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Micro-computed tomography film thickness evaluation in different indirect restoration luting techniques

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Coimbra, junho de 2022

Micro-computed tomography film thickness evaluation in different indirect restoration luting techniques

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Table of contents

| Resumo | |
|-----------------------|---|
| Abstract | 7 |
| Introduction | |
| Materials and methods | |
| Results | |
| Discussion | |
| Conclusions | |
| Acknowledgments | |
| References | |
| | |

Resumo

Objetivo: Avaliar a espessura de película resultante da cimentação de restaurações indiretas em preparações laboratoriais padronizadas, utilizando um cimento resinoso e duas resinas compostas, termo-modificados e/ou sujeitos a vibração ultrassónica, utilizando microtomografia computadorizada (micro-CT).

Materiais e métodos: Oito (8) blocos de resina composta fresados com morfologia de um molar preparado para onlay e oito (8) restaurações cerâmicas foram aleatoriamente emparelhados e distribuídos por oito (8) grupos experimentais (n = 1), de acordo com o agente cimentante utilizado (Variolink Esthetic LC, IPS Empress Direct ou Estelite Omega) e com a técnica de cimentação (sem pré-aquecimento/pré-aquecimento a 68°C e/ou vibração ultrassónica). Posteriormente, a espessura de película foi determinada recorrendo a secções obtidas por micro-CT, através da média de cinco pontos de avaliação pré-definidos ao longo da interface em cada secção.

Resultados: As espessuras de película de cimento mais reduzidas foram encontradas nos grupos VLC (22.78 μ m) e VLC-UV (15.56 μ m), ao passo que as amostras do grupo IPS-PH exibiram a interface mais espessa (348.48 μ m). Os grupos IPS-PH-UV e IPS-UV apresentaram valores médios de espessura de película de 101.03 μ m e 105.78 μ m, respetivamente. Quanto aos grupos Estelite-PH, Estelite-PH-UV e Estelite-UV os valores médios foram de 293.14 μ m, 221.49 μ m e 178.61 μ m, correspondentemente. Adicionalmente, constatou-se uma maior uniformidade dos valores ao longo da interface nos grupos de cimento resinoso comparativamente com os grupos de resinas compostas.

Conclusão: O cimento resinoso Variolink Esthetic LC, com ou sem vibração ultrassónica, apresentou a menor espessura de película. O uso de vibração ultrassónica representa uma técnica eficaz na redução da espessura de película em todos os materiais testados. Mais estudos são necessários para avaliar o impacto no deslizamento da restauração e da formação de bolhas de ar, aquando do uso de resinas compostas pré-aquecidas.

Palavras-chave: Cimentação adesiva, cimentos resinosos, espessura de película, resinas compostas, termo-modificação, vibração ultrassónica.

Abstract

Aim: To evaluate film thickness obtained by luting indirect restorations to standardized laboratory preparations, using a resin cement and two composite resins, thermomodified and/or ultrasonically vibrated, through micro-computed tomography (micro-CT) analysis.

Materials and methods: Eight (8) composite resin blocks milled according to molarshaped onlay preparation and eight (8) matching ceramic restorations were randomly paired and assigned to eight experimental groups (n = 1), according to the used luting agent (Variolink Esthetic LC, IPS Empress Direct or Estelite Omega) and cementation technique (no preheating/preheating at 68°C and/or ultrasonic vibration). Subsequently, the film thickness obtained was observed and measured based on micro-CT sections by calculating the mean value of five pre-defined interface evaluation points in each.

Results: The lowest film thickness was found in both VLC (22.78 μ m) and VLC-UV (15.56 μ m) groups, whereas IPS-PH group presented the highest (348.48 μ m). IPS-PH-UV and IPS-UV groups had mean film thickness values of 101.03 μ m and 105.78 μ m, respectively. Estelite-PH, Estelite-PH-UV and Estelite-UV exhibited a mean value of 293.14 μ m, 221.49 μ m and 178.61 μ m, correspondingly. Moreover, resin cement groups showed greater values' consistency along the interface when compared with composite resin groups.

Conclusions: The lowest film thickness was obtained with Variolink Esthetic LC resin cement, with or without ultrasonic vibration. The use of ultrasonic vibration proved to be effective in reducing film thickness for all tested materials. Further research is needed to evaluate the impact of restoration sliding and air bubble formation within the cement layer when using preheated composite resins.

Keywords: Adhesive cementation, composite resins, film thickness, resin cements, thermo-modification, ultrasonic vibration.

1. Introduction

Dental restorations can be classified as direct or indirect. Regarding the latter, a restoration is fabricated outside the oral cavity using an impression of the prepared tooth and then stabilized into place.^{1,2} These can be made of ceramic or composite resin. As for ceramics, they are considered superior to composite resins, mainly for the color, shape, and texture stability over time, as well as higher fracture and wear resistance.³

Indirect restorations must be retained by a luting agent that also acts as a barrier against microleakage by sealing the tooth-restoration interface.¹ This type of restoration provides greater predictability regarding the anatomy of the contact point, greater control of polymerization shrinkage, better physical and mechanical properties, ideal occlusal morphology, and wear compatibility with opposing natural dentition.² Ensuring indirect restorations' retention, marginal seal, and durability depends heavily on effective luting.⁴ The cementation techniques can be classified as classic (non-adhesive) or adhesive. Adhesive cementation benefits not only from mechanical retention but also chemical, accomplished by using an adhesive system and a resin luting agent. This aspect changed the preparation principles for indirect restorations, allowing more remarkable structural preservation and reinforcement of ceramic restorations and the remaining dental structure, with the main responsible for the long-term success being the luting agent.^{5,6}

The outcome of ceramic restorations depends on obtaining a strong, durable bond between the resin cement and dentin/enamel; hence choosing the appropriate luting agent is crucial.^{5,7} This choice has become increasingly complex as several new cements are designed to perform better, be easier to handle and more predictable than materials used in the past. The main characteristics of an ideal luting agent include biocompatibility, radiopacity, low solubility, micromechanical union between the dental remnant and the ceramic, high shear bond and tensile bond strengths, resistance to marginal microleakage, easy handling, color stability, and finally, the ability to provide a thin film thickness.⁷ The ideal film thickness should range between 5 and 25 µm, never surpassing 50 µm, which allows a higher bond strength and a dwindled microleakage. Thicker films may lead to marginal misfit and produce more significant shrinkage stresses during light curing, leading to premature debonding and intrinsic tensile stress that can damage the ceramic.^{5,8-15}

Resin cements are composite resins composed of a resin matrix and filler particles. They differ from restorative composites, primarily in their lower filler content and

viscosity.^{5,7,15,16} According to the polymerization mechanism, cements may be classified into chemically, light or dual-cure.^{1,4,7,10,16} Many advantages can be assigned to resin cements, such as high compressive and tensile strengths, good aesthetic qualities, low solubility, resistance to wear and high micromechanical bonding to both tooth and ceramics.^{1,4,5,7,10} Their viscosity is related to filler materials' lower weight percentage (wt%), which gives the luting agent a higher flowability.^{5,7,17}

Composite resins have been pointed out as an alternative luting agent in adhesive cementation of indirect restorations due to their lower cost, extensive range of shades available and superior mechanical properties.^{15,18-20} The problem resides in their high viscosity, leading to a very thick film of material and consequent marginal misfit and increased microleakage. Pre-heating techniques have been described to reduce the composite viscosity and increase flowability, aiming to reach minimal film thickness values (below 50 μ m) compatible with long-term rehabilitation success.^{15,21-40} Another alternative to improve the flowability of composite resins and reduce film thickness via a thixotropic effect is using ultrasound energy alone or combined with preheating.^{28,41-43}

Technological equipment capable of yielding high-resolution images may become essential to adequately evaluate the film thickness achieved with different materials. Micro-computed tomography (micro-CT), a system consisting of an X-ray source and a detector between which the sample is placed, is highly recommended for that purpose. The X-ray projections acquired at each rotation angle are used to rebuild tomographic images, visualized as 2D slices or 3D volumes of the specimen.^{44,45}

According to our research, limited literature compares the film thickness achieved with heated composite resins and resin cements.⁴⁶⁻⁴⁸ This study aims to evaluate film thickness resulting from luting indirect restorations to standardized laboratory preparations that mimic an actual clinical situation, with resin cement and two different composite resins with thermo-modification and/or ultrasonic vibration, using micro-CT as a tool.

The research hypotheses of the current study are the following:

- 1. There is no difference in the obtained film thickness between the tested materials and techniques.
- 2. Using ultrasound energy, with or without preheating, decreases the film thickness of the tested materials.

2. Materials and methods

2.1 Study design:

Considering the various categories, formulas, and manufacturers, one conventional resin cement and two restorative resin composites were chosen. Table 1 summarizes their characteristics.

The room temperature was set to 22°C, and the clinically preferred temperature for luting with preheated composite resin was 68°C. After restoration seating and removal of significant excesses, the effect of ultrasound energy application on film thickness was investigated using an ultrasonic tip. Following cementation, the film thickness obtained was observed and measured using micro-computed tomography (micro-CT) imaging.

| | | | Compos | ition | Particles | | |
|--|---|----------------------------------|-----------------------------|-------------------------|-----------|------------------------------|--|
| Resin- based luting agent | Manufacturer | Туре | Resin monomers | Filler wt% (vol%) | Shape | Mean size (range) | |
| Variolink Esthetic LC | lvoclar Vivadent, Schaan, Liechtenstein | Light-cured resin cement | UDMA, DDMA | (38) | Irregular | 0.1 μm (0.04 μm - 0.2 μm) | |
| IPS Empress Direct | lvoclar Vivadent, Schaan, Liechtenstein | Nanohybrid composite resin | Bis-GMA, UDMA, TCDDMA | 75-79 (52-59) | Irregular | 550 nm (40 nm - 3 μm) | |
| Estelite Omega | Tokuyama, Tokyo, Japan | Supranano composite resin | Bis-GMA, TEGDMA | 82 (71) | Spherical | 200 nm (100 nm - 300 nm) | |
| UDMA, urethane dimethacrylate; DDMA, 1,10-decandiol dimethacrylate; Bis-GMA, bisphenol-A glycidyl dimethacrylate; TCDDMA, tricyclodocane dimethanol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate. | | | | | | | |

Table 1. Characteristics of the luting agents used in the luting protocol.

2.2 Sample standardization and preparation:

A human lower first molar was extracted for periodontal reasons and prepared by a calibrated operator for a standard onlay restoration following the biomimetic technique, with a composite resin core and a preparation to accommodate an enamel-equivalent ceramic restoration. The preparation was done to replicate one of the most common destructions, affecting two cusps to a greater extent.

After preserving the prepared natural tooth in water, the preparation was digitalized using a DS10 contact scanner (reused to design a restoration). Afterward, eight 6x10mm composite resin blocks (Tetric CAD, MT A2/C14, Ivoclar Vivadent, Schaan, Liechtenstein) were milled using the STL from the original prepared tooth. The corresponding ceramic restorations were fabricated by injection molding, resorting to lithium disilicate ceramic tablets (IPS e.max, Ivoclar Vivadent, Schaan, Liechtenstein).

2.3 Experimental protocol:

Using an online randomizer software (<u>https://www.randomizer.org/</u>) the tooth models and corresponding ceramic restorations were randomly paired and assigned to eight experimental groups (n=1) according to luting agent (Variolink Esthetic LC, IPS Empress Direct or Estelite Omega) and cementation technique (no preheating/preheating at 68°C and/or ultrasonic vibration), summarized in Table 2. Each restoration was cemented following the protocol corresponding to each group:

VLC group: samples cemented with Variolink Esthetic LC resin cement at room temperature (22°C).

VLC-UV group: samples cemented with Variolink Esthetic LC resin cement at room temperature (22°C), ultrasonically vibrated.

IPS-PH group: samples cemented with preheated composite resin IPS Empress Direct at 68°C.

IPS-PH-UV group: samples cemented with preheated composite resin IPS Empress Direct at 68°C with the addition of ultrasonic vibration.

IPS-UV group: samples cemented with composite resin IPS Empress Direct at room temperature (22°C), ultrasonically vibrated.

Estelite-PH group: samples cemented with preheated composite resin Estelite Omega at 68°C.

Estelite-PH-UV group: samples cemented with preheated composite resin Estelite Omega at 68°C with the addition of ultrasonic vibration.

Estelite-UV group: samples cemented with composite resin Estelite Omega at room temperature (22°C), ultrasonically vibrated.

| Experimental group n=1 | Luting agent | Temperature | Ultrasonic vibration | |
|---------------------------|------------------------------------|-------------|-------------------------|--|
| VLC | Variolink Esthetic LC ¹ | 22°C | No | |
| VLC-UV | Variolink Esthetic LC ¹ | 22°C | Yes | |
| IPS-PH | IPS Empress Direct ² | 68°C | No | |
| IPS-PH-UV | IPS Empress Direct ² | 68°C | Yes | |
| IPS-UV | IPS Empress Direct ² | 22°C | Yes | |
| Estelite-PH | Estelite Omega ² | 68°C | No | |
| Estelite-PH-UV | Estelite Omega ² | 68°C | Yes | |
| Estelite-UV | Estelite Omega ² | 22°C | Yes | |

 Table 2. Experimental groups.

¹Resin cement; ²Composite resin. Abbreviations: PH, preheated; UV, ultrasonically vibrated.

2.4 Luting protocol:

The resin tooth models and respective ceramic restorations were identified and numbered. During the luting procedure, the simulated tooth preparations were included and stabilized in a simulation model of the oral cavity. On each group, the luting protocol was carried out as follows.

Firstly, to achieve a homogeneously rough surface and allow the penetration of the bonding agent, as well as simulate intra-oral conditions, all resin tooth models were sandblasted with 30 µm aluminum oxide particles (1 cm, 45°, 2 bar). The surfaces were cleaned with 37.5% phosphoric acid (Gel Etchant; Kerr, Orange, California, USA) and rinsed with an air-water combined stream for 30 seconds, followed by airdrying.

Regarding the preparation of the restorations, the internal surface of the ceramic was etched for 20 seconds with 5% hydrofluoric acid and then thoroughly rinsed with water for one minute. The resultant surface was cleaned by immersion in an ultrasonic cleaner (Transistor/Ultrasonic T.14; L&R, Rengsdorf, Germany) with 90% alcohol for 5 minutes. After using a vigorous air stream to dry the restoration, a universal ceramic primer

(Monobond Plus, Ivoclar Vivadent, Schaan, Liechtenstein) was applied and allowed to dry.

The adhesive system OptiBond FL (Kerr, CA, Orange USA) was applied to the model tooth preparation surfaces and in the ceramic restorations with the help of a microbrush. For approximately 10 s, both the restoration and preparation were air-dried to obtain a shiny and immobile adhesive layer, without light-curing.

All restorations were seated with a controlled force of 23 N, which was measured using a patented mechanical device with a flat soft 4 mm diameter surface (publication number WO/2020/167153), according to the technique described by Falacho *et al.*⁴⁸

2.4.1 Variolink Group (resin cement)

Following the common procedures to all research groups, the luting agent Variolink Esthetic LC was placed over the unpolymerized bonding layer in the internal surface of the ceramic restoration, which was further placed over the tooth preparation with bonding agent. A controlled force of 23 N was exerted over the ensemble for 60 s using the previously mentioned mechanical device. After excesses removal with OptraSculpt (Ivoclar Vivadent, Schaan, Liechtenstein) and a brush, the interface was light-cured fourteen times for 10 s each in two passes, resorting to a polywave LED curing light source with a measured intensity of 1200 mW/cm² (Bluephase Style; Ivoclar Vivadent, Schaan, Liechtenstein). Finally, a glycerin-based aqueous gel (Liquid Strip; Ivoclar Vivadent, Schaan, Liechtenstein) was placed over the exposed limits of the adhesive interface and light-cured seven times for 10 s each in order to remove the oxygen-inhibited layer.

2.4.2 Variolink-UV group (ultrasonically vibrated resin cement)

The Variolink-UV group followed the same cementation protocol as the Variolink group, with the exception that while the restorations were set, ultrasonic vibration was applied. Smooth movements from the center to the periphery of the samples were conducted using a CM4 ultrasonic tip (CVDentus, São Paulo, Brazil), placed in an ultra-sound handpiece (CVDentus, São Paulo, Brazil) that worked at 30% power without irrigation.

2.4.3 IPS-PH and Estelite-PH groups (preheated composite resin)

The cementation protocol was followed as in the Variolink group, with the exception that IPS Empress Direct and Estelite Omega composite resins were used as luting agents in groups IPS-PH and Estelite-PH, respectively. Both were preheated at 68°C for 30 minutes prior to the luting procedure using a heating unit (Calset; AdDent,

Danbury, Connecticut, United States of America). The ceramic restorations were left inside the heater after silanization to reduce heat loss during the cementation process. In order to guarantee maximal resin overflow, excess removal with an OptraSculpt (Ivoclar Vivadent, Schaan, Liechtenstein) was conducted four times, every 15 s, throughout the controlled seating of the restorations.

2.4.4 Estelite-PH-UV and IPS-PH-UV groups (preheated and ultrasonically vibrated composite resin)

The protocol described for the IPS-PH and Estelite-PH groups was followed in the IPS-PH-UV and Estelite-PH-UV groups, but with the addition of ultrasonic vibration during the seating of the restorations.

2.4.5 IPS-UV and Estelite-UV groups (ultrasonically vibrated composite resin)

The identical cementation protocols were adopted in the IPS-UV and Estelite-UV groups as in the other groups, with the exception that the luting agent was used at room temperature (22°C) without being preheated. The Estelite-PH-UV and IPS-PH-UV groups were both submitted to ultrasonic vibration during the seating process.

2.5 Micro-CT imaging:

In this study, the specimens were scanned using a high-resolution micro-CT device (Bruker Skyscan 1275, Kontich, Belgium) provided by the Center for Mechanical and Automation Technology (TEMA), Department of Mechanical Engineering, University of Aveiro. In order to obtain the best image quality possible, the X-ray tube voltage and current were set at 80 kV and $125 \,\mu$ A, respectively. The distance between the sample/object and the X-ray source was 31.744 mm, while the distance between the X-ray source and the camera was 286.0 mm. Lastly, the resolution (image pixel size) was 9.000786 μ m and the overall scanning time for each tooth sample was around 60 minutes.

2.6 Measurement of the luting agent film thickness:

On each sample, the film thickness was measured in five cuts/sections at five distinct segments, as shown in figure 1. The mean values of the five segments assessed on each sample were used to get the average thickness value of each group.



Figure 1. Schematic representation of the evaluated segments in each specimen.

3. Results

3.1 Micro-CT analysis:

A representative section of each experimental group can be observed in figure 2.



Figure 2. Micro-CT representative sections of each experimental group: (A) VLC group; (B) VLC-UV group; (C) IPS-PH group; (D) IPS-PH-UV group; (E) IPS-UV group; (F) Estelite-PH group; (G) Estelite PH-UV group; (H) Estelite PH-UV group.



Figure 3. Micro-CT representative section of the IPS-PH group: a more distal cut and two air bubbles within the cement layer can be seen.

3.2 Film thickness measurement:

| Table 3. Mean v | alues of the film | thickness in | micrometers | (µm) for each | tested group. |
|-----------------|-------------------|--------------|-------------|---------------|---------------|
|-----------------|-------------------|--------------|-------------|---------------|---------------|

| | VLC | VLC- UV | IPS- PH | IPS- PH- UV | IPS- UV | Estelite- PH | Estelite- PH-UV | Estelite- UV |
|----------------|-------|------------|------------|-------------------|------------|-----------------|--------------------|-----------------|
| Segment 1 | 30.20 | 14.99 | 263.00 | 126.33 | 103.04 | 335.76 | 260.15 | 165.97 |
| Segment 2 | 30.00 | 18.03 | 376.38 | 165.99 | 91.91 | 334.33 | 193.87 | 210.82 |
| Segment 3 | 12.41 | 17.36 | 349.70 | 85.04 | 102.88 | 365.26 | 272.74 | 206.89 |
| Segment 4 | 12.30 | 13.39 | 258.76 | 32.30 | 108.48 | 231.53 | 194.99 | 162.07 |
| Segment 5 | 28.98 | 14.05 | 494.55 | 95.48 | 122.61 | 198.84 | 185.68 | 147.29 |
| Mean values | 22.78 | 15.56 | 348.48 | 101.03 | 105.78 | 293.14 | 221.49 | 178.61 |

4. Discussion

According to the findings of this study, our first research hypothesis is rejected since the groups in which the Variolink Esthetic LC resin cement was used as a luting agent, whether ultrasonically vibrated or not, offered an expressively lower film thickness. Regarding the second hypothesis, our findings supported the premise that ultrasonic energy reduced the film thickness of both composite resins tested, with or without preheating.

Effective cementation is vital for achieving good retention, marginal seal, and longevity of indirect restorations. A restoration's longevity is determined by several factors, one of which is the chosen luting agent. Using an inadequate luting agent or manipulating it incorrectly can have a considerable impact on the durability of an indirect restoration.¹ The luting agent's film thickness is critical, as thicker films are more prone to degradation, leading to marginal misfit, and have also been linked to lower flexural strength of ceramic restorations.^{8,10,15,46} Furthermore, when thicker layers of resin material undergo a polymerization reaction, added volumetric shrinkage is expected, as well as increased polymerization shrinkage stress on both the ceramic and the tooth surfaces, which will intensify the possibility of early debonding, gap creation and overall susceptibility to failure.^{11,15,46} May et al.¹² found that the occlusal cement thickness had a substantial impact on the failure loads of feldspathic ceramic crowns, whether they were bonded or not, and that when the occlusal cement layer thickens (from 50 to 500 nm), the polymerization shrinkage has a significant impact on the tensile stresses in bonded restorations. Moreover, increased polymerization stress is expected when preheating the luting agent as molecular mobility and collision frequency of reactive species increase with temperature.^{21,31,32,46} Linking these two aspects, it becomes clear that using preheated luting materials in higher volumes leads to greater polymerization stress and thus this combination of factors should be avoided.

According to ISO standard 4049:2019, clinicians should maintain a minimal ideal film thickness between 5 and 25 µm independently of the luting material, and on no occasion shall it exceed 50 µm.^{5,9,28} Reduced film thickness allows to minimize the effect of water sorption and its consequences to the properties of the cement and respective support for the ceramic restoration.⁸ According to the results of this research, only Variolink Esthetic LC resin cement (ultrasonically vibrated or not) provided values that met the optimum range of film thickness, with all the other groups having film thickness mean values exceeding 50 µm, rendering them incompatible with the acceptable interface thickness limits.⁹

The luting agent viscosity is directly related with film thickness. This property determines the degree of molecular mobility of a resin-based material³³ and is influenced by the material's structure, which includes the resin matrix composition as well as the filler content.^{20,29,36} Improved marginal adaptation, lower microleakage and greater contact with the prepared tooth surfaces are some of the advantages shown by low viscosity materials.^{22,25,27,29,35} Resin cements differ from restorative composites essentially by their reduced filler content and, as a result, their lower viscosity^{5,7} which allows for an adequate film thickness in the luting procedure, but also by a higher content of photoactivators which contribute to an improved light-curing response. Composite resins, on the other hand, contain more inorganic particles and, thus, are less flowable.

Alternative strategies, as preheating, have been developed to lower the viscosity of composites^{15,21,25,26,29,33,37,38,47}. conventional consequently decreasing film thickness.^{15,20,25,36} According to some studies, this approach may lead to an easier restoration placement and a higher monomer conversion due to higher free radical and monomer mobility, as well as the collision frequency of unreacted groups, resulting in increased surface hardness and wear resistance.16,21,23-25,30,33,36 However, one of the most challenging aspects of dealing with preheated composites in the daily clinical routine is that once preheating is stopped and the luting agent is transported and applied to the tooth structure, heat dissipates quickly, making it difficult to maintain the flowability achieved upon heating.^{20,22,39,46} According to Daronch et al., 50% of the heat loss occurs within two minutes after the composite is removed from the heater and almost 90% after 5 minutes.^{20,29,34,35,38} Therefore, a short interval between preheating the composite and applying it on the restoration or tooth surface is crucial for a film thickness reduction. In this study, to minimize heat loss during the luting process, the ceramic restorations were left inside the heater after silanization.

When comparing resin cements and preheated composite resins regarding the obtained film thickness, Falacho *et al.* evaluated and compared the film achieved with a resin cement (Variolink Esthetic LC) and two composite resins (IPS Empress Direct and Estelite Omega) preheated and/or ultrasonic vibrated as luting agents, concluding that Variolink Esthetic LC and IPS Empress Direct preheated and ultrasonically vibrated achieved the lowest film thickness, both below 50 µm.⁴⁸ In another study, Coelho *et al.* investigated the performance of bonded feldspathic ceramic disks that simulated veneers using three thermo-modified restorative composite resins (Filtek Z100–microhybrid, Empress Direct–nanohybrid, Estelite Omega) and one photoactivated resin cement (RelyX Veneer). The resin cement produced a thinner film than all the composites tested.²⁰ Additionally, the authors concluded that the most viscous composite resin at room temperature (IPS Empress Direct) was also the most fluid material at 69°C,

demonstrating that different composite resins react differently to preheating, potentially affecting viscosity, flowability, and film thickness.²⁰ This study's results show that groups where the resin cement was used as a luting agent had the lowest film mean values, which is consistent with what has previously been found. Even though thermomodification is intended to lower viscosity and promote flowability of the restorative composite resin materials, substantially thicker interfaces are seen in our study and in others when compared to resin cements. Materials' viscosity tends to rise as the filler load increases, which explains why composite resins have a higher viscosity than resin cements, even when preheated, resulting in a thicker luting interface.^{17,20}

Another strategy for reducing film thickness via a thixotropic effect is using ultrasound energy to increase the flowability and wetting properties of the composite resin materials when applied over the ceramic restoration.^{28,41,43} Cantoro *et al.* resorted to microtensile bond strength tests and microscopy observations of the luting interface to analyze the bonding capacity of self-adhesive resin cements.⁴² Through SEM analysis, a reduction in density and size of porosities was reported within the luting agent when the ultrasonic insertion technique was used, as well as a thinner cement layer and higher microtensile bond strength.⁴² According to some studies, ultrasonic vibration also promotes a faster restoration seating and lower air bubbles formation within the cement layer.^{41,42}

Based on the findings of this investigation, the group cemented with Variolink Esthetic LC ultrasonically vibrated (VLC-UV) had the lowest mean film thickness (15.56 µm). In the Estelite Omega composite resin groups, the results show that employing solely ultrasound energy produces a thinner interface than combining both preheating and ultrasonic energy. As for the IPS Empress Direct composite resin groups, the sample cemented only with ultrasonic energy provided a very similar film thickness as the one preheated and ultrasonically vibrated. As a consequence, and according to this study's results, employing ultrasonic energy when luting indirect restorations appears to be recommended when using both tested composite resins, not only to aid but possibly to replace preheating processes. Moreover, compared to the groups where ultrasonic energy was not employed, ultrasonic vibration's introduction decreased film thickness on all the tested materials.

In the present study, the VLC and VLC-UV groups' mean results in each segment assessed on each sample were more similar and uniform due to homogeneity in thickness along the luted surface. This might explain why the VLC and VLC-UV groups' micro-CT images (2A and 2B) show a proper seating of the restoration rather than restoration shifting, as verified in some composite resin groups, specifically in IPS-PH and Estelite-PH (2C and 2F). During the luting procedure, the simulated tooth preparations were included and stabilized in a simulation model of the oral cavity with

the reproduction of contact points and all aspects of positioning an indirect restoration *in situ*, as well as the angle of force exerted. Therefore, the sliding of the restoration observed in the micro-CT images is assumed to also occur clinically. Based on these results, it is possible to infer that when heated composite resins are used as luting agents, restoration sliding often occurs upon application of the seating force, potentially originating a mismatch, resulting in irregular film thickness values throughout the sample. This mismatch may pass unnoticed clinically as the composite tends to fill the over or undercontour in a bevel-like structure that masks these defects and renders an apparent but illusory sense of flawless finishing lines, as shown in the figures 2E, 2F and 2G. It is also essential to notice the voids, more specifically air bubbles, imprisoned within the luting material layer in heated composite groups (figure 3). Such discontinuities could influence the resistance and, thus, the clinical longevity of luted restorations.⁴²

As aforementioned, different formulations of composite resins may react differently to preheating.^{20,28,32,36} In this study, the composite resin IPS Empress Direct at 68°C had the highest film thickness (348.48 µm), which is connected to the material's flowability. As previously stated, IPS Empress Direct comprises irregularly shaped particles (sizes ranging from 40 nm to 3 µm), accounting for the greater viscosity. The application of ultrasonic vibration resulted in a drastic reduction of the film thickness, with the IPS-PH-UV group having a mean film thickness value of 101.03 µm. As for the Estelite Omega groups where ultrasonic vibration was used (Estelite-PH-UV and Estelite-UV), a decrease in film thickness mean values was also verified when compared to the Estelite Omega group that was only preheated (Estelite-PH). This decrease, however, was not as pronounced as in the IPS Empress Direct groups, indicating that ultrasonic vibration does not affect all tested resins in the same manner. This discrepant behavior could be explained by the presence of circular and homogeneous shape particles (size ranges between 100 nm and 300 nm) found in the Estelite Omega composition, as well as the inorganic load accounting for 71% of the total volume, making the structural organization denser than the alternative composite resin tested. As a result of the large number of regular spherical particles included in Estelite Omega, less inter-particle free space filled by a scarce resinous matrix is expected. This potentiates an apparently lower viscosity at room temperature, however, particle movement and disorganization are constricted when exposed to preheating or ultrasonic energy, resulting in a far lesser viscosity variation relating to the initial flowability, when compared to composite resins with more irregular particles, such as IPS Empress Direct.

As for the seating force applied during the cementation procedure, according to a prior study, it is a crucial variable that has a significant impact on the luting agent's film thickness.¹⁴ To our knowledge, only one study that compares the film thickness obtained

with preheated composite resins and resin cements, specifies how the seating force was measured.⁴⁸ Therefore, the authors of this study found immense relevance in using the patented force application instrument that may provide a controlled pressure of 23N and a force application setup that closely resembles the clinical scenario, allowing for a rigorous standardization and clinical relevance of the force applied during sample cementation.

Micro-computed tomography (micro-CT) was chosen to evaluate the specimens' film thickness in this research. This technique produces radiographs, also known as projection images, that are taken incrementally throughout a total rotation of 180 or 360 degrees. The obtained radiographs are then processed using a computer software, resulting in a series of reconstructed images (2D slices) that allow the internal structure of the item to be observed and a three-dimensional (3D) volume to be recreated.^{40,45}

Micro-CT has the advantage of being a non-destructive technique that keeps the sample intact. Additionally, it grants the possibility to evaluate sections with a perfectly perpendicular axis to the cementation line, as the virtual slices may easily be previewed, adapted, rotated, and positioned using the software until no distortion is present. This option eliminates one of the most common drawbacks in using SEM, the subjective positioning when cutting the samples which produces distortions in the cementation line measurements, as seen in most available literature. However, one of the difficulties found in this research was distinguishing between the different materials within the specimen as they held similar radiopacities.

Finally, resin cements appear to preserve their position as the gold standard, offering more benefits than preheated composite resins. The use of Variolink Esthetic LC in the cementation protocol already allows a shallow film thickness, with additional strategies to reduce film thickness proving unnecessary. These cements can be light-cured, self/auto-cured, or dual-cured and come in various shades, allowing for aesthetic shade matching.^{7,10} Although technique sensitivity and difficulty with excess removal are some of the challenges when working with these materials.⁴

Limitations of the study

This study aimed to use micro-computed tomography (micro-CT) imaging to evaluate and compare the thickness of the restoration-luting agent-tooth interface using various resin-based agents and procedures in adhesive cementation, resorting to 3D tooth preparation/restoration simulation models.

The use of only one resin cement in the luting procedure, as well as the small number of samples tested on each group, were both limitations of this study.

Future perspectives for this ongoing research line include testing alternative resin cements and composite resins, as well as comparing different types of tooth preparations.

5. Conclusions

Taking into consideration the limitations of this *in vitro* study, the following conclusions were drawn:

- The lowest film thickness was achieved in the samples cemented with Variolink Esthetic LC resin cement, with or without ultrasonic vibration.
- Using ultrasonic vibration during the cementation procedure proved to be effective in reducing film thickness. Nonetheless, in the composite resin groups (with or without preheating), ultrasonic energy didn't allow reaching thickness values below 50 µm.
- Further research is needed to evaluate the impact of restoration sliding and air bubble formation within the cement layer when using preheated composite resins.

Acknowledgments

No epílogo deste meu percurso académico, que se mostrou uma página inesquecível e irrepetível da minha vida, a tese de mestrado representa o seu culminar.

Um caminho tão longo e árduo não poderia ter sido percorrido sozinha.

Ao meu orientador, Professor Doutor Fernando Guerra, pelos ensinamentos transmitidos e contínua disponibilidade.

Ao meu co-orientador, Prof. Doutor Rui Falacho, pelo incansável e vigilante acompanhamento, disponibilidade e paciência, sem olvidar a força e serenidade que me transmitiu durante todo o meu percurso académico, em particular neste último ano. O modo como fomenta o espírito crítico e autónomo dos seus alunos e o altruísmo na partilha de conhecimentos de elevado rigor científico é deveras admirável. Foi desde o primeiro dia um modelo de profissionalismo, dedicação e resiliência.

Ao Prof. Doutor João Carlos Ramos pela disponibilidade preciosa para a realização deste trabalho.

Ao Dr. Raphaël Gameiro, do Laboratório Raphaël Gameiro Prótese Dentária de Pombal, e à Dra. Isabel Duarte do Departamento de Engenharia Mecânica (TEMA) da Universidade de Aveiro, pelo contributo prestado nos segmentos experimental e laboratorial deste estudo.

À lvoclar Vivadent pela disponibilidade que demonstrou e pela cedência do material utilizado nesta investigação.

A todos os docentes e auxiliares que cruzaram o meu caminho e que deixaram em mim uma parte de si em cada passo.

Aos meus avós, em especial ao meu avô Zé, que não estando mais entre nós fisicamente, permanecem para sempre no meu coração e na minha mente. Onde quer que esteja, sei que está orgulhoso das minhas conquistas.

Aos meus pais, a quem devo tudo o que sou hoje. Obrigada por serem o meu maior exemplo de resiliência e tenacidade, coragem e dedicação. A vocês vos devo o ensinamento de que tudo está ao nosso alcance e nada é impossível se formos benevolentes, humildes e empenhados. Nunca poderei retribuir tudo quanto me deram.

Aos meus irmãos, André, Sandra e Patrícia tenho a agradecer-lhes serem a extensão de tudo aquilo que os nossos pais são e nos ensinaram. São o meu porto-seguro e os meus melhores amigos para toda a vida.

Ao meu namorado, Miguel, pelo amor, compreensão, confiança e apoio inabaláveis. O meu companheiro de viagem. Por, tantas vezes, não permitir que duvidasse de mim mesma e, por tantas outras, me fazer encontrar forças onde não imaginava que existissem.

À Alcinda, Cristiana, Diana, Luís e Mariana, companheiros nesta memorável caminhada académica, a quem devo dirigir um especial agradecimento por me encorajarem, incansavelmente, nos momentos de dificuldade e hesitação e aplaudirem os meus momentos de sucesso.

A todos o meu sincero e profundo Muito Obrigada!

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