.

FU is RBRM recent launched in the market that contains AUDMA, UDMA, DDDMA in combination with nanofiller loading about 58.4% by volume in attempt to achieve good mechanical and optical properties. The DOC values obtained were within the expected given the manufacturer's indication for increments of 2 mm of thickness.

BE presented a DOC of 2.7 mm according to the ISO 4049 method at T1 and when the exposure time was double of recommended by manufacturer (T2), DOC was 3.1 mm. For the solvent-dissolution method at T1, the DOC was at least 1.9 mm at the side region of the sample with THF, but DOC differed from ISO 4049 regardless the solvent used. At the middle, the lowest DOC value was obtained with ACE and it was the only measurement that did not differ from ISO 4049. For T2, all measurements differed from the scraping method, except for side region with CHL.

BE contains the alumino-fluoro-borosilicate glass, called surface pre-reacted glass ionomer (S-PRG) filler, which is a type of filler that release and recharge ions and is able to inhibit recurrent caries as it increases remineralization, according to its manufacturer. It has been shown that these particles are generally larger than those of others composites (Tsujimoto et al., 2017, Ruivo et al., 2019), but they did not seem to have influenced the polymerization of BE, since it is a conventional resin indicated for increments of up to 2 mm (Tiba et al., 2013).

It is important to evidence that at both T1 and T2, the DOC values obtained with ISO 4049 for both conventional RBRMs had the same value. Although the curing times recommended by the two manufacturers are the same (10 s), the composition of the resins is different, which may reinforce the suggestion that ISO 4049 tends to yield low-accuracy results (Flury et al., 2012) and depends on the force applied by the operator to remove the poorly or uncured composite (Leprince et al., 2012).

FO showed a DOC of 4.4 mm following the ISO 4049 method and using the curing time of 20 s recommended by manufacturer (T1). Extending 100% the curing time (T2), the DOC was 4.9 mm that differed from the manufacturer's recommendation (T1). For the technique using organic solvents at T1, the DOC was at least 3.9 mm at the side with ACE and CHL that did not differ from ISO 4049 method. At the middle, the DOC value was at least 5.6 mm with CHL, but it differed from manual scrapping. For T2, ISO 4049 did not differ when the measurements were performed at the side of the samples. Conversely, it differed regardless the solvent at the middle region of the sample at T2.

FO contains aromatic monomers and filler loading combining technology of a non-agglomerated/non-aggregated silica filler, which allows the refractive index of resin matrix to be close to filler. Nanofilled RBRMs have high translucency because the particles are smaller than the wavelength of light and cause low scattering of photons (Alrahlah et al., 2014). Thus, the light might be successfully transmitted through the material (Pires et al., 1993; Barghi et al., 1994; Miletic et al., 2016; Ferracane et al., 2017) and reach the DOC claimed by the manufacturer. Also, the light-curing unit from the same manufacturer optimized for the light absorption by this *bulk-fill* resin that improves the DOC.

BB presented a DOC of 4.3 mm measured by ISO 4049 standard method at T1 and 4.8 mm when was performed the double of the exposure recommended time. These values were significant different between them. For the solvent-dissolution method at T1, higher DOC values ranged from 2.9 to 3.4 mm with

ACE, ETH and THF at the side region of the samples, but all the solvents differed from ISO 4049. In the middle, the DOC was at least 4.9 mm with CHL and THF, that did not differ from ISO 4049. For T2, this scrapping method did not differ with ACE and THF at the side region and with CHL at the middle region of the sample.

As well as BE, BB composite contains the S-PRG fillers, which is a type of filler particles in Giomers that is generally larger than other composites (Tsujimoto et al., 2017). Despite the same type of particles, a greater amount is present in the composition of the *bulk-fill* resin (74.5%) than the conventional resin (69%). The increase in fillers content results in light scattering at the filler-matrix interface, which might impair the light penetration. In addition to filler quantity, filler type, size and shape can also influence the efficiency of light scattering (Yu et al., 2017).

ImageJ allowed the visual inspection of the samples and proved that the solvent-dissolution technique is sensitive for detecting the effects of organic solvents and on the determination of DOC (Romano et al., 2020). The ImageJ evaluation and microscopy images showed differences in DOC between the side and middle of the sample’s regions. The bottom of the samples was attacked by the solvents and appears whitish (Figure 1). This region has been described as a “frosty” area (Price et al., 2016) and corresponds to the region that was not fully polymerized and serves for determination of the DOC of the composite.

Although Elipar Deepcure presents a homogeneous light beam profile, higher levels of irradiance are emitted in the central area (Shimokawa et al., 2018) where this energy concentration produced greater polymerization in depth at the middle region of the samples. In this study, a silicone mold was used, different from the one recommended by ISO, which absorbs light instead of reflecting it.

The images showed shallower DOC at the side region of the samples that may be clinically related to the restoration proximal boxes in posterior teeth and where most restoration failures occur (Price et al., 2016). Due to the absorption, refraction and scattering of light, a longer exposure time may be required to achieve adequate polymerization over the entire length of the deepest regions of restorations. An increase in exposure duration can extend the DOC of RBRMs and might be an alternative to compensate the light loss through restorations

(Mendonça et al., 2021), mainly when using light-curing units that emit low energy.

The microhardness determination can also provide useful information on the degree of conversion of resin-based materials as an indirect method (Yan et al., 2010; Price et al., 2014). There is evidence indicating that the monomeric conversion rate may influence various mechanical (Fronza et al., 2015; Yan et al., 2010; Miletic et al., 2016) and biological properties (Alrahlah et al., 2014) of composite restorative materials. When polymerization is reduced at greater depths, the material cannot reach enough strength and not bond to tooth structures (Rueggeberg et al., 1994). In general, for all RBRMs tested, KHN decreased as depth increased and the same was reported by other studies, because RBRMs receive less light photons at the bottom of restoration (Fronza et al., 2015; AlQahtani et al., 2015; Soto-Montero et al, 2020). When the light-curing time recommended by the manufacturers was performed, KHN reduction started to appear from 3 mm for conventional resins (FU and BE) and 4 mm for *bulk-fill* resins (FO and BB), Thus, these values reached with the LED curing-unit used in this study the manufacturers' specifications. The 100% increase in light-curing time was enough for KHN values not show a significant difference up to 4 mm for conventional RBRMs and normally up to 5 mm for *bulk-fill* ones.

The third hypothesis stating that the type of organic solvent does not influence the resin dissolution and consequently the DOC was rejected. Analysis of area affected by the organic solvents showed that ETH was the solvent that less affected the RBRMs. The capacity of a solvent to dissolve a polymer can be estimated by means of the Hildebrand solubility parameter. A solvent is suitable for a polymer if both have similar values of the solubility parameter (Kerby et al., 2009; Dimian et al., 2014). The solubility parameters of the monomers most commonly used in RBRMs range from 17 a 22 MPa^{½} (Kerby et al, 2009; Pacheco et al, 2015). As reported at Table 2, ACE, CHL and THF present similar solubility parameters at this range, while ETH presents almost 5 MPa^{½} of difference, which justify its low effect compared to other solvents. Therefore, ETH has limited indication for dissolution DOC technique.

The most common dimethacrylate resin monomers used in current RBRMs are Bis-GMA, TEGDMA and UDMA (Kerby et al., 2009), which are in the composition of the materials used in this study. BE and BB were the most affected resins by solvents compared to FU and FO. Bis-GMA and TEGDMA monomer are present in BE and BB, thus the polymers made with these monomers tend to be somewhat hydrophilic (Shobha et al., 1997). Polymers made with the UDMA monomer like for FU and FO exhibit similar or slightly less water sorption than those prepared from Bis-GMA and TEGDMA (Venz and Dickens, 1991). Also, the inorganic composition of BE and BB (S-PRG filler particles) may cause chemical degradation of the material by the absorption of fluids that can debond the matrix from particles, releasing the residual monomer and oligomers. The releasing of ions from the inorganic part of these restorative materials might increase the solvent diffusion capacity. At the same time (Scougall et al., 2007; Gonulol et al., 2014)

The dissolution of an RBRM is reflected by the amount of leachable unreacted monomers (da Silva et al, 2008). It can be assumed that solubility is increased with a decrease in conversion, corresponding to a reduction in microhardness, as percentages less C = C aliphatic bonds mean greater amounts of unreacted monomers available for leaching (Berger et al., 2009). The microhardness mapping displayed in Figure 6 illustrates that there was variability in hardness across the samples for all RBRMs. The KHN top-to-bottom ratio reduces as depth increases, as reported by other studies (Bouschlicher et al., 2004; AlQahtani et al., 2015; Yap et al., 2016; Soto-Montero et a.l, 2020; Mendonça et al., 2021) and may more pronounced in resins with Giomers. FU had a 50% KHN ratio close to 4 mm depth, while at this same depth, BE samples had already shown 33% of this ratio in the lateral regions of the samples. A ratio of 50% of the KHN top-to-bottom was observed from 7 mm for FO, while BB presents this rate from 4 mm of depth.

ISO 4049 can be used (Rueggeberg et al., 2009), but its results must be carefully interpreted, and their limitations considered. On the other hand, chemical removal of uncured or inadequately polymerized resins using organic solvents can provide a reliable technique that allows uncured resin removal, the visualization the poorly cured polymer and the well-cured one (Leprince et al., 2012; Price et al., 2016; Romano et al., 2020). However, some of these organic solvents are products of restricted or controlled use in some countries, therefore not easy to acquire.

The fabrication of the samples for this study followed ideal laboratory conditions, where the parameters of accessibility, alignment and distance of the light-curing unit to the composite were well controlled. Therefore, clinicians must consider the limiting intraoral factors they face with the need to provide additional radiant energy to achieve the desired polymerization by applying additional light exposure than that required by the material (Price et al., 2014).

Conclusion

Based on the limitations of this in vitro experiment, the following conclusions can be addressed:

1. ISO 4049 method resulted in different depth of cure measurements compared to other methods.

2. Solvent-dissolution technique revealed to be sensitive to detect poor and well-cured composite areas.

3. The KHN decreased with increasing depth from 3 to 4 mm for conventional composites and 4 to 5 mm for *bulk-fill* materials, but depended on the curing time.

4. The extending of curing time can increase the depth of cure of composites.

5. The area of composite samples affected by the solvent was dependent on the type of material and organic solvent used.

Acknowledgements

This study was financed by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior – Brasil (CAPES) – Finance Code 001 and Conselho Nacional de Desenvolvimento Científico e Tecnológico – Brasil (CNPq) #130481/2020-3.

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[1] De acordo com as normas da UNICAMP/FOP, baseadas na padronização do International Committee of Medical Journal Editors - Vancouver Group. Abreviatura dos periódicos em conformidade com o PubMed

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ANEXOS

ANEXO 1 – Comprovação da submissão do artigo

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