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Microscopic inspection of the resin-matrix cement and flowable composite layers after low loading cementation of composite onlays to dentin and enamel:

An in vitro study

Tiago João Carvalho Magalhães

Dissertação conducente ao Grau de Mestre em Medicina Dentária (Ciclo Integrado)

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Trabalho realizado sob a Orientação de "Júlio Souza"e Co-orientador "Orlanda Torres"

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Resumo

Contexto: A camada de cimento resinoso pode variar na interface de restaurações indiretas, em função do protocolo de cimentação e do tipo de cimento resinoso. Uma camada espessa de cimento resinoso é suscetível à presença de defeitos e falhas mecânicas por fratura.

Materiais e Métodos: Inicialmente, uma pesquisa de literatura foi feita na PubMed de artigos publicados entre Janeiro de 2008 até Maio de 2022 usando as palavras chaves seguintes: "onlay" AND "resin composite" AND "resin cement" AND "thickness" OR "defect" OR "pore" or "fissure" AND "microscopy". Onlays em resina composta (GrandioSO™ block, VOCO, Germany) foram produzidos por CAD-CAM e cimentados sobre as superfícies dentarias seguindo o protocolo padrão. Os espécimenes foram divididos em quatro grupos consoante o tipo de cimento. Os grupos de espécimenes foram seccionados para a avaliação da microestrutura por microscopia ótica com uma ampliação de x50 até x1000.

Resultados

Nas imagens obtidas por microscopia ótica, os maiores valores médios de espessura de camada de cimento foram registados a 405 µm para um dos cimentos resinosos. Diferenças estatísticas nas espessuras vertical ou horizontal foram detetadas entre o cimento resinoso e os outros grupos ($p < 0.05$). Nas margens das restaurações com onlay, os maiores valores médios de espessura de camada de cimento foram registados a 285 µm para os outros cimentos resinosos ($p < 0.05$). Os valores mais baixos de espessura foram registados para as resinas compostas mais fluidas. Nenhuma diferença estatística foi registada entre as resinas compostas ($p < 0.05$). A camada de adesivo foi observada em várias imagens. A espessura da camada de adesivo variou de 12 até 40 µm para os cimentos resinosos enquanto as resinas compostas apresentaram uma espessura de 7-10 µm.

Conclusions

Na cimentação sob baixa carga, a espessura da camada de cimentos resinosos e de compósitos foi variável nas interfaces do onlay com a dentina e o esmalte. Dois compósitos resinosos fluidos revelaram características de viscosidade e microestruturas adequadas para a cimentação quando comparadas com dois cimentos resinosos. No entanto, um aumento da carga na cimentação poderia promover o escoamento dos cimentos resinosos e do adesivo resultando numa espessura adequada da camada nas interfaces entre o onlay com a dentina ou o esmalte.

Abstract

Background: The resin matrix-cement layer can vary at indirect restorations to tooth interfaces depending on the cementation procedure and type of resin-matrix cement. An increased thickness of resin-matrix cement is susceptible to defects and mechanical failures by fracture.

Purpose: Thus, the main aim of this study was to evaluate the microstructure and the thickness of the resin-matrix cement and flowable composites at resin-matrix composite onlays to dentin and enamel interfaces after cementation on low loading.

Materials and Methods: At first, a literature search on PubMed was conducted on the articles published from January 2008 up to May 2022 using the following search terms: "onlay" AND "resin composite" AND "resin cement" AND "thickness" OR "defect" OR "pore" or "fissure" AND "microscopy". Resin-matrix composite onlays (GrandioSO™ block, VOCO, Germany) were manufactured by CAD-CAM and then cemented to tooth surfaces after drilling, shaping, cleaning, and conditioning with adhesive following standard guidelines. Specimens were divided into four groups regarding the type of resin-matrix cement. Groups of specimens were cross-sectioned for microstructural evaluation by optical microscopy at magnification from x50 up to x1000.

Results

The highest mean values of resin-matrix layer thickness were recorded at 405 μm for one of the resin-matrix cements. Statistical differences in vertical or horizontal thickness were detected between the resin-matrix cement and the other groups ($p < 0.05$). Considering the margins of the onlay restorations, the highest mean values of resin-matrix layer thickness were recorded at 285 μm for another resin-matrix cement ($p < 0.05$). The lowest values of layer thickness were recorded for the flowable resin-matrix composites. No statistical differences were recorded between the resin-matrix composites ($p < 0.05$). The adhesive layer was detected in several images. The thickness of the adhesive layer ranged from 12 up to 40 µm for the resin-matrix cements while the resin-matrix composites showed a thickness of around 7-10 µm.

Conclusions

On low loading cementation, the layer thickness of resin-matrix cements and composites was variable at the onlay restorations to dentine and enamel interfaces. Two flowable resin-matrix composites revealed proper flowing and microstructure features when compared with two resin-matrix cements. However, an increase in the cementation loading could promote the flowing of the resin-matrix cements and adhesive resulting in adequate layer thickness at the onlay restoration to dentin or enamel interface.

Key words: onlay, resin composite, cementation, resin cement, dentin

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1. Introduction

Dental restorations such as crowns or onlays can be performed by using chair-side clinical techniques or laboratorial procedures (1–4). The type of dental restoration is dependent on several factors related to the tooth damage, remanent tooth tissues, aesthetic outcomes, and patient-related conditions (2,5,6). For instance, the damage of teeth involving cusps determines the indication for onlays restorations depending on the degree of loss of teeth structures (6–9).

The computer-assisted design/computer assisted manufacturing (CAD-CAM) has been increasingly used for the designing and the manufacturing of dental restorations (10–14). Nevertheless, the use of CAD-CAM was firstly restricted to manufacturing complete denture due to the limitations of CAD software. Nowadays, several commercial CAD software systems are available for designing onlay restorations (4,7,9,15,16). Also, the CAD-CAM technology allows to accomplish the onlay restoration over chair-side procedures (4,7,17). At first, the digital impression by CAD is performed and then the manufacturing process of the onlay is carried out on milling (CAM) a block or disc of resin-matrix composite or ceramic (10,17,18). The CAD system allows to register the maxillofacial relationship, occlusal plane orientation, tooth mold, fitting, and the selection of shade and colors (10,11,19). CAD-CAM blocks of resin-matrix composites are industrially polymerized under standard and controlled conditions (temperature and pressure) to form hybrid, nano-filled or nano-hybrid resin composites (7,15,18,20). Thus, the mechanical and optical stability are higher when compared to traditional manufacturing procedures.

The cementation of onlay restorations over tooth structures is currently performed by using resin-matrix cements (6,21–25). The organic matrix of resin-matrix cements is composed of a cross-linking among methacrylate monomers (22–60%wt) such as Bis-

GMA, UEDMA, and TEGDMA, while the inorganic content is composed of ytterbium fluoride, colloidal silica, zirconium or barium silicate fillers (5,26). The combination of monomers and content of inorganic fillers determine the mechanical properties and flowability of the resin-matrix cements. The cementation procedures involve an intrinsic technique sensitivity leading to a variable thickness of resin-matrix cement (21–25). Several studies recommend a previous conditioning of the onlay inner surfaces with silane and methacrylate-based adhesive systems to enhance the bonding to resin-matrix cements and the long term-success of the restoration (21,23,25,27,28). The low-viscosity of the methacrylate-based adhesive promotes a flowability throughout micro-scale peaks and valleys and. On cementation, the resin-matrix cement does compress the low-viscosity adhesive towards micro-scale retentive regions to establish the mechanical interlocking after polymerization.

Flowable resin-matrix composites can become alternative materials for cementation (29–31). That can bring advantages on the mechanical performance of the interface considering resin-matrix composites have higher elastic modulus, fracture toughness, and flexural strength when compared with resin-matrix cements (32,33). The organic matrix involves a cross-linking of several methacrylate-based monomers (ie., Bis-GMA, TEGDMA, UDMA, BisEMA) embedding inorganic fillers ranging from 40 up to 90 wt% composed of colloidal silica, ytterbium fluoride, and zirconium or barium silicates (32,34,35). Inorganic fillers are added at different size and morphological aspects although currently available resin composites involves a combination of micro- (1-5 µm) and nano-scale (40-60 nm) particles (32,34,35). However, the viscosity and thickness of the cement layer using resin-matrix composites are the major issue since a minimum cement layer is clinically recommended.

2. Objective and hypothesis

The main aim of the present project is to evaluate the microstructure and the thickness of the resin-matrix cement or composite layer at onlay restoration to dentin and enamel interfaces after cementation on low loading. It was hypothesized that the resin-matrix cement or composite layer varies at the onlay restorations to dentine and enamel interfaces depending on the type of material used after cementation on low loading.

3. Materials and Methods

3.1. Search of previous studies

At first, a literature review was performed on PubMed database to identify relevant studies regarding microscopic analyses of the resin-matrix cement layer at the resin-matrix composite onlay restorations to tooth interfaces. The literature search on PubMed (via National Library of Medicine) was conducted using the following a combination of key words and MeSH terms: "onlay" AND "resin composite" AND "resin cement" AND "thickness" OR "defect" OR "pore" OR "fissure" AND "microscopy". The inclusion criteria encompassed articles published in English language from January 2008 up to May 2022 on the cementation of onlay restorations over human teeth. The eligibility inclusion criteria used for article searches also involved *in vitro* studies, meta-analyses, randomized controlled trials, and prospective cohort studies. Also, a hand-search was performed on the reference lists of all primary sources and eligible studies for pursuing relevant findings. Studies based on publication date were be restricted during the search process. Selected studies were individually read and analyzed concerning the focus of the present review, which was to discuss and summarize findings on the microstructure and thickness of resin-matrix cement layer at the onlay restoration to dentin and enamel interfaces. Author names, journal, publication year, objectives, methods, and main outcomes were retrieved from the selected relevant articles. The present search of studies was carried out in accordance with previous integrative review articles (5,26,34,36).

3.2. Preparation of extracted teeth

Twelve extracted third molars gathered from human participants were firstly immersed in distilled water for 10 min and then in a solution of 2% sodium hypochlorite (NaOCl) for 10 min. Afterwards, the teeth were immersed in a 10% formalin solution for 7 days. Finally, teeth will be stored in 0.9% NaCl solution for rehydration over a period of 7 days prior to the cementation procedure (13,17,20,21). The use of extracted teeth was approved by the Human Research Ethics Committee at the University Institute of Health Sciences, cod. 13/CE-IUCS/CESPU/2022, that is in accordance with the Helsinki declaration of 1964. The volunteers signed the informed consent prior to inclusion in the study since the purpose of the study was described. The participants did not suffer from any systemic diseases. Each participant was in good oral health, with no history of antibiotic treatment during the previous 6 months.

At first, onlay preparation was drawn on each tooth with 4 mm-depth measure from the occlusal cuspid (Figure 1A). Then, a standard onlay preparation was performed using taper conical diamond burs following standard guidelines for cementation of onlay preparation. Therefore, onlay preparations involved removal of one occlusal cusp and tooth preparation with 4 mm depth (Figure 1B). All preparations presented smooth inner angles and rounded transitional surfaces using spherical diamond burs. All the teeth were sectioned at the root with a taper conical end diamond burs (4,6,37–39). Teeth were mounted in acrylic resin using a dental inspector apparatus (Ney surveyor, Germany) to align the pulp floor preparation at parallelly to the plan of surface (Figure 1A and E). Tooth preparations were scanned using a digital scanner (S600 Scanner™, Zirkozahn, Germany) for further milling of the onlays.

On the tooth preparation, dentin and enamel were etched with orthophosphoric acid (Optibond ™, Kerr, Germany) for 15 s and 30 s, respectively. Then, surfaces were rinsed with air/water jet for 30 s. The excessive amount of water on dentin and enamel was removed using cotton. At last, dentin and enamel were conditioned by a universal adhesive by rubbing with a microbrush for 20 s. An oil-free air was applied onto the adhesive layer for 5 s.

3.3. Preparation of onlay restorations

CAD files were acquired from digital scanning for each specimen using the Archiver software™ (Zirkozahn, Germany) (Figure 1B). The modelling resolution of the preparation area and the onlay restoration was cautiously enhanced considering the tooth morphological aspects. CAD files were exported as STL files using the Modellier software™ (Zirkozahn, Germany). The STL files and specimens were correlated for further cementation of the onlay restorations as seen in Figure 1.

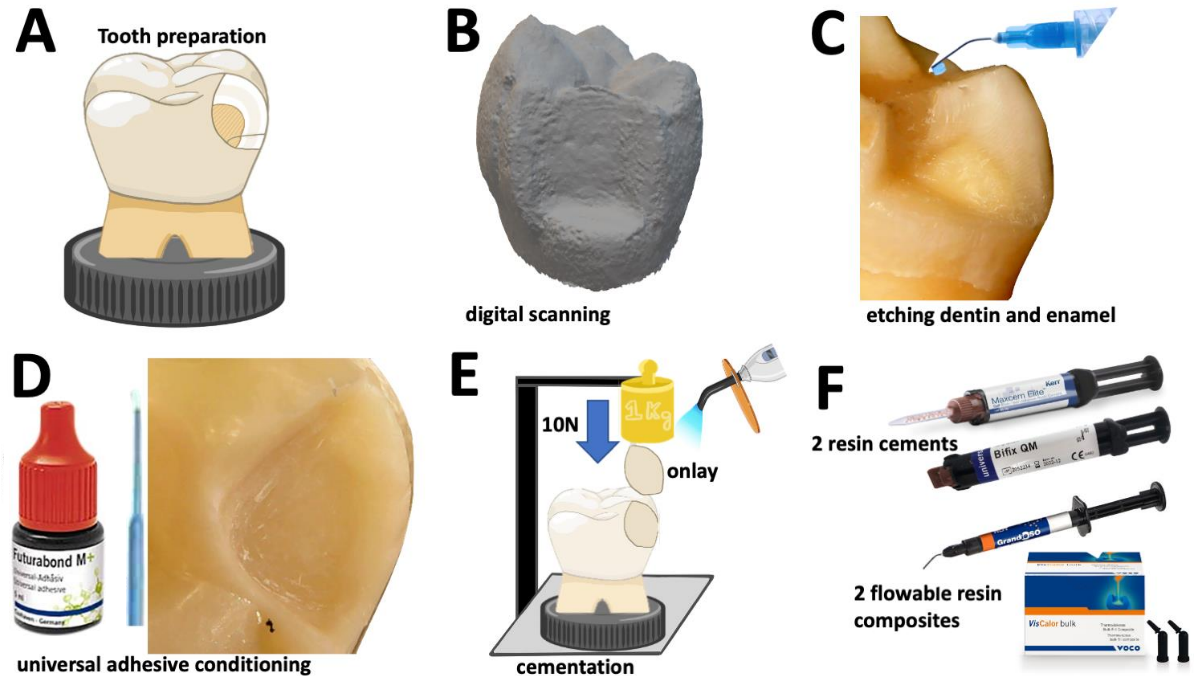

Figure 1. Schematics of the preparation of specimens for optical microscopy. (A) Tooth preparation and (B) digital scanning image. (C) Total etching procedure and (D) adhesive conditioning on dentin and enamel. (E) Cementation on 1 kg loading. (F) Resin-matrix cements.

Twelve specific resin-matrix composite onlays (GrandioSO disc™, VOCO, Germany) were produced by CAD-CAM to ensure accurate placement over each tooth preparation. Onlay restorations were milled using a CAM (Imes-icore™, Coritec 250i,Germany) operated by a software (Hyperdent™, LaserMaq, Portugal). The positioning of the onlays were confirmed using a predictive animation of the milling process prior to the manufacturing. The inner surfaces of the onlay restorations were grit-blasted with 50 µm aluminum oxide (Al_2O_3) at 2 bar and 10 mm away from the surface for 20s. Surfaces were ultrasonically cleaned in isopropyl alcohol for 10 min and then in distilled water for 10 min. The inner surfaces of the onlay were conditioned with a silane compound for 60 s and the gently oil-free air dried for 5 s (2,6). At last, the inner surfaces were conditioned by a universal adhesive (Futurabond M+™, VOCO, Germany) by rubbing with a microbrush for 20 s. An oil-free air was applied onto the adhesive layer for 5 s to remove any solvents (2,6).

3.4. Cementation procedure and specimens

For cementation, two self-adhesive resin-matrix cements were assessed: group M (Max Cem Elite™, KERR, USA); and group B (Bifix QM ™, VOCO, Germany). Also, a traditional flowable resin-matrix composite, named group G (GrandioSO Heavy Flow ™, VOCO, Germany) and a thermal-induced flowable resin-matrix composite, name group V (VisCalor bulk- fill ™, VOCO, Germany), were assessed in this study for comparison with resin-matrix cements (Figure 1F and Table 1). The universal adhesive was not light-cured before the cementation with the resin-matrix cements and the traditional flowable resin-matrix composite. Before the cementation with the thermal-induced flowable resin-matrix composite, the universal adhesive was light-cured under visible light (400-500 nm) using a LED unit (SmartLite Focus ™, Dentsply Sirona, USA) at 1200 mW/cm^2 for 20 s. The resin-matrix cements and composites were applied onto each onlay inner surfaces and then placed over the corresponding tooth preparation on 10 N (1 kg) axial loading for 60 s. The thermal-induced flowable resin-matrix cement was heated up to 61° C to achieve a flowable consistency for application following the manufacturer's recommendations.

A silicone key and the dental inspector apparatus were used to ensure a positioning stability avoiding horizontal dislocation of the restoration and tooth on axial loading (24,25). The excessive cement layer was removed with a microbrush (2,6) and then the materials were light cured under visible light (400-500 nm) using a LED unit (SmartLite Focus ™, Dentsply Sirona, USA) at 1200 mW/cm^2 for 40 s per segment (2,6,40). The clinical cementation loading was mimicked following previous studies (6,24,41).

Table 1. Details on the commercial materials used in this study.

Material (Brand, Manufacturer)	Organic matrix	Fillers %(w/w)	Fillers %(v/v)	Filler type	Elastic modulus (GPa)
Dual-curing resin cement (Max Cem Elite™ , KERR Orange, USA)	Bis-GMA, HEMA, GPDM; UDMA; 1,1,3,3-tetramethylbutyl hydroperoxide TEGDMA,CHPO,MEHQ Bis-GMA, GPDM, co-monomers (33wt%)	67	46	Barium alumina silica glass, fluoroalumina silicate glass borosilicate (30-60%) glass, Ytterbium fluoride (10-30%), amorphous silica (1-5%). (size ~ 3.6 µm).	4.5
Dual curing, resin cement (Bifix QM™, VOCO, Germany)	Bi-functional methacrylate, acid methacrylate, Bis-GMA, benzoyl peroxide, amines and BHT, UDMA, Gly-DMA, phosphate monomers (30 wt%)	70	61	Glass fillers, amorphous silica; (size ~ 2. 9 µm)	6-7.5
Flowable resin composite (GrandioSO Heavy Flow™, VOCO, Germany)	BisGMA, BisEMA, TEGDMA, HDDMA, CQ, amine and BHT (17 wt%)	83	68	nanoparticles of SiO_2 (size ~ 20–40 nm); glass-ceramic; (size ~ 1 µm)	11.5
Thermal-induced flowable resin composite (VisCalor bulk- fill ™ , VOCO, Germany)	Bis-GMA, aliphatic dimethacrylate (17 wt%)	83	68	nanoparticles of SiO_2 (size ~ 20–40 nm); glass-ceramic (size ~ 1 µm)	12.3-17.5

Onlay restorations cemented over teeth assemblies were embedded in autopolymerizing polyether-modified resin (Technovit 400; Kulzer GmbH) in polyvinyl chloride mold (2). Then, assemblies were cross-sectioned at 90 degrees relative to the plane of the restoration pulp floor. Specimens were cross-sectioned by wet-griding on low speed using a standard laboratory materialographic machine (Struers, USA) and SiC papers ranging from 120 down to 2400 mesh (2). Surfaces were ultrasonically cleaned in isopropyl alcohol for 5 min and the dried at room temperature. A photomicrography of a cross-sectioned specimen at x10 is shown in Figure 2A.

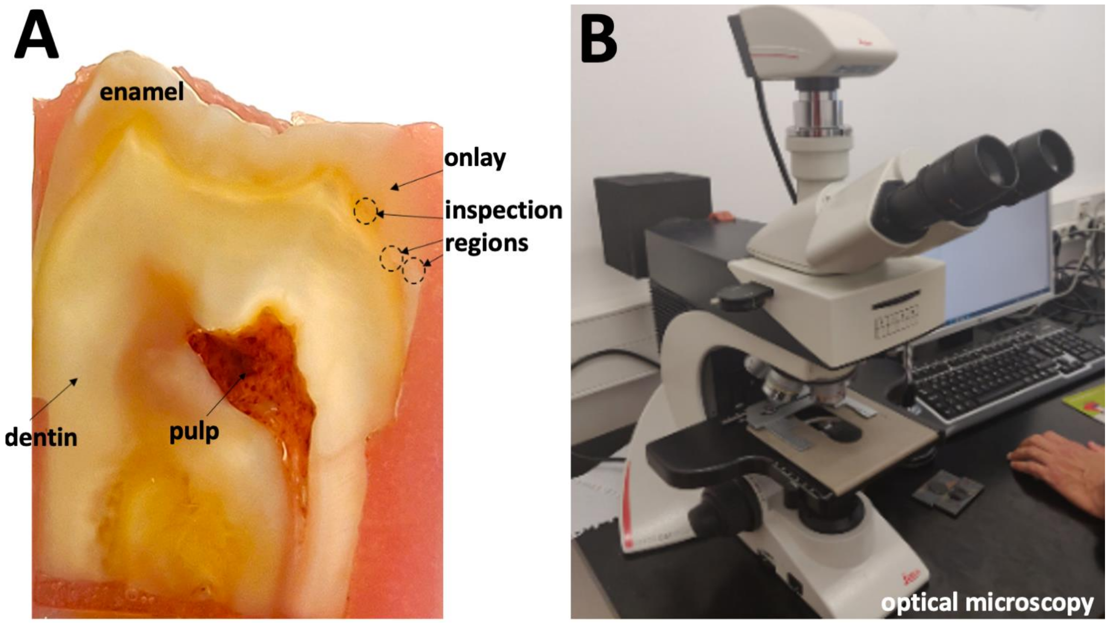

Figure 2. (A) Optical micrography at x10 of the cross-sectioned onlay to dentin and enamel interface. (B) Optical microscopy.

3.5. Microscopic analyses

Cross-sectioned specimens were inspected by optical microscopy at magnification ranging from x50 up to x1000. Microstructural analyses were performed using an optical microscope (Leica DM 2500 M™; Leica Microsystems, Germany) connected to a computer for image processing as seen in Figure 2B. Images were acquired using a Leica Application Suite™ software (Leica Microsystems, Germany). A number of six micrographs were acquired at x50 magnification, for each specimen ($n = 18$). The software Adobe Photoshop™ (Adobe Systems Software, Ireland) was used to analyze black and white images, with the black regions representing the pores and the white regions representing the bulk material. The measurement of thickness dimensions of the resin-matrix and adhesive layers was performed by using Image J™ software (National Institutes of Health, USA).

Results were statistically analyzed by normality test Shapiro-Wilk and two-way ANOVA to determine statistical differences in the resin-matrix cement thickness values between groups. The t student test was used to compare the resin-matrix cement thickness results regarding the use of etching before the cementation. A probability value <0.05 was considered significant. The power analysis was performed by t student test or ANOVA, to determine the number of samples for each group (n), and to reveal a test power of 100% in the present study. Statistical analyses were carried out using Origin Lab statistical software (Origin Lab, Northampton, MA, USA).

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4. Results

Optical microscopy images of the interfaces of the onlay restorations cemented using a resin-matrix cements and shown in Figure 3 and 4.

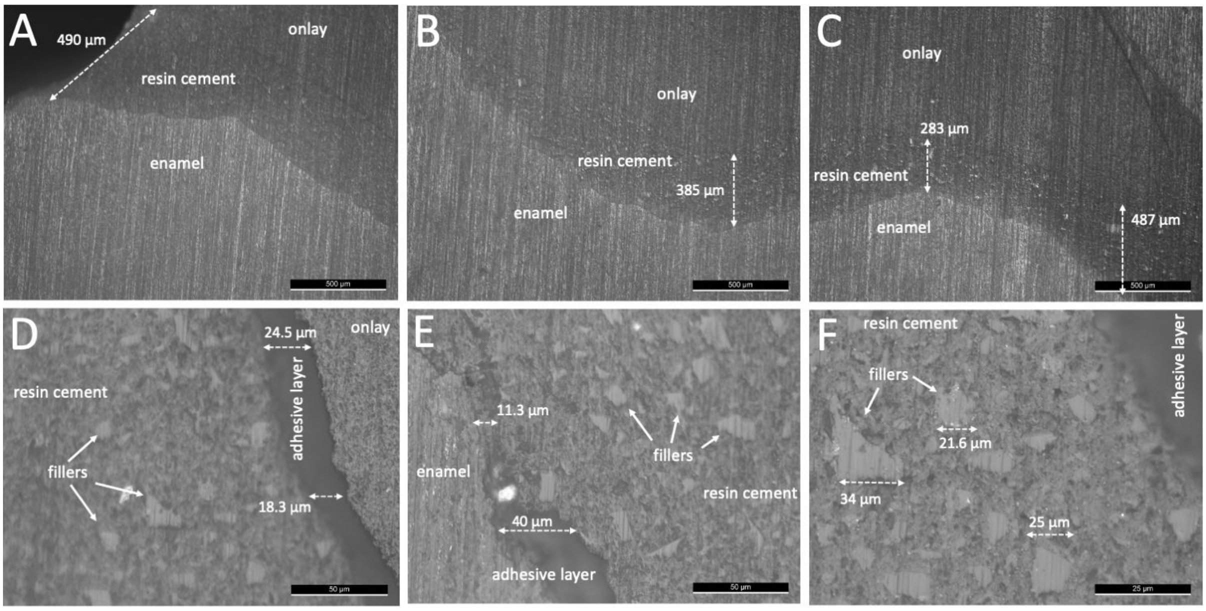

Figure 3. Optical microscopy images of onlay restorations cemented with MaxCem resin cement at magnification of (A,B,C) x50, (C,D), x500, and (E) x1000.

The resin-matrix cement specimens from group M showed a thick layer at the coronal and margin regions as seen in Figure 3. On the coronal region, the thickness values of the resin-matrix cement achieved up to 490 µm while the thickness at the margin was around 480 µm. The thickness of the resin-matrix cement layer ranged from 280 up to 490 µm.

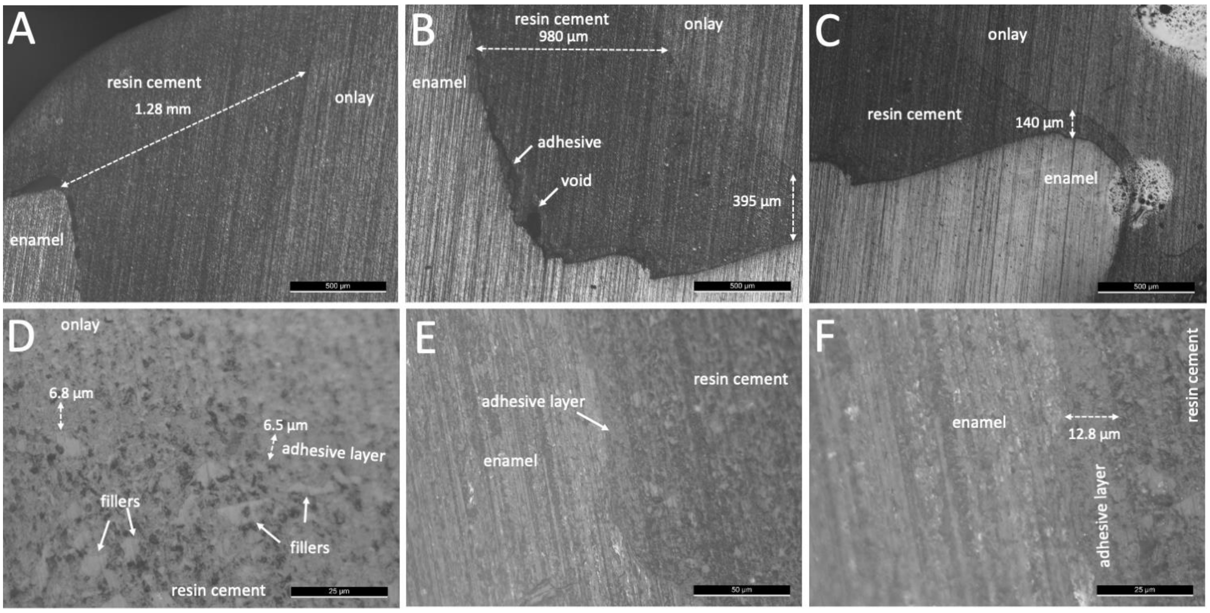

Figure 4. Optical microscopy images of onlay restorations cemented with Bifix resin cement at magnification of (A,B,C) x50, (C,D), x500, and (E) x1000.

Also, the adhesive layer did not flow on cementation resulting in an adhesive cement layer thickness ranging from 18 up to 40 µm as seen in Figure 3D, E and F. On higher magnification images, inorganic fillers were noticed with a mean size ranging from 20 up 35 µm (Figure 3D). As seen in Figure 4, the resin-matrix cement specimens from group B also showed a thick layer at the coronal and margin regions. On the coronal region, the thickness values of the resin-matrix cement achieved up to 1.2 mm while the thickness at the margin showed the lowest mean values of around 140 µm. Indeed, the low loading at 10 N did not provide the fitting of the onlay restoration resulting in a thick resin-matrix cement layer from both groups M and B (Figure 3 and 4). In the same way, the adhesive layer did not flow on cementation resulting in a mean thickness value of adhesive cement layer at 12.8 µm as seen in Figure 4E and F. On higher magnification images, inorganic fillers were noticed with a mean size at 7 µm (Figure 4D).

Optical microscopy images of the interfaces of the onlay restorations cemented using a flowable resin-matrix composites are shown in Figure 5 and 6. The flowable resin-matrix composite specimens from group M showed a thick layer at the coronal and margin regions as seen in Figure 5. On the coronal region, the thickness values of the resin-matrix composite achieved up to 490 µm while the thickness at the margin was around 480 µm. The thickness of the resin-matrix composite layer ranged from 280 up to 490 µm. The adhesive layer was also noticed as seen in Figure 5F. On higher magnification images, inorganic fillers were smaller than 5 µm (Figure 3D).

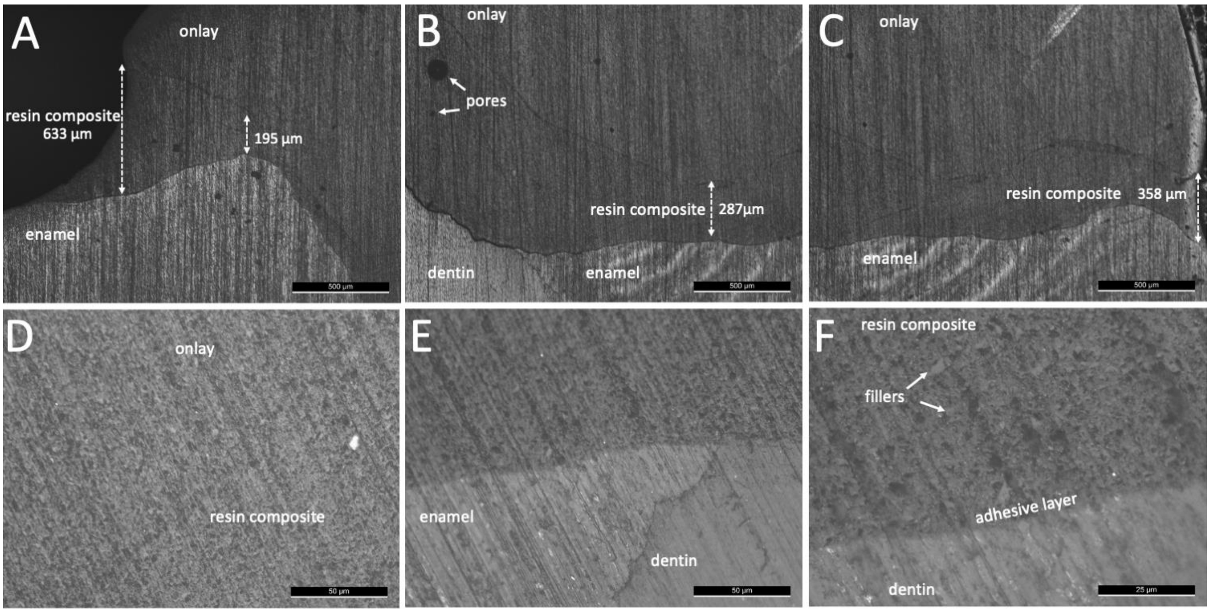

Figure 5. Optical microscopy images of onlay restorations cemented with GrandioSO heavy flow at magnification of (A,B,C) x50, (C,D), x500, and (E) x1000.

The thickness of the thermal-induced flowable resin-matrix composite specimens from group V at the coronal region was recorded at 455 µm and the margin interface revealed a thickness of around 158 µm (Figure 6). Low thickness values of the thermal-induced flowable resin-matrix composite layer were recorded at 38 and 56 µm (Figure 6C and D). The thickness of the adhesive layer was measured at around 7-10 µm (Figure 6F).

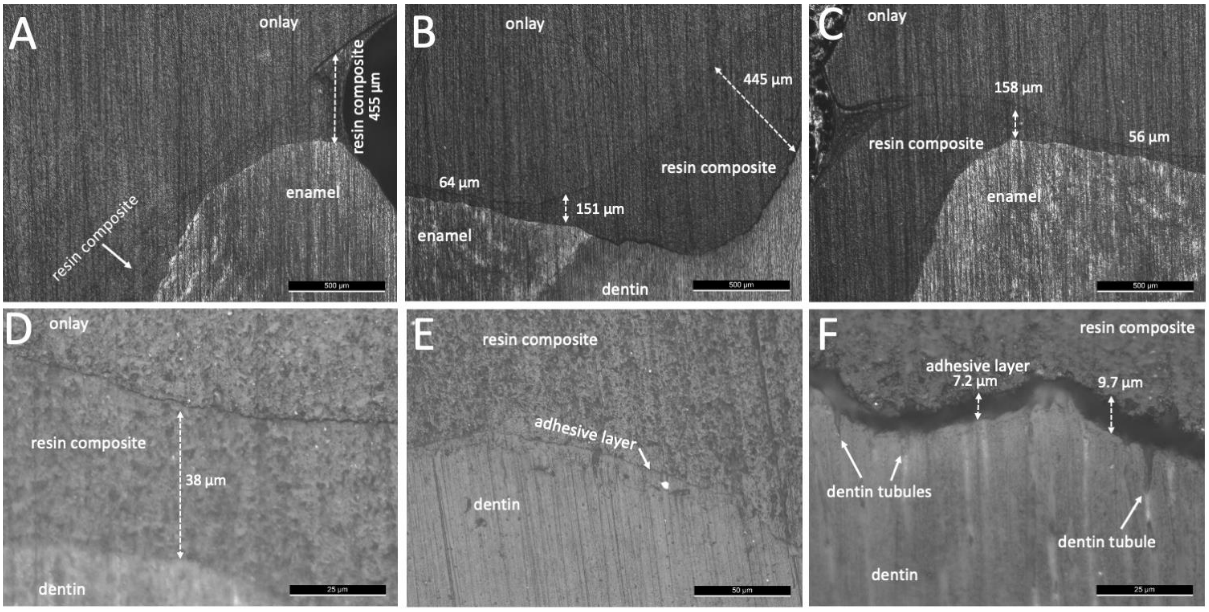

Figure 6. Optical microscopy images of onlay restorations cemented with Viscalor Bul fil at magnification of (A,B,C) x50, (C,D), x500, and (E) x1000.

Mean values of layer thickness recorded for resin-matrix cements and composites are shown in Figure 7.

The highest mean values of resin-matrix layer thickness were recorded at 405 µm for group B. Statistical differences were detected between group B and the other groups ($p < 0.05$). Considering the margins of the onlay restorations, the highest mean values of resin-matrix layer thickness were recorded at 285 µm for group M and statistical differences were detected between group M and B or G ($p < 0.05$). The highest vertical and horizontal values of resin-matrix layer thickness were recorded for group B at 385 µm and 425 µm, respectively. Statistical differences in vertical or horizontal thickness were detected between group B and the other groups (p <0.05). No statistical differences were recorded between the resin-matrix composites.

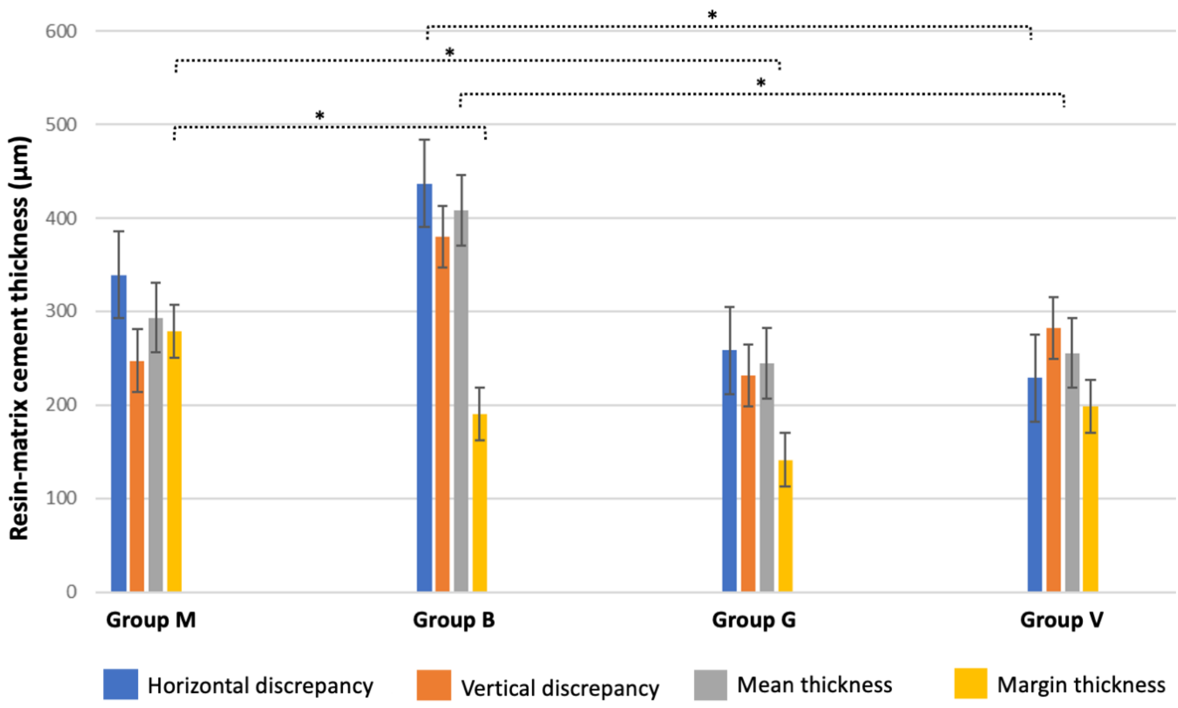

Figure 7. Mean values and standard deviation of the layer thickness values recorded for resin-matrix cements (M and B) and composites (G and V) at different parameters. Statistical differences were identified as * (p <0.05).

5. Discussion

This study reported a detailed microscopic evaluation of the microstructure of resin-matrix cements and flowable resin-matrix composites after cementation of resin-matrix composite onlays to tooth surfaces. Also, the measurement of the layer thickness of the resin-matrix cements and flowable resin-matrix composites was carried out at different regions after cementation on loading. The resin-matrix cement and flowable composites revealed an irregular layer thickness and presence of defects such as macro-scale voids and pores. The adhesive showed also a thick layer due to the low flowability of the materials. Thus, the findings validate the hypothesis of this study. A comprehensive discussion on the main factors affecting the cementation of resin-matrix cements and flowable composites is fundamental to guide professionals in choosing the type of materials and mode of cementation.

After grit-blasting with alumina particles, a conditioning of the surfaces using silane and methacrylate-based adhesive provide a high integrity interface with the resin-matrix cements and flowable composites (2,42–46). At first, a coating with silane increased the surface wettability of surfaces by condensing hydroxyl and SiO_2 groups onto the onlay surfaces. Then, a chemical bonding takes place through SiO_2 and hydroxils groups on the onlay inner surface. On the cementation, a chemical reaction occurs between free radicals in the monomers' matrix and the hydroxyl and SiO_2 groups to establish a chemical bonding (36). Micro-scale irregularities on the tooth and onlay inner surfaces can be filled by conditioning with low-viscosity methacrylate-based adhesives such as universal adhesive used in the present study. Then, rough surfaces are coated with the adhesive layer and resin-matrix cement establishing a mechanical interlocking after polymerization (47,48). A relatively viscous resin-matrix cement or flowable composite could not reach the deepest micro-scale valleys on the surface without the adhesive layer. A low flowing of a resin-matrix cement and flowable composite promotes the formation of pores or voids and the lack of mechanical retention. Then, an absence of adhesive between indirect restorations and resin-matrix cement or composites could decrease the bond strength of the interface (6,21,24,49,50). Another issue is related to the application of low-viscosity adhesive by using a hand-held micro-brush under reciprocating sliding (rubbing movement) onto the surfaces for 20 s. The adhesive conditioning has an intrinsic sensitivity concerning the operator-induced factors such as: movement, load, time, air drying, and amount of adhesive. Consequently, the layer thickness of adhesive also varies depending on the application mode, surface conditions, and type of adhesive. In this study, the thickness of the adhesive layer ranged from 12 up to 40 µm for the resin-matrix cements while the resin-matrix composites showed a thickness of around 7-10 µm. As seen in Figure 4 and 6, a thick layer of low-viscosity methacrylate-based adhesive was accumulated at certain regions of the surfaces probably due to the amount and low loading cementation. On the physical properties, the adhesive layer is the most mechanically susceptible material at the interface and therefore mechanical failures can take place by stresses under polymerization shrinkage, mastication loading (1-500 N), or thermal oscillations (i.e, 5- 50° C). The strength and elastic modulus values of the adhesive are lower when compared to the resin-matrix cement and flowable resin composites (Table 1).

As seen in optical microscopy images, the highest values of resin-matrix layer thickness were recorded for the resin-matrix cements. Indeed, the low loading cementation affected the fitting and flowing of the resin-matrix cements. On the other hand, the thermal-induced and the traditional flowable resin-matrix composites revealed a proper flowing considering the lower values of layer thickness after low loading cementation.

Cementation procedures at low loading can occur in clinical situations since it depends on the professional expertise and sensitivity. A previous study measured the cementation pressure applied from different dentists the loading values ranged from 12 up 67 N (51). The loading magnitude assessed in this study corroborates with the values reported in literature. Thus, the fitting and strength of the interface are enhanced when the cementation loading increased (49,52). However, marginal discrepancy dimensions at indirect restorations to tooth surfaces should be less than 100 µm (52). An increase in the layer thickness of resin-matrix cements or flowable composites also increase the risks of defects such as micro- and macro-scale pores and voids, as seen in Figure 4 and 5.

Thus, the thickness of the cementation layer can be affected by the inorganic filler content, the filler size, organic matrix components, materials' viscosity, and the polymerization reaction (5,21,26). The traditional flowable resin-matrix composites revealed an adequate viscosity and therefore flowing on low loading cementation. A pre-heating of thermal-induced flowable resin-matrix composites enhanced both radical and monomer mobility resulting in higher overall flowing and degree of conversion of monomers. The size of inorganic fillers of a resin-matrix cement was detected at approximately 35 µm that determined its minimum layer thickness. Additionally, the size of the micro-scale inorganic fillers at high content (67wt%) decreases the viscosity of the resin-matrix cement. As seen in Table 1, the flowable resin-matrix composites possess micro- (1-3 µm) and nano-scale (40-60 nm) dimensions at high content (83wt%) that avoided a large cementation layer.

The present in vitro study revealed a detailed microscopic analysis of onlay restorations cemented to dentin and enamel surfaces using resin-matrix cements and flowable composites. However, limitations are related to the sensitivity of the adhesive conditioning and cementation procedures even though a single operator performed the preparation of specimens. Indeed, the loading and mode of cementation are dependent from the operator. The fitting of the onlay restoration to the tooth preparation depends on the digital scanning resolution and processing of onlays. This study focused on the use of resin-matrix composite onlay prepared by using a standard CAD-CAM protocol although the assessment of lithium disilicate or zirconia could be interesting for comparison with the composite blocks regarding the prosthetic fitting, surface conditions, and the layer thickness of the resin-matrix cements and flowable composites. The treatment of the onlay inner surfaces was performed only using grit-blasting with alumina particles followed by conditioning with silane compounds and methacrylate-based adhesives. The increase of roughness of the inner surface of the onlay restorations also increases the mechanical interlocking of the adhesive and resin-matrix cement or flowable composite. Several types of adhesives and resin-matrix cements should be assessed since their chemical composition and physical properties determine the mechanical interlocking, the cementation layer thickness, and the mechanical integrity of the interfaces. Considering the cementation, the loading can vary and therefore it should be correlated with the type of resin-matrix cement and flowable composite. The polymerization procedures should be controlled regarding the equipment conditions, mode, and exposure time. In fact, the cementation procedures of prosthetic crowns onto titanium base are not well-designed and the all the above-mentioned variables do affect the long-term success of the restorations.

6. Conclusions

Within the limitations of this in vitro study, the main concluding remarks can be drawn as follow:

- Higher mean values of resin-matrix layer thickness were recorded for resin-matrix cements when compared to flowable resin-matrix composites after cementation at low loading. The layer thickness of the resin-matrix cements and flowable composites varied along the onlay to dentin and enamel surfaces. An increased cementation layer thickness is more susceptible to the presence of defects such as micro- and macro-scale pores and voids;
- The adhesive layer also varied at the interfaces due to the lack of flowing of the resin-matrix cement and flowable composites on low loading cementation. A thick layer of adhesive and resin-matrix cement or flowable composite also negatively affect the mechanical integrity of the interface since those materials reveal the lowest values of mechanical properties such as elastic modulus, flexural strength, and fracture toughness;
- Cementation procedures on low loading can occur in several clinical situations due to the operator technical sensitivity. An increase in the cementation loading could promote the flowing of the resin-matrix cements and adhesive resulting in adequate layer thickness at the onlay restoration to dentin or enamel interface.

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