U.PORTO

MASTER

ORAL REHABILITATION

Estudo comparativo de protocolos de condicionamento de cerâmicas para restaurações adesivas.

Elsa Cristina Reis Carneiro

M

2022

U.PORTO

MASTER

ORAL REHABILITATION

Ceramic conditioning protocols for adhesive restorations: a comparative study.

Elsa Cristina Reis Carneiro

M

2022

Dissertação apresentada à Faculdade de Medicina Dentária da Universidade do Porto, para obtenção do grau de Mestre em Reabilitação Oral

Autora

Elsa Cristina Reis Carneiro
Aluna do 2º ano do Mestrado em Reabilitação Oral
da Faculdade de Medicina Dentária da Universidade do Porto
elsacreiscarneiro@gmail.com

Orientador

Prof. Doutor Paulo Júlio Andrade de Almeida
Professor Associado Convidado
da Faculdade de Medicina Dentária da Universidade do Porto

Coorientador

Doutor Luís Miguel Vilhena Pereira da Silva
Investigador Auxiliar do Departamento de Engenharia Mecânica
da Faculdade de Ciências e Tecnologia da Universidade de Coimbra

Porto, 2022

“Enquanto não alcances
Não descanses.
De nenhum fruto queiras só metade.”

Miguel Torga

Acknowledgments

Ao Prof. Doutor Paulo Almeida,

Pela simpatia, partilha de saber, dedicação e orientação durante este percurso.

Ao Doutor Luís Vilhena,

Pelo conhecimento, encorajamento e ajuda preciosa na execução dos ensaios mecânicos.

Ao Professor Doutor Sampaio Fernandes,

Por toda a dedicação e motivação ao longo destes 2 anos.

Ao Eng. Filipe Ferreira,

Pela amizade, disponibilidade e ajuda na preparação das amostras.

Ao Dr. Carlos Almeida,

Pelo valioso contributo no tratamento estatístico dos dados e toda a disponibilidade.

À VOCO e à Exaktus®,

Por fornecerem materiais para este estudo.

À minha Família,

Pelo apoio incondicional, pela paciência e por acreditarem sempre em mim.

Aos meus Amigos,

Pela presença constante.

Ao Porto.

O meu mais sincero bem-haja!

Abstract

Introduction: Despite the development and advances in adhesive restorations, allowing for greater bonding performance, there are still challenges and questions regarding the formulation of new compositions and chemical reactions of dental materials. Glass-ceramics' conditioning with hydrofluoric acid followed by silane is considered the "gold-standard", however some questions arise regarding its handling, due to toxicity and time the protocol takes. The aim of this study was to evaluate and compare three different ceramic conditioning protocols (hydrofluoric + silane/ sandblasting + silane – CP vs. self-etch ceramic primer – MEP vs. sandblasting + self-etch ceramic primer – SDBM), regarding the bond strength and surface characteristics obtained in three different glass-ceramics and a resin nanoceramic, by means of shear bond strength testing (SBS) and surface' etching pattern.

Materials and Methods: A total of 17 blades of 4 different CAD/CAM ceramic (leucite-reinforced glass-ceramic – LEU, feldspathic ceramic – FLD, lithium disilicate ceramic – LDC and hybrid ceramic – HYB) were cut and distributed into three groups, according to the surface conditioning protocol. Afterwards, specimens were divided in two groups: one group not subjected to thermocycling (n=5) and one group submitted to 5000 cycles of TC with temperature variation from 5 ºC to 55 ºC, during 60 s (n=2). Ceramics surface was prepared and bonded 4 resin cubes (3x3x3mm) in each ceramic surface. A single operator carried out all specimens' preparation. Then, each specimen was submitted to shear testing (SBS) using a universal testing machine. Surface roughness was evaluated, and SEM was performed.

Data statistical analysis was performed using Kruskal-Wallis. In the presence of normality, the ANOVA test was performed using Tukey's HSD.

Results: After thermocycling, statistically significant differences in roughness mean values were found in LEU_MEP, LDC_MEP, HYB_MEP, LEU_CP and FLD_SDBM ($p<0.05$). Among all type of ceramics, statistically significant differences in bond strength were only found in LEU_SDBM ($p=0.002$).

Conclusions: All ceramics presented similar bond strength values, regardless the preparation technique and LDC showed the highest values, although not statistically significant. About roughness, SDBM produced higher mean roughness values, despite the type of ceramic. MEP

can be a valid pretreatment for FLD and LDC. Moreover, failures were all by the adhesive joint. Further studies in this line of research are needed with standardized protocols to establish clear relations between the evaluated parameters.

Keywords: self-etching ceramic primer; hydrofluoric acid etching; glass ceramic; alumina sandblasting; silane.

Resumo

Introdução: Apesar do desenvolvimento e avanços nas restaurações aderidas, permitindo maiores forças adesivas, ainda existem desafios e dúvidas quanto à formulação de novas composições, reações químicas dos materiais. O condicionamento das cerâmicas com matriz vítrea com a aplicação de ácido hidrofluorídrico, seguido de silano é considerado o protocolo de eleição, no entanto surgem algumas questões relativas à sua manipulação, devido à toxicidade e ao tempo que demora o protocolo. O objetivo deste estudo foi avaliar e comparar três protocolos diferentes de condicionamento cerâmico (ácido hidrofluorídrico + silano/ jateamento + silano – CP vs. primer cerâmico autocondicionante – MEP vs. jateamento + primer cerâmico autocondicionante – SDBM) em três cerâmicas com matriz vítrea e uma resina nanocerâmica, através de testes de resistência ao cisalhamento (SBS) e do padrão de condicionamento da superfície.

Materiais e Métodos: No total 17 lâminas de 4 cerâmicas CAD/CAM diferentes (cerâmica vítrea reforçada com leucite – LEU, cerâmica feldspática – FLD, dissilicato de lítio – LDC e cerâmica híbrida – HYB) foram cortadas e distribuídas em três grupos, de acordo com o protocolo de condicionamento de superfície. Em seguida, foram criados dois grupos: um grupo não submetido a TC (n=5) e um grupo submetido a 5000 ciclos de termociclagem, com variação de temperatura entre 5 ºC e 55 ºC, durante 60 s (n=2). A superfície da cerâmica foi preparada e foram colados 4 cubos de resina (3x3x3mm) em cada superfície. Um único operador realizou toda a preparação dos espécimes. De seguida, cada amostra foi submetida ao teste de cisalhamento (SBS), usando uma máquina de testes universal. A rugosidade da superfície foi avaliada e SEM foi realizado.

A análise estatística dos dados foi realizada pelo teste de Kruskal-Wallis. Na presença de normalidade, foi realizado o teste ANOVA utilizando o Tukey' HSD.

Resultados: Após termociclagem, foram observadas diferenças estatisticamente significativas nos valores médios de rugosidade nos grupos LEU_MEP, LDC_MEP, HYB_MEP, LEU_CP e FLD_SDBM ($p<0.05$). Entre todos os grupos de cerâmicas, diferenças estatisticamente significativas nos valores de força adesiva foram apenas encontradas no grupo LEU_SDBM ($p=0.002$).

Conclusões: Todos os protocolos adesivos apresentaram valores de SBS semelhantes e, portanto, independentemente do tipo de condicionamento de superfície. Em relação ao tipo de cerâmica, DLC apresentou os maiores valores de resistência ao cisalhamento, embora não sejam estatisticamente significativos. Quanto à rugosidade, o protocolo SDBM produziu maiores valores médios de rugosidade, independentemente do tipo de cerâmica. O MEP pode ser um pré-tratamento válido em cerâmicas FLD e LDC. Todas as falhas que ocorreram foram adesivas. Mais estudos nesta linha de pesquisa são necessários com protocolos padronizados para estabelecer relações claras entre os parâmetros avaliados.

Palavras-chave: primer cerâmico autocondicionante; condicionamento ácido; cerâmicas vítreas; jateamento com óxido de alumínio; silano

Table of Contents

Acknowledgements ix
Abstract xi
Resumo xiii
List of Figures xvii
List of Tables xix
List of terms and acronyms xxi
1. Introduction 1
1.1. Ceramics 1
1.1.1. Glass-based systems 1
1.1.2. Glass-based systems with fillers 2
1.1.3. Crystalline-based systems with glass fillers 2
1.1.4. Polycrystalline solids 2
1.1.5. Resin ceramic materials 2
1.2. Ceramic conditioning protocols 3
1.2.1. “Gold-standard”: hydrofluoric acid + silane 3
1.2.2. Alternative methods 4
1.3. Purpose 5
2. State of the art 7
3. Materials and Methods 9
3.1. Materials 9
3.2. Specimen selection and preparation 10
3.3. Experimental protocol 11
3.4. Laboratory tests 16
3.4.1. Surface roughness 17
3.4.2. Shear bond strength testing 17
3.4.3. Scanning electron microscopy 18
3.4.4. Thermocycling 18
3.5. Statistical analysis 19
4. Results 21
4.1. Roughness 21
4.1.1. Control groups 22

4.1.2. Initial evaluation vs. surface treatment 23
4.1.3. Before vs. after thermocycling 24
4.2. Shear bond strength (SBS) 25
4.3. Scanning Electron Microscopy (SEM) 27
5. Discussion 29
6. Conclusion 31
7. References 33
8. Attachments 37
8.1. Author´s Statement 37
8.2. Advisor´s Statement 38
8.3. Co-Advisor´s Statement 39
8.4. Data Measurement Tables 41

List of Figures

Fig. 1 – Schematic illustrating the division of each CAD/CAM ceramic bloc ……. 10

Fig. 2 – Schematic illustrating the division of each CAD/CAM resin composite bloc ……..…… 10

Fig. 3 – Schematic illustrating the successive steps of surface preparation with CP ……..……... 12

Fig. 4 – Schematic illustrating the successive steps of surface preparation with CP for Cerasmart® ……..……. 13

Fig. 5 – Schematic illustrating the successive steps of surface preparation with MEP …….. 13

Fig. 6 – Schematic illustrating the successive steps of surface preparation with SDBM ……..…….. 14

Fig. 7 – Schematic illustrating the successive steps of surface preparation of resin specimens ……..……. 15

Fig. 8 – Schematic illustrating the bonding procedure ……..……. 15

Fig. 9 – Shear-bond strength test: (a) configuration at the Shimadzu universal testing machine; (b) schematic illustration ……..... 18

Fig. 10 – SBS measurements of four different ceramics for each surface treatment, before thermocycling ……...…….. 25

Fig. 11 – SBS measurements of four different ceramics for each surface treatment, after thermocycling …… 26

Fig. 12 – Micrographs from the adhesive joint from specimen LDC_MEP: (a) adhesive joint surface; (b) micrograph with higher magnification showing corner detail; (c) surface after MEP treatment and before being adhered …… 27

Fig. 13 – Micrograph showing the adhesive joint from specimen LDC_MEP …..…… 28

List of Tables

Table 1 – CAD/CAM blocs studied, manufacturers, lot and expiration date, chemical composition 9

Table 2 – Group distribution, according to surface treatment and type of ceramic 11

Table 3 – Materials studied, manufacturers, lot and expiration date, chemical composition 16

Table 4 – Roughness (Ra, Rz, and Rsk) of HYB, for each surface treatment 21

Table 5 – Roughness (Ra, Rz, and Rsk) of LDC, for each surface treatment 21

Table 6 – Roughness (Ra, Rz, and Rsk) of FLD, for each surface treatment 22

Table 7 – Roughness (Ra, Rz, and Rsk) of LEU, for each surface treatment 22

List of terms and acronyms

LDC	Lithium dissilicate ceramic
FLD	Feldspathic ceramic
LEU	Leucite-reinforced glass-ceramic
HYB	Hybrid ceramic
RNC	Resin nanoceramic
PICN	Polymer-infiltrated ceramic network
CP	Conventional protocol
MEP	Monobond® Etch&Prime
SDBM	Sandblasting + Monobond® Etch&Prime
SEM	Scannig Electronic Microscopy
EDS	Energy-dispersive X-ray spectroscopy
TC	Thermocycling
Ra	Arithmetical mean roughness
Rq	Root mean square
Rsk	Roughness skewness
MPa	Megapascal
SBS	Shear Bond Strength
µm	Micrometre
s	Seconds

1. INTRODUCTION

Minimal invasive restorative techniques are based in micro retention principles that allows better conservation of the dental structure. [1] The success and long-term survival of adhesive restorations depends, among other factors, on the quality and durability of the bond between ceramic, luting agent and dental substrate. [2-4] The main factor is the luting agent, which must guarantee an effective, durable bond between dental structure and the restoration and also provide marginal integrity. [1] Differences in chemical nature of materials leads to variations in their bonding performance to different luting agents and their mechanical properties. [5]

Nowadays, ceramic restorations are widely used due to their superior esthetic properties, toughness, color stability, translucency, fluorescence, and biocompatibility. [6,7]

1.1. Ceramics

The term “ceramic” technically refers to a crystalline material. Porcelain is a mixture of glass and crystal components. On the other hand, non-crystalline containing material is simply an amorphous glass. The structure of ceramics is crystalline, exhibiting a regular periodic arrangement of the component atoms with ionic or covalent bonds. These materials are strong in compression but weak in tension. In what concerns about microstructural classification, these materials can be defined as: glass-based systems, glass-based systems with crystalline fillers interpenetrating phase ceramic/ glass-based systems with glass fillers, mainly alumina; and polycrystalline solids. [8]

1.1.1. Glass-based systems

These ceramics are made from materials that mainly contain silicon dioxide (silica or quartz) and some amounts of alumina. Feldspars are aluminosilicates with various amounts of potassium and sodium. [8]

1.1.2. Glass-based systems with fillers

Their composition has some glass of the pure glass category but with different glass-crystalline ratios and crystal type of leucite, lithium disilicate and fluorapatite. Leucite has a larger amount of potassium oxide and lithium disilicate has an addition of lithium oxide. Low to moderate leucite-containing feldspathic glass are commonly referred as “feldspathic porcelains”. These materials are the typical powder/liquid materials that are commonly used to veneer core systems. The leucite crystals tend to be much finer and have a very even particle distribution. High-leucite-containing glass consists of a glass-matrix surrounding a second phase of individual fillers, formed by a secondary heat treatment that nucleates and grow crystals. Lithium disilicate glass-ceramic has a higher content of micron-size crystals (around 70%) in between submicron lithium orthophosphate crystals, creating a highly filled glass matrix. [8]

1.1.3. Crystalline-based systems with glass fillers

These systems consist at least of two phases that are intertwined and extend continuously from the internal to the external surface. These materials are generally fabricated by first creating a porous matrix and filling them by a second-phase material, a lanthanum aluminosilicate glass. The most common is In-Ceram. [8]

1.1.4. Polycrystalline solids

These materials are formed by directly sintering crystals together with no matrix, such as zirconia and alumina. They are solid sintered monophase ceramics. [8,9]

1.1.5. Resin ceramic materials

Hybrid ceramics are made of PICN whose composition is resin embedded in a ceramic network [10] or RNCs made of nanometer sized ceramic fillers (silica or zirconia) randomly dispersed in a polymer matrix. [11] Therefore, these materials has a hybrid surface that could be conditioned in the same way as either an indirect composite or an etchable ceramic. [10]

1.2. Ceramic conditioning protocols

Adhesion in ceramics requires a surface treatment based on micromechanical retention and chemical bonding [12]. Ceramics pretreatment is based on proper cleaning of the internal surfaces of the restoration, creating roughness patterns on the surface [1], followed by the placement of a ceramic primer promoting chemical bonding to inorganic component of the surface and the resin cement. [2,3,13,14]

Depending on the glass-phase content of the ceramic, it may be sensitive to acid-etching [13] or resistant to acid, as for aluminum oxide ceramics, aluminum oxide ceramics reinforced with zirconium oxide and zirconium oxide ceramics. [1,7] Polycrystalline ceramics are “glass-free matrix”, due to the absence of a glass phase, so it is challenging to create a reliable and durable bonding with hydrofluoric acid etching. On the other hand, glass-matrix ceramics, as lithium disilicate and feldspathic, are conditioned with hydrofluoric acid followed by an application of a silane coupling agent as a standard procedure for adhesive cementation. [14,15]

1.2.1. “Gold-standard”: hydrofluoric acid + silane

Hydrofluoric acid etching followed by priming with a silane coupling agent is considered the gold standard for silica-based ceramics. Hydrofluoric acid is an aqueous solution of hydrogen fluoride that can be used for dissolution of the ceramic glass phase surface by reacting with silicon dioxide. The reaction is based on the fluoride substitution to oxygen due to electronegativity in glass that forms the Si.F glass. [7] Hydrofluoric acid leads to the dissolution of the glassy matrix, creating a rough surface and promoting a mechanical interlocking on the surface [16]. Although this etchant can effectively enhance the strength bond between ceramics and resin, it is highly toxic and volatile [17]. [18] To maximize the affinity with polymers, silane agents are applied after the etching step. Silane is a bifunctional molecule that provides a chemical covalent and hydrogen bond, reacting with the inorganic part of the ceramic and the organic matrix of the resin agent. Thus, enhancing the physico-chemical interaction between the luting agent and the glass ceramic, in order to prepare the surface for adhesive cementation, improve the longevity of indirect restorations [19] and the surface energy. [1,7,13,14,20-22] Silanes have been shown to reduce the contact angle and increase the wettability of the ceramic surface. [23,24]

In what concerns about intraoral repair of chipped ceramics, etching with hydrofluoric acid is recommended to create a more reactive and greater contact surface that increase bond strength of the etched substrate [25,26] and/or air-abrasion and chemical adhesion with the use of silane coupling agents. [27] However, hydrofluoric acid is a caustic product with harmful effects when in contact with the skin and mucous membranes, requiring rigorous handling. [25,28]

1.2.2. Alternative methods

Although the surface treatment with hydrofluoric acid + silane is widely used and accepted for glassy ceramic, other alternatives have been proposed to enhance the bond between ceramic and resin cement, simplifying the protocol and provide greater biosafety. [2,12,14,29]

Monobond® Etch & Prime (Ivoclar Vivadent, Schaan, Liechtenstein) is a newly introduced self-etch ceramic primer, combining ammonium polyfluoride and a silane coupler (trimethoxypropyl methacrylate) in one bottle. The milder etchant produces a partial dissolution of the glassy matrix of ceramics, but still enough to promote an adhesive interlocking with the surface. The silane agent attaches to the ceramic surface forming a permanent thin and stable layer, even after rinsing and drying. This material allows conditioning and silanization of the ceramic surface in just one step. Moreover, the primer aims to eliminate the hazardous effects of the application of hydrofluoric acid as well as reducing the clinical time and technique sensitivity. [13,29-32]

The additional step of sandblasting the ceramic surface with aluminum oxide particles also contributes to mechanical attachment [1,7,22] as well as chemical treatments, such as 10-MDP ceramic primers or a silica coating procedure to promote a durable bond, usually used in ceramics without silica. [1,22]

1.3. Purpose

The aim of the present *in vitro* study is to evaluate and compare three different ceramic conditioning protocols:

1. Conventional protocol (CP): hydrofluoric acid + silane or sandblasting + silane
2. Monobond® Etch&Prime (MEP)
3. Sandblasting + Monobond® Etch&Primer (SDBM),

in four different ceramics (leucite-reinforced glass-ceramic – LEU vs. lithium disilicate – LDC vs. feldspathic ceramic – FLD vs. hybrid ceramic – HYB), regarding the bond strength by means of shear bond strength testing and surface' characteristics by means of roughness values and SEM.

The tested null hypothesis states there are no statistically significant differences between distinct surface' pretreatments protocols on 1) shear bond strength and 2) roughness values for the four different types of ceramic.

2. STATE OF THE ART

The effectiveness of surface treatments can be expressed by several parameters, such as roughness, surface energy, and bond strength. [33]

El-Damanhoury et al. [13] assessed the bond strength between different surface pretreatment methods in ceramics. The mean SBS values of LDC were significantly higher than those recorded for FLD and HYB, regardless the method of surface treatment. The only exception was for HYB prepared with MEP, where no statistically significant differences were found. Also, Lima et al. [4], when comparing the efficiency of pretreatment with CP and MEP in FLD, obtained higher values for FLD_CP than FLD_MEP.

According to Román-Rodriguez et al. [29] and Lima et al. [4], similar results of bond strength in LDC treated with MEP or 4.9% hydrofluoric acid were found. Likewise, Siqueira et al. [24,30] assessed the bond strength between different adhesive protocols, in lithium disilicate. In all experimental groups, solo use of MEP had similar bond strength values to conventional two-step protocol. Therefore, the less-pronounced etching pattern produced by MEP cannot be seen as a disadvantage, since a more-pronounced etching pattern (hydrofluoric acid + silane) did not bring any significant benefits in terms of bond strength. Moreover, most specimens showed adhesive/ mixed failures [24,30], which is accordance with the study of Román-Rodriguez et al. [29] and Murillo-Gómez [20]. The superior bonding performance of resin luting materials to lithium disilicate should be mainly attributed to better chemical interaction between the hydrophobic resin and the ceramic surface, rather than the mechanical interlocking to the rough surface [13].

Donmez et al. [17] reported significantly lower SBS values for LDC treated with MEP than those prepared with 5% hydrofluoric acid. According to the results of Cardenas et al. [15], MEP causes a partial dissolution of the glass-ceramic matrix, yielding a less-pronounced etching pattern in ceramic surfaces, compared to hydrofluoric acid.

Murillo-Gómez et al. [20] observed that HYB etched with 5% hydrofluoric acid for 60 s produced a more irregular surface compared to MEP treatment. In other study, the same author [34] showed that LEU, LDC and HYB specimens treated with 10% hydrofluoric acid for 60 s

presented higher mean roughness values compared to MEP etching. Both findings are in agreement with the results of Donmez et al. [17]. In what concerns about HYB, the acid etch removes part of the glass matrix and dissolves part of the polymer, creating microporosities. Moreover, the methoxy groups of silane agent bonds with the SiO_2 and integrated polymer components of the PICN, polymerizing with the methacrylate groups of the resin available in the matrix. [13]

Despite some studies mention that the use of MEP is similar to ceramics' etching with hydrofluoric acid and silane [22,30,35], studies are still scarce about the influence of this new protocol on the adhesive strength of ceramics, a fundamental factor for the success of an adhesive rehabilitation.

3. MATERIALS AND METHODS

3.1. Materials

A leucite-reinforced glass-ceramic (LEU) – IPS Empress® CAD (Ivoclar Vivadent, Schaan, Liechtenstein), a feldspathic ceramic (FLD) – Vitablocs® Mark II (VITA Zahnfabrik, Bad Säckingen, Germany), a lithium disilicate ceramic (LDC) – Cameo© (Aidite Technology Co., Qinhuangdao, China), a resin nanoceramic (RNCs) – GC Cerasmart™ (GC Dental Products Europe, Leuven, Belgium) and a composite resin - Grandio® blocs (VOCO, Cuxhaven, Germany) were used (Table 1).

Table 1 – CAD/CAM blocs studied, manufacturers, lot and expiration date, chemical composition.

Material (Manufacturer)	**Shade/Lot #**	**Composition**
IPS® Empress CAD (Ivoclar Vivadent AG, Schaan, Liechtenstein)	A1 LT #Z02F9W	Leucite crystal (35–45%vol)
Vitablocs Mark II (Vita Zahnfabrik, Bad Säckingen, Germany)	A1C #39230	Silicon dioxide (56-54wt%), aluminium oxide, sodium oxide, potassium oxide, calcium oxide, titanium dioxide
Cameo (Aidite Technology Co., Qinhuangdao, China)	A1 HT #28388300	Ytrium oxide (9.28wt%), aluminium oxide, silicon dioxide, iron oxide and cobalt tetraoxide
GC Cerasmart™ (GC Dental Products Europe, Leuven, Belgium)	A3 LT #008512	BisMEPP, UDMA, DMA, silica (71wt%), barium glass nanoparticles
Grandio®blocs (VOCO, Cuxhaven, Germany)	A3 LT #2151075; #2140321	Barium aluminium borosilicate glass, silicon dioxide, polymethacrylate, stabilisers, pigments

3.2. Specimen selection and preparations

Twenty-one plates (14x12x3 ± 0.5 mm) of each ceramic and three hundred thirty-six sticks (3x3x3±0.2 mm) of composite resin and were cut from CAD-CAM blocks using a precision cut-off machine and a diamond saw (Discoplan-TS, Struers, USA), under constant irrigation.

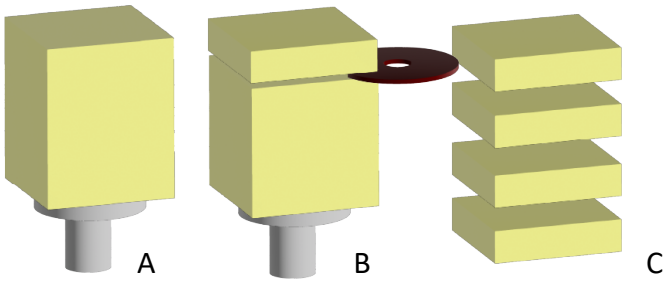


Fig. 1 – Schematic illustrating the division of each CAD/CAM ceramic bloc.
A – Initial sample; B – Division with a diamond saw; C – Divided sample

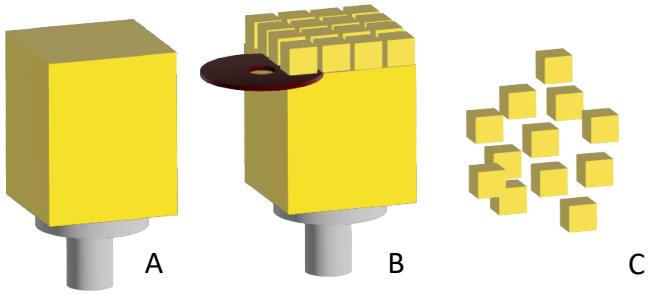


Fig. 2 – Schematic illustrating the division of each CAD/CAM resin composite bloc.
A – Initial sample; B – Division with a diamond saw; C – Divided sample

All specimens were polished in LaboPol-5 equipment (Struers, Ballerup, Denmark) using several SiC abrasive papers with 600 and 1000 grit, with a continuous passage of water, until minimal visible imperfections were reached and ultrasonically cleaned in distilled water for 5 minutes. Also, each specimen was measured with a thickness gauge (Mitutoyo Digital Caliper, Japan) and the adhesive interface area was calculated.

After that, each ceramic bloc was positioned in a polytetrafluorethylene tube filled with a self-polymerizing acrylic resin (Vertex™ – LOT WX431P03/ Exp 2027-04, Vertex-Dental, Soesterberg, Netherlands), until the full length of the bloc was embedded in the resin, obtaining cylinders to facilitate subsequent procedures.

The ceramic specimens were divided in three groups, according to the surface preparation: Hydrofluoric acid + Silane / Sandblasting + Silane (CP); Monobond® Etch&Prime (MEP); Sandblasting + Monobond Etch&Prime (SDBM) (Table 2).

Table 2 – Group distribution, according to surface treatment and type of ceramic.

Protocols / Ceramics	CP	MEP	SDBM
Lithium dissilicate	LDC_CP	LDC_MEP	LDC_ SDBM
Leucite	LEU_CP	LEU_MEP	LEU_ SDBM
Feldspathic	FLD_CP	FLD_MEP	FLD_ SDBM
Hybrid ceramic	HYB_CP	HYB_MEP	HYB_ SDBM

Then, all specimens were then divided in to two groups: one group not submitted to TC (n=5) and one test group (n=2) submitted to TC.

3.3. Experimental protocol

A single operator carried out all specimen preparation, as well as all cementation procedures.

Resulting resin sticks were analyzed under a stereomicroscope (Leica EZ4 HD, Switzerland) and all samples with defects were excluded.

Every ceramic was prepared with a single cementation protocol, according to the group they were placed in.

The adhesive procedures were performed to the ceramic surfaces in a controlled environment (23ºC; 50 ± 3%RH), with the following methodology:

Protocol 1 – Conventional protocol (CP)

Hydrofluoric acid (5%) was applied during the recommended time for each material (LDC: 20s, LEU: 60s) and hydrofluoric acid (10%) was applied to FLD during 90s; rinsed with water for an equal amount of time until etchant has been completely removed. 37,5% phosphoric acid was applied for 15 seconds and rinsed with water until etchant has been completely removed. Ultrasonic bath with still water was performed for 5min. Silane was applied to surface and remains for 60 s. Air stream was used for 10 s.

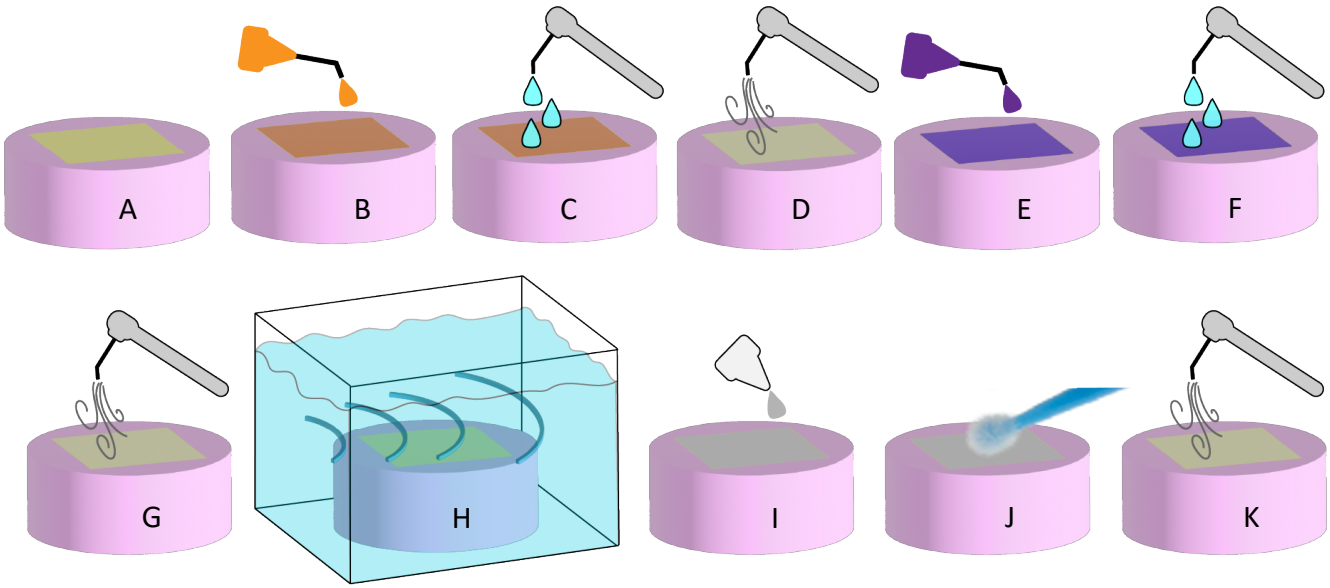


Fig. 3 – Schematic illustrating the successive steps of surface preparation with CP.
A – Initial sample; B – Hydrofluoric acid application; C – Rinse with water; D – Air stream; E – Phosphoric acid application; F – Rinse with water G – Air stream; H – Ultrasonic bath; I – Silane application; J – Active application; K – Air stream

For HYB, sandblasting with 50 µm aluminum oxide 2bar was performed during 10s, ultrasonic bath with still water for 5min, blow with oil-free air syringe, clean with alcohol to remove residues. Silane was applied to surface and remains for 60 s. Air stream was used for 10 s.

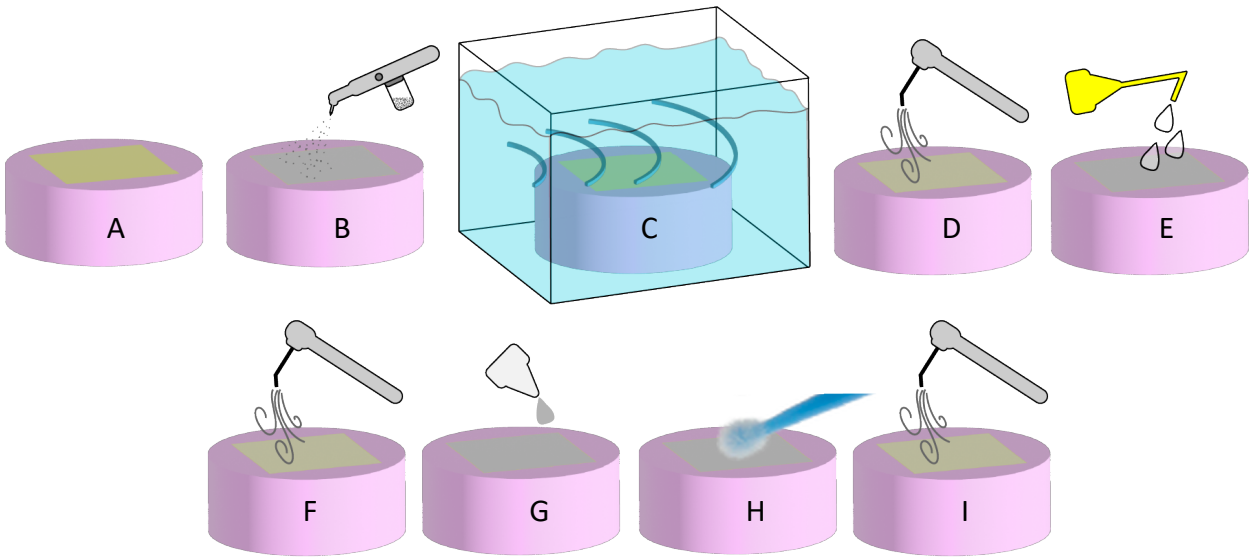


Fig. 4 – Schematic illustrating the successive steps of surface preparation with CP for Cerasmart®.
A – Initial sample; B – Sandblasting; C – Ultrasonic bath; D – Air stream; E – Rinse with alcohol; F – Air stream; G – Silane application H – Active application; I – Air stream

Protocol 2 – Monobond® Etch&Prime (MEP)

For all type of ceramics, MEP was actively scrubbed using a microbrush for 20 s and then remains on the surface for further 40 s. The primer was rinsed with water and air dried for 10 s.

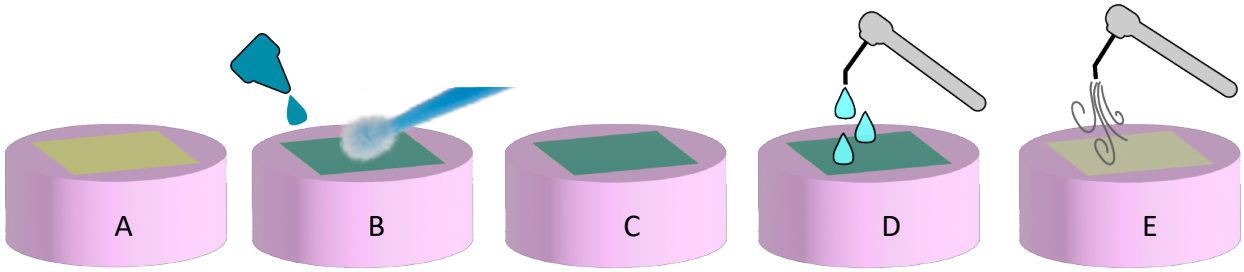


Fig. 5 – Schematic illustrating the successive steps of surface preparation with MEP.
A – Initial sample; B – MEP application; C – Waiting for reaction D – Rinse with water; E – Air stream

Protocol 3 – Sandblasting + Monobond® Etch&Prime (SDBM)

For all type of ceramics, sandblasting with 50 µm alumina 2bar was performed during 10s, followed by an ultrasonic bath with still water for 5min and blow with an oil-free air syringe. MEP was actively scrubbed using a microbrush for 20 s and then remains on the surface for further 40 s. The primer was rinsed with water until has been completely removed and air stream was used for 10 s.

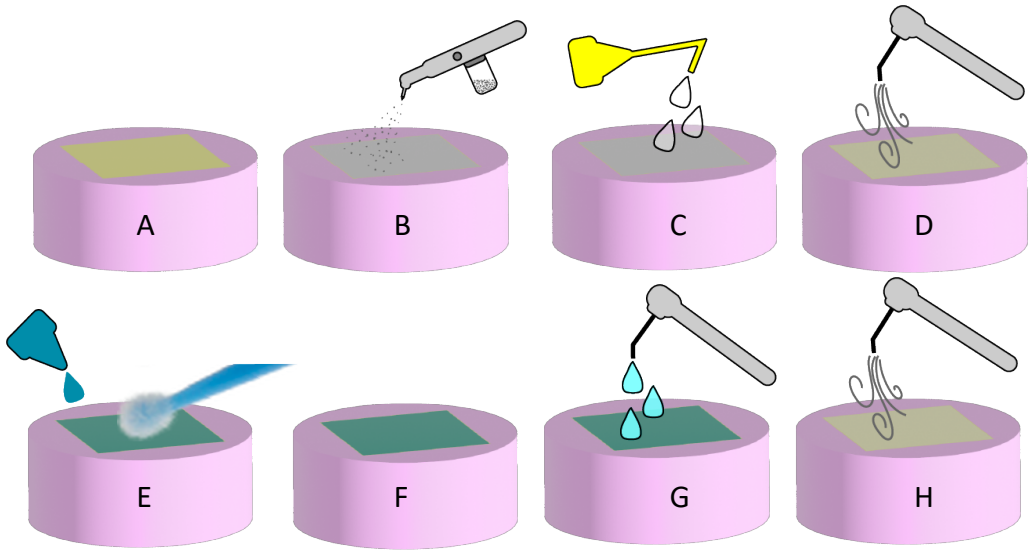


Fig. 6 – Schematic illustrating the successive steps of surface preparation with SDBM.
A – Initial sample; B – Sandblasting; C – Rinse with alcohol; D – Air stream; E – MEP application; F – Waiting for reaction G – Rinse with water; H – Air stream

For Grandio® blocs (VOCO, Cuxhaven, Germany), sandblasting with 50 µm alumina 2bar was performed during 10s, blow with oil-free air syringe during 10s. Silane was applied to surface and remains for 60 s. Air stream was used for 10s.

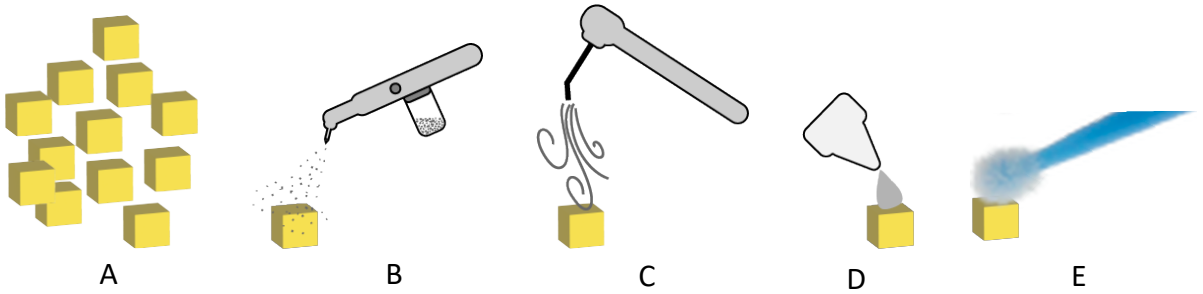


Fig. 7 – Schematic illustrating the successive steps of surface preparation of resin specimens.
A – Initial sample; B – Sandblasting; C – Air stream; D – Silane application; E – Active application

Then, each resin block (Grandio® blocs, VOCO, Cuxhaven, Germany) was cemented with thick increments of Variolink Esthetic LC (Ivoclar Vivadent, Schaan, Lichenstein), neutral color to the ceramic block, forming a square bonded area of 9mm^2.

The excess cement was removed with using a microbrush, and the luting cement was photocured using a LED light-curing unit at 1,200mW/cm^2 ± 10% (Bluephase®, Ivoclar Vivadent, Schaan, Lichenstein) for 60 s. Glycerin gel was applied on the margins and final polymerization was performed for 60 s.

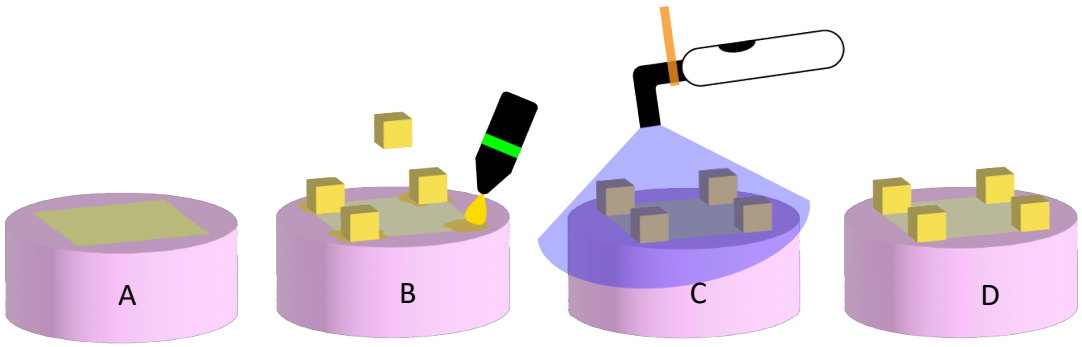


Fig. 8 – Schematic illustrating the bonding procedure.
A – Prepared ceramic surface; B – Restorative procedures; C – Photopolymerization; D – Final sample

Manufacturers, lot and expiration date and chemical composition of each material used in adhesive procedures is summarized in table 2.

Table 3 – Materials studied, manufacturers, lot and expiration date, chemical composition.

Material (Manufacturer)	Lot/Exp	Composition
Monobond® Etch&Prime (Ivoclar Vivadent, Schaan, Liechtenstein)	Z02RG0 2023-10-13	Butanol, tetrabutylammonium dihydrogen trifluoride, methacrylated phosphoric acid ester, bis(triethoxysilyl)ethane, silane methacrylate, colourant, ethanol, water
Variolink Esthetic LC (Ivoclar Vivadent, Schaan, Liechtenstein)	Y47608 2023-05-23	urethane dimethacrylate, methacrylate monomers, ytterbium trifluoride, spheroid mixed oxide, initiators, stabilizers, pigments
Silane (Ultradent, South Jordan, UT, USA)	BLZX5 2025-01-31	3-Methacryloxypropyltrimethoxysilane, ethanol, water
Hydrofluoric acid 5% (Maquira, PR, Brazil)	J21808 2023-10-30	5% Hydrofluoric acid, thickener, surfactant, water, pigment
Hydrofluoric acid 10% (Microdont, SP, Brazil)	I87596 2024-06-01	10% Hydrofluoric acid, thickener, surfactant, water, pigment
OptiBond™ Gel Etchant (Kerr, Orange, CA, USA)	8036228 2024-02-29	37.5% phosphoric acid

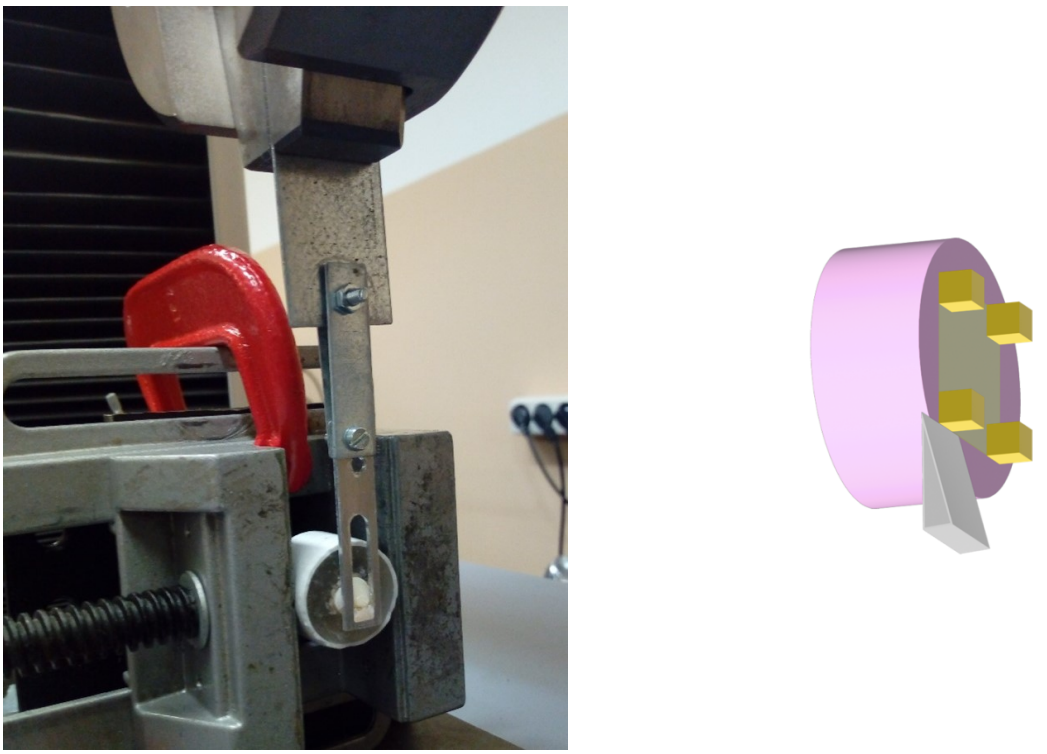
3.4. Laboratory tests

3.4.1. Surface roughness

To characterize the surface roughness of the different materials under investigation, several roughness parameters were evaluated using a Mitutoyo Surftest SJ 500P profilometer (Mitutoyo Co., Kawasaki, Japan). This device has an extendable arm and a stylus tip at its end that consists of a conical diamond with a tip radius of 5 mm and 60 degrees. The specimens are placed on the profilometer platform so that the diamond tip travels across the surface of the sample during its movement. This instrument is connected to a computer and controlled via the Formtracepak software (Mitutoyo, Aurora, IL, USA). As a means of removing shape deviation and waviness, the roughness profile (R-profile) was isolated using a cut-off wavelength λc filter. Surface roughness measurements: Ra (arithmetical mean roughness), Rq (root mean square) and Rsk (roughness skewness), were in accordance with DIN/ISO standards, with 2D roughness parameters being calculated according to EN ISO 4287. These parameters were measured 5 times for each different group, with and without thermocycling.

3.4.2. Shear bond strength testing

A shear bond strength test was performed in accordance with ISO 29022 (2013) and DIN 13990 (2017) standards. For this propose, a properly calibrated Shimadzu Autograph AG-X-5kN universal testing machine (Shimadzu, Kyoto, Japan) was used (Figure 9). It was operated at a constant rate of crosshead motion of 0.5 mm/min. At least five specimens (out of approximately ten) were used for each condition as stated in the standard. This parameter was measured for each sample, with and without thermocycling.



(a) (b)

Fig. 9 – Shear-bond strength test: (a) configuration at the Shimadzu universal testing machine; (b) schematic illustration.

3.4.3. Scanning Electron Microscopy (SEM)

Scanning Electron Microscope (Hitachi-SU-3800) and an optical microscope (LEICA DM 2500) were used. Also, EDS was performed, enabling the chemical characterization/ elemental analysis of a LDC_MEP sample.

3.4.4. Thermocycling (TC)

Specimens were subjected to 5000 cycles of thermocycling with a temperature variation between 5 °C and 55 °C for 60 s. All samples were placed in a vat and held repeatedly first in 5 °C cold water during 25 s, then out of water for 5 s and afterwards in 55 °C hot water during 25 s, until all cycles were completed.

3.5. Statistical analysis

Results were processed in a database (Microsoft Excel, Microsoft, USA), using tables, cross-tabulation, and graphs. Descriptive statistical measures such as the mean and the mean and standard deviation for the 12 evaluated groups were calculated.

The normality of the distribution was verified using the Kolmogorov-Smirnov and Shapiro-Wilk statistical tests. The homoscedasticity of the distribution was assessed by Levene's test.

In the absence of normality, the Kruskal-Wallis test was applied to analyze possible differences between the groups. In the presence of normality, the ANOVA test was performed using Tukey's HSD to find means that were significantly different from each other.

All statistical analysis was performed using the SPSS software (SPSS for Windows, version 28, SPSS Inc., Chicago, USA). For all tests, $\alpha=0.05$ was used, and p-values equal to or lower than 0.05 ($p \leq 0.05$) were considered statistically significant.

4. RESULTS

4.1. Roughness

The roughness results were represented by different variables considered in the study: Ra, Rq, and Rsk; for HYB (Table 4), LDC (Table 5), FLD (Table 6) and LEU (Table 7); before and after thermocycling.

Table 4 – Roughness (Ra, Rz, and Rsk) of HYB, for each surface treatment.

	TC	R_a (µm)			R_q (µm)			R_{sk} (µm)		
		R_a Average	R_a STDEV	p	R_q Average	R_q STDEV	p	R_{SK} Average	R_{SK} STDEV	p
C		1.30	0.11	---	1.63	0.16	---	-0.23	0.20	---
CP	Before	1.87	1.31	0.841	2.53	1.75	0.841	-0.99	0.45	0.056
	After	2.18	0.64		3.15	1.03		0.18	0.81	
MEP	Before	0.87	0.28	0.032	1.18	0.31	0.032	-1.31	0.5	0.421
	After	0.56	0.12		0.76	0.18		-0.86	0.53	
SDBM	Before	2.27	0.21	0.151	2.83	0.23	0.222	-0.09	0.15	0.310
	After	2.18	0.64		3.15	1.03		0.18	0.81	

Table 5 – Roughness (Ra, Rz, and Rsk) of LDC, for each surface treatment.

	TC	R_a (µm)			R_q (µm)			R_{sk} (µm)		
		R_a Average	R_a STDEV	p	R_q Average	R_q STDEV	p	R_{SK} Average	R_{SK} STDEV	p
C		2.15	0.46	---	2.72	0.65	---	-0.43	0.33	---
CP	Before	2.05	0.22	0.151	2.61	0.29	0.095	-0.60	0.29	0.421
	After	1.85	0.21		2.37	0.26		-0.78	0.20	
MEP	Before	2.16	0.37	0.008	2.80	0.49	0.008	-0.76	0.24	0.056
	After	0.77	0.13		1.05	0.2		-1.10	0.22	
SDBM	Before	2.42	0.22	0.151	3.02	0.29	0.151	-0.42	0.11	1.000
	After	2.15	0.46		2.72	0.65		-0.43	0.33	

Table 6 – Roughness (Ra, Rz, and Rsk) of FLD, for each surface treatment.

	TC	R_a (µm)			R_q (µm)			R_{sk} (µm)		
		$R_{a\ Average}$	$R_{a\ STDEV}$	p	$R_{q\ Average}$	$R_{q\ STDEV}$	p	$R_{SK\ Average}$	$R_{SK\ STDEV}$	p
C		1.37	0.05	---	1.70	0.07	---	-0.22	0.19	---
CP	Before	2.03	0.17	0.095	2.52	0.23	0.222	-0.44	0.12	1.000
	After	1.84	0.17		2.28	0.18		-0.46	0.20	
MEP	Before	0.78	0.13	0.095	1.08	0.29	0.151	-0.64	0.42	0.222
	After	0.96	0.09		1.22	0.10		-0.59	0.14	
SDBM	Before	1.05	0.25	0.016	1.35	0.32	0.032	-0.35	0.46	0.548
	After	1.53	0.24		1.92	0.30		-0.31	0.25	

Table 7 – Roughness (Ra, Rz, and Rsk) of LEU, for each surface treatment .

	TC	R_a (µm)			R_q (µm)			R_{sk} (µm)		
		$R_{a\ Average}$	$R_{a\ STDEV}$	p	$R_{q\ Average}$	$R_{q\ STDEV}$	p	$R_{SK\ Average}$	$R_{SK\ STDEV}$	p
C		1.36	0.02	---	1.70	0.03	---	-0.34	0.17	---
CP	Before	1.26	0.15	0.016	1.60	0.18	0.008	-0.40	0.39	0.310
	After	1.08	0.04		1.36	0.05		-0.53	0.16	
MEP	Before	0.77	0.08	0.421	1.03	0.11	0.548	-0.20	0.46	0.032
	After	0.91	0.23		1.18	0.33		-0.86	0.36	
SDBM	Before	1.40	0.09	0.421	1.75	0.12	0.690	-0.38	0.09	0.690
	After	1.36	0.02		1.70	0.03		-0.34	0.17	

4.1.1. Control groups

In the initial evaluation of all ceramics' surfaces (control groups), statistically significant differences were found in the evaluation of Ra and Rq (p=0.01). LDC was the only ceramic among the others, that showed statistically significant differences in Ra and Rq parameters (p<0.05), obtaining the highest values in these parameters. HYB had the lowest values for Ra and Rq. About Rsk, no statistically significant differences were found (p=0.425).

4.1.2. Initial evaluation vs. surface treatment

For LDC, statistically significant differences were only found in the evaluation of Ra, before and after surface treatments (p=0.045). Differences were found when comparing CP vs. SDBM treatment (p=0.028) and CP vs. control group (p=0.012). For the parameter Rsk, no statistically significant differences were found (p=0.089), nor for Rq (p=0.134).

For FLD, statistically significant differences were found in the evaluation of Ra and Rq, before and after surface treatments (p<0.001). For the parameter Rsk, no statistically significant differences were found (p=0.292). For Ra, differences were found when comparing CP to the other groups (p<0.001), control vs. MEP (p<0.001), and control vs. SDBM (p=0.035). For Rq, statistically significant differences appear in MEP vs. CP (p<0.001), MEP vs. control (p=0.006), SDBM vs. CP (p<0.001), and control vs. CP (p<0.001).

For LEU, statistically significant differences were found in the evaluation of Ra and Rq (p=0.001), before and after surface treatment. For the parameter Rsk, no statistically significant differences were found (p=0.569). In Ra and Rq, differences were found when comparing MEP to the other groups (p<0.005).

For HYB, statistically significant differences were found in the evaluation of Ra (p=0.024), Rq (p=0.035), and Rsk (p<0.001), before and after surface treatment. The differences were found between MEP and SDBM for all parameters (Ra: p=0.023, Rq: p=0.046, Rsk: p<0.001). In Rsk, there were also differences in MEP vs. control (p=0.001), SDBM vs. CP (p=0.006), and CP vs. control (p=0.019).

4.1.3. Before vs. after thermocycling

After thermocycling, there was a decrease in Ra and Rq values for HYB_MEP, LEU_CP and HYB_MEP. For FLD_SDBM there was an increase in Ra and Rq values. LEU_MEP had a decrease in Rsk values.

For LDC, statistically significant differences were found for MEP, before vs. after thermocycling ($p<0.05$). No statistically significant differences were found in CP and SDBM groups ($p>0.05$).

For FLD, no statistically significant differences were found in CP and MEP groups ($p>0.05$). When treated with SDBM, statistically significant differences were found in Ra ($p=0.016$) and Rq ($p=0.032$) parameters.

For LEU, there were statistically significant differences in the CP group for Ra ($p=0.016$) and Rq ($p=0.008$) and in MEP for the Rsk parameter ($p=0.032$). No statistically significant differences were found for the SDBM group, neither for Ra and Rq in the MEP group nor for Rsk in CP ($p>0.05$).

For HYB, statistically significant differences were found in MEP for Ra and Rq ($p=0.032$). There were no statistically significant differences in CP and SDBM groups ($p>0.05$), nor in MEP for Rsk ($p=0.421$).

In what concerns about roughness, SDBM produced higher mean roughness values, despite the type of ceramic.

4.2. Shear bond strength (SBS)

The shear bond strength results were represented in Fig. 10, before TC and Fig. 11, after TC. The outcomes were expressed in MPa.

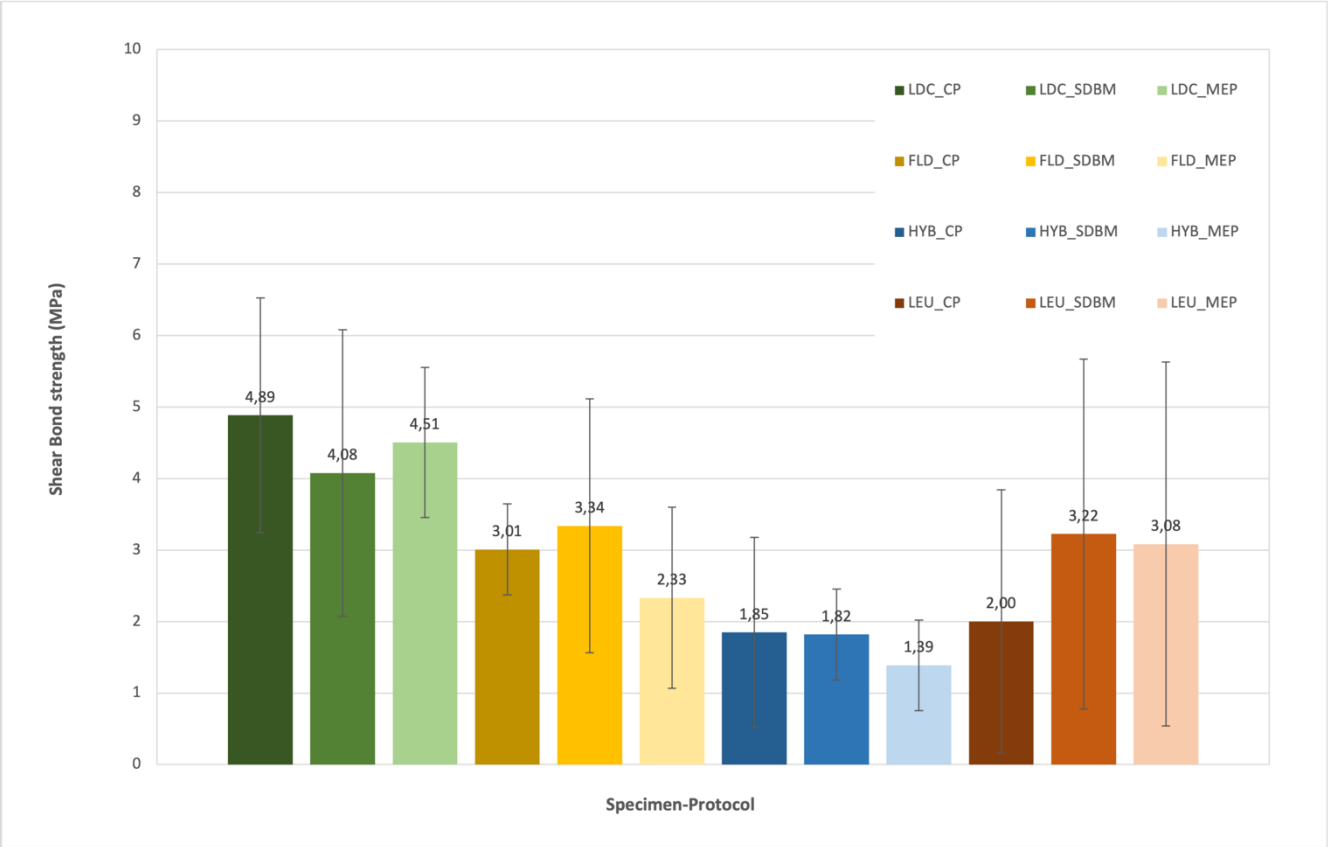


Fig. 10 – SBS measurements of four different ceramics for each surface treatment, before TC.

In the initial evaluation, before thermocycling, LDC has the highest bond strength value, for all kind of pretreatments and HYB obtained the lowest ones.

In what concerns about surface preparation, statistically significant differences were found in HYB_MEP ($p<0.001$), with the lowest SBS value among all groups; and LDC_CP ($p<0.001$) with the highest one. For SDBM, no differences were observed among all types of ceramics ($p=0.089$).

In each type of ceramic, no statistically significant differences were found, regardless of surface pretreatment: LDC: $p=0.492$; HYB: $p=0.311$; LEU: $p=0.284$; FLD: $p=0.305$.

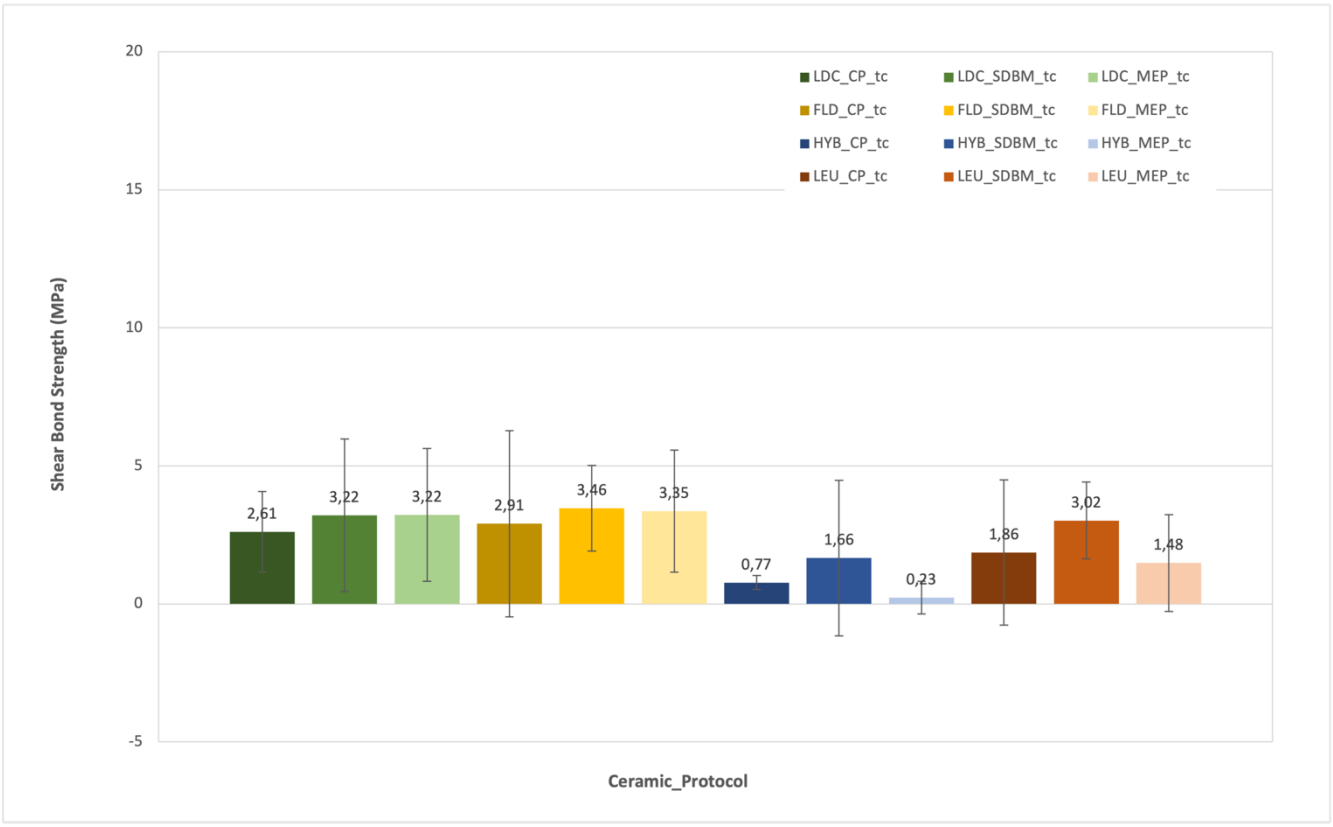


Fig. 11 – SBS measurements of four different ceramics for each surface treatment, after TC.

After thermocycling, the highest bond strength values were obtained for FLD and the lowest for HYB.

Among the different ceramics, statistically significant differences were only found in LEU_SDBM ($p=0.002$). In what concerns about pretreatment protocols differences were observed for HYB with all kinds of surface treatment, with the lowest SBS values; and in LEU_MEP. The bond strength values decreased significantly.

When comparing the same ceramic with different pretreatments, SBS values had a significant decrease in LDC_CP, LDC_MEP and HYB_MEP.

All adhesive protocols presented similar SBS values, regardless the preparation technique. Concerning the type of ceramic, LDC showed the highest SBS values, independently of the pretreatment strategy applied, although not statistically significant.

4.3. Scanning Electron Microscopy (SEM)

After performing the adhesion tests, it was possible to conclude by observing the adhesive joint of the different specimens, using optical microscopy, that the failures were entirely adhesive, and it was not possible to observe cohesive or mixed failures.

Fig. 12 shows some micrographs, obtained using scanning electron microscopy and energy dispersive spectroscopy (SEM/EDS) of the LDC_MEP specimen.

As can be seen through Fig. 12, the adhesive joint with a rectangular shape (Fig. 12 a) does not contain parts of composite resin material, leaving it integrally, as well as the ceramic surface does not have material pulls, being the separation by the adhesive joint.

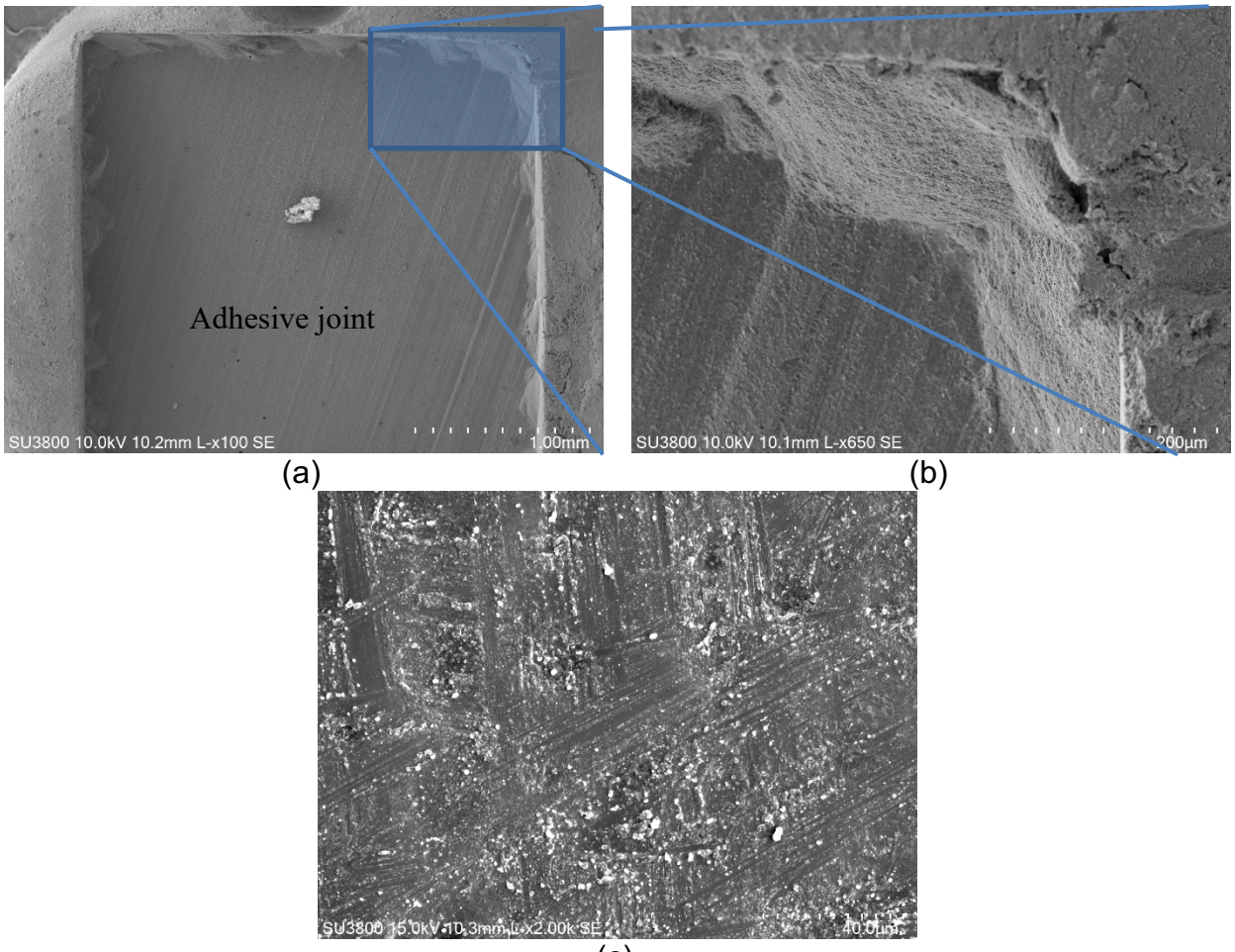


Fig. 12 – Micrographs from the adhesive joint from specimen LDC_MEP: (a) adhesive joint surface; (b) micrograph with higher magnification showing corner detail; (c) surface after MEP treatment and before being adhered.

Fig. 13 shows once again that the analysis of the adhesive joint shows that part of the adhesive material at the surface was left intact, and part was stuck to the composite resin leaving the ceramic surface exposed.

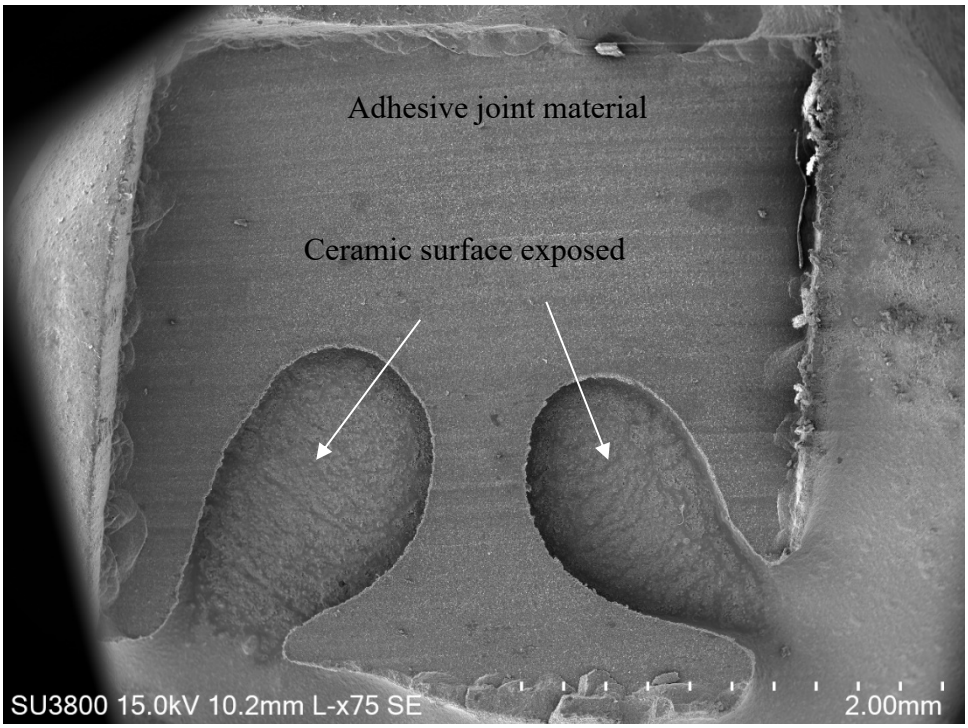


Fig. 13 – Micrograph showing the adhesive joint from specimen LDC_MEP.

5. DISCUSSION

Recent developments in adhesive procedures have changed the conventional techniques applied to the preparation of ceramics' surface. The new concepts lead to advancements on materials, due to the necessity of simplifying the protocols, while maintaining the bonding strength.[13,29,30] The main mechanism for the ceramic-resin bond is based on the morphology and chemical properties of the ceramic surface.[7]

To simulate the oral environment and temperature changes, thermocycling tests can be applied, resulting in aging and fatigue. In thermal cycling, specimens are subjected to between 1000 and 100,000 cycles between 5° and 55°C [36,37]. These cycles could generate hoop stresses at the bonding interface, caused by a change in thermal expansion coefficient of different materials. Ozcan et al. [27] showed that thermocycling could significantly decrease the bond strength between resin cement and ceramic.

The purpose of the present *in vitro* study was to evaluate the influence three different ceramic conditioning protocols for adhesive restorations: CP vs. MEP vs. SDBM; may have in bonding strength and surface characteristics, as the literature about this topic is scarce and not consensual. Also, an insight on this subject would present undoubted clinical relevance. The protocol with the additional step of sandblasting before MEP (SDBM) is an attempt to improve surface' roughness and increase micro retention.

In what concerns about shear bond strength, in this investigation, LDC showed the highest mean SBS values, regardless of the surface treatment and HYB had the lowest ones. With MEP pretreatment, significant differences were found in HYB followed by LEU. Moreover, with CP, differences were only observed in LDC. The results obtained for LDC are in accordance with El-Damanhoury et al. [13] and Siqueira et al. [24]. SDBM promoted intermediate bond strength among all surface treatments, except for FLD ceramic in which this preparation protocol had higher values.

After thermocycling, all studied materials had a decrease in their SBS values, except FLD_SDBM and FLD_MEP which had an increase in SBS values. The reason for these different results might be due to the relatively short number of cycles, thus resulting in a shorter water storage and fatigue time. Although, only significant decreases were obtained for

LDC_MEP and HYB_MEP. However, this static method has its shortcomings related to the variables of test specimens, preparation procedures, and test mechanics [38]. This static method has the potential of creating localized high stress areas due to the uneven distribution of load, which results in SBS values lower than the actual stress. [13]

The arithmetic average of the absolute heights (R_a) and the mean square root of the height of profiles [29] were selected due to their large use and permit to compare sample roughness in what concerns their esthetic aspects. Skewness (R_{sk}) is related with the surface load capability. A negative value of R_{sk} indicates the valleys in the surface. [39,40]

In the present study, SDBM promoted the highest roughness mean values (R_a) for all ceramics, except for feldspathic which had the highest values with CP . The increase in surface roughness of indirect restorative materials submitted to sandblasting pretreatment has also been reported in previous studies [10,15,33,41]. These values can be explained due to the fact LDC and LEU contains a lower volume of glassy phase and increased concentration of highly compacted ceramic, thus becoming less etchable than FLD. [34] Nevertheless, although sandblasting promotes higher values of roughness, this pretreatment causes more aggressive changes in ceramics' surface. [33,42] In the case of thin adhesive restorations of LEU and FLD, the use of this protocol may weaken the material [30]. The lowest values were obtained with MEP, which agrees with the study of Lima et al. [42]

After thermocycling, roughness parameters varied significantly among all type of ceramics. There was an increase of values on the following groups: FLD_SDBM, HYB_MEP. A decreased was observed for LDC_MEP and LEU_CP. This fact could be attributed to reduced number of thermocycles.

The null hypothesis, H_0: "There are no statistically significant differences between distinct surface' pretreatments protocols on 1) shear bond strength and 2) roughness values for the four different types of ceramic" is rejected by the findings of this study.

6. CONCLUSION

Within the limitations of the present *in vitro* study, our findings suggest:

- All ceramics present similar SBS values, except hybrid ceramic (HYB). MEP can be a valid pretreatment for adhesive restorations, in case of feldspathic (FLD) and lithium disilicate (LDC) ceramics. However, the clinical decision must rely on other aspects such as the technique's predictability and clinical reproducibility.

- Sandblasting + MEP (SDBM) produced higher mean roughness values, despite of ceramic surface type. Although, it can promote good bond strength values, it may weaken the ceramic structure.

- Concerning the type of ceramic, lithium disilicate (LDC) showed the highest values of shear bond strength independently of the bonding strategy, although not statistically significant.

- After performing the adhesion tests, it was possible to conclude the failures were entirely adhesive.

Further studies in this line of research are needed with standardized experimental protocols to establish clear relations between the evaluated parameters.

7. REFERENCES

1. Santos GC, Jr., Santos MJ, Rizkalla AS. Adhesive cementation of etchable ceramic esthetic restorations. J Can Dent Assoc. 2009;75(5):379-84.
2. Guimarães HAB, Cardoso PC, Decurcio RA, Monteiro LJE, de Almeida LN, Martins WF, et al. Simplified Surface Treatments for Ceramic Cementation: Use of Universal Adhesive and Self-Etching Ceramic Primer. Int J Biomater. 2018;2018:2598073.
3. Dejak B, Mlotkowski A. Three-dimensional finite element analysis of strength and adhesion of composite resin versus ceramic inlays in molars. J Prosthet Dent. 2008;99(2):131-40.
4. Lima RM, IAF; Campos, DES; Murillo-Gómez, F; Andrade, AKM; Duarte, RM; de Souza, GM. Effect of universal adhesives and self-etch ceramic primers on bond strength to glass ceramics: A systematic review and meta-analysis of in vitro studies. J Prosthet Dent. 2022;10:S0022-3913(22)00055-5.
5. Giordano R. Materials for chairside CAD/CAM-produced restorations. J Am Dent Assoc. 2006;137 Suppl:14S-21S.
6. Stewart GP, Jain P, Hodges J. Shear bond strength of resin cements to both ceramic and dentin. J Prosthet Dent. 2002;88(3):277-84.
7. Tian T, Tsoi JK, Matinlinna JP, Burrow MF. Aspects of bonding between resin luting cements and glass ceramic materials. Dent Mater. 2014;30(7):e147-62.
8. Giordano R, McLaren EA. Ceramics overview: classification by microstructure and processing methods. Compend Contin Educ Dent. 2010;31(9):682-4, 6, 8 passim; quiz 98, 700.
9. Shenoy AS, N. Dental ceramics: An update. J Conserv Dent. 2010;13(4):195-203.
10. Campos F, Almeida, C., Rippe, M., de Melo, R., Valandro, L., Bottino, M. Resin Bonding to a Hybrid Ceramic: Effects of Surface Treatments and Aging. Oper Dent. 2016;41(2):171-78.
11. Ceren NT, V.; Emir, F.; Akgungor, G.; Ayyildiz, S.; Sen, D. Nanoceramics and hybrid materials used in CAD/CAM systems. Aydın Dental. 2016:55-62.
12. Zhang Y, Sailer I, Lawn BR. Fatigue of dental ceramics. J Dent. 2013;41(12):1135-47.
13. El-Damanhoury HM, Gaintantzopoulou MD. Self-etching ceramic primer versus hydrofluoric acid etching: Etching efficacy and bonding performance. J Prosthodont Res. 2018;62(1):75-83.
14. Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: a review of the literature. J Prosthet Dent. 2003;89(3):268-74.

15. Cardenas AFM, Quintero-Calderon AS, Siqueira FSF, Campos VS, Wendlinger M, Pulido-Mora CA, et al. Do Different Application Modes Improve the Bonding Performance of Self-etching Ceramic Primer to Lithium Disilicate and Feldspathic Ceramics? J Adhes Dent. 2019;21(4):319-27.
16. Della Bona A, Anusavice KJ. Microstructure, composition, and etching topography of dental ceramics. Int J Prosthodont. 2002;15(2):159-67.
17. Donmez MB, Okutan Y, Yucel MT. Effect of prolonged application of single-step self-etching primer and hydrofluoric acid on the surface roughness and shear bond strength of CAD/CAM materials. Eur J Oral Sci. 2020;128(6):542-9.
18. Bertolini J. Hydrofluoric acid: a review of toxicity. J Emerg Med. 1992;10:163-68.
19. Lung CY, Matinlinna JP. Aspects of silane coupling agents and surface conditioning in dentistry: an overview. Dent Mater. 2012;28(5):467-77.
20. Murillo-Gómez F, De Goes MF. Bonding effectiveness of tooth-colored materials to resin cement provided by self-etching silane primer after short- and long-term storage. J Prosthet Dent. 2019;121(4):713.e1-.e8.
21. Sheets JL, Wilcox CW, Barkmeier WW, Nunn ME. The effect of phosphoric acid pre-etching and thermocycling on self-etching adhesive enamel bonding. J Prosthet Dent. 2012;107(2):102-8.
22. Colombo LDA, Murillo-Gómez F, De Goes MF. Bond Strength of CAD/CAM Restorative Materials Treated with Different Surface Etching Protocols. J Adhes Dent. 2019;21(4):307-17.
23. Meng X YK, Taira Y, Kamada K, Luo X. Effect of siloxane quantity and ph of silane coupling agents and contact angle of resin bonding agent on bond durability of resin cements to machinable ceramic. J Adhes Dent. 2011;13:71-8.
24. Siqueira FSF, Campos VS, Wendlinger M, Muso RAC, Gomes JC, Reis A, et al. Effect of Self-Etching Primer Associated to Hydrofluoric acid or Silane on Bonding to Lithium Disilicate. Braz Dent J. 2019;30(2):171-8.
25. Tolidis KG, P.; Boutsiouki, C. Intraoral Ceramic Restoration Repair Techniques: Report of 3 Cases. Balk J Stom. 2012;16:103-08.
26. Matinlinna JP, Vallittu PK. Bonding of resin composites to etchable ceramic surfaces - an insight review of the chemical aspects on surface conditioning. J Oral Rehabil. 2007;34(8):622-30.
27. Ozcan MV, P. K. Effect of surface conditioning methods on the bond strength of luting cement to ceramics. Dent Mater. 2003;19(8):725-31.
28. Ozcan M, Allahbeickaraghi A, Dündar M. Possible hazardous effects of hydrofluoric acid and recommendations for treatment approach: a review. Clin Oral Investig. 2012;16(1):15-23.
29. Roman-Rodriguez JL, Perez-Barquero JA, Gonzalez-Angulo E, Fons-Font A, Bustos-Salvador JL. Bonding to silicate ceramics: Conventional technique compared with a simplified technique. J Clin Exp Dent. 2017;9(3):e384-e6.

30. Siqueira FS, Alessi RS, Cardenas AF, Kose C, Souza Pinto SC, Bandeca MC, et al. New Single-bottle Ceramic Primer: 6-month Case Report and Laboratory Performance. J Contemp Dent Pract. 2016;17(12):1033-9.
31. Wille S, Lehmann F, Kern M. Durability of Resin Bonding to Lithium Disilicate and Zirconia Ceramic using a Self-etching Primer. J Adhes Dent. 2017;19(6):491-6.
32. Volkel TB, E. Monobond Etch and Prime. Ivoclar Vivadent. Scientific Documentation. 2015.
33. Strasser T, Preis V, Behr M, Rosentritt M. Roughness, surface energy, and superficial damages of CAD/CAM materials after surface treatment. Clin Oral Investig. 2018;22(8):2787-97.
34. Murillo-Gómez F, Palma-Dibb RG, De Goes MF. Effect of acid etching on tridimensional microstructure of etchable CAD/CAM materials. Dent Mater. 2018;34(6):944-55.
35. Lyann SK, Takagaki T, Nikaido T, Uo M, Ikeda M, Sadr A, et al. Effect of Different Surface Treatments on the Tensile Bond Strength to Lithium Disilicate Glass Ceramics. J Adhes Dent. 2018;20(3):261-8.
36. Meng X, Yoshida K, Taira Y, Kamada K, Luo X. Effect of siloxane quantity and ph of silane coupling agents and contact angle of resin bonding agent on bond durability of resin cements to machinable ceramic. J Adhes Dent. 2011;13(1):71-8.
37. Nagai T, Kawamoto Y, Kakehashi Y, Matsumura H. Adhesive bonding of a lithium disilicate ceramic material with resin-based luting agents. J Oral Rehabil. 2005;32(8):598-605.
38. Sirisha K, Rambabu T, Shankar YR, Ravikumar P. Validity of bond strength tests: A critical review: Part I. J Conserv Dent. 2014;17(4):305-11.
39. Neto J. Metrologia e Controle Dimensional. 1st ed ed: Elsevier: Amsterdam, The Netherlands; 2012.
40. Gadelmawla ESK, M. M.; Maksoud, T. M. A.; Elewa, I. M.; Soliman, H. H. Roughness Parameters. J Mater Process Tech. 2002;123(1):133-45.
41. Spitznagel FA, Horvath, S.D., Guess, P.C., Blatz, M.B. Resin bond to indi- rect composite and new ceramic/polymer materials: a review of the literature. J Esthet Restor Dent. 2014;26:382-93.
42. Lima CM, Silva NRD, Martins JD, Miranda JS, Tanaka R, Souza R, et al. Effect of different surface treatments on the biaxial flexure strength, Weibull characteristics, roughness, and surface topography of bonded CAD/CAM silica-based ceramics. Dent Mater. 2021;37(3):e151-e61.

8. ATTACHMENTS

8.1. Author's Statement

Ex.mo Senhor
Professor Doutor Paulo Rui Galrão Ribeiro de Melo
Diretor da Faculdade de Medicina Dentária
da Universidade do Porto
CC/ Ex.mo Senhor
Professor Doutor João Carlos Antunes Sampaio Fernandes
Diretor do Mestrado em Reabilitação Oral
da Faculdade de Medicina Dentária da Universidade do Porto

Porto, 29 de Julho de 2022.

Assunto: Pedido de Admissão a Provas Públicas do(a) Estudante do Mestrado em Reabilitação Oral, Elsa Cristina Reis Carneiro.

Elsa Cristina Reis Carneiro, estudante do Mestrado em Reabilitação Oral, solicita a V.ª Ex.ª admissão a provas públicas de mestrado, com a dissertação intitulada "Ceramic conditioning protocols for adhesive restorations: a comparative study", sob a orientação do Professor Doutor Paulo Júlio Andrade de Almeida, Professor Associado Convidado da Faculdade de Medicina Dentária da Universidade do Porto, e sob a coorientação do Doutor Luís Miguel Cardoso Vilhena Pereira da Silva, Investigador Auxiliar do Departamento de Engenharia Mecânica da Faculdade de Ciências e Tecnologia da Universidade de Coimbra.

Para o efeito, junta em anexo:
- 1 Exemplar da dissertação em formato digital (CD ou Pen Drive);
- Parecer dos orientadores (sobre a admissão a provas públicas);

Com os melhores cumprimentos,

Elsa Cristina Reis Carneiro

Elsa Cristina Reis Carneiro

8.2. Advisor's Statement

Parecer do Orientador

Na qualidade de orientador da estudante do Mestrado em Reabilitação Oral, Elsa Cristina Reis Carneiro, com a dissertação intitulada "Ceramic conditioning protocols for adhesive restorations: a comparative study", apresento o meu parecer sobre a admissibilidade a provas públicas:

1 – O trabalho de investigação foi realizado sob minha orientação, no decurso do Mestrado em Reabilitação Oral da Faculdade de Medicina Dentária da Universidade do Porto.

2 – A tese cumpre todas as normas científicas e de apresentação escrita, satisfaz os objectivos a que se propôs, apresenta uma introdução completa e atualizada no que diz respeito aos conteúdos apresentados. Os materiais e métodos estão bem definidos e descritos, os resultados corretamente apresentados e discutidos com perspicácia. A bibliografia é recente, completa e adequa-se ao tema da investigação.

3 – Por este motivo sou da opinião de que a candidata reúne as condições necessárias para se submter a provas públicas.

Faculdade de Medicina Dentária da Universidade do Porto, 29 de Julho de 2022.

O Orientador,

Assinado por: **PAULO JÚLIO ANDRADE DE ALMEIDA**
Num. de Identificação: 04443408
Data: 2022.07.28 08:10:09 +0100

CHAVE MÓVEL

Paulo Júlio Andrade de Almeida
Professor Associado Convidado

8.3. Co-Advisor's Statement

Parecer do Coorientador

Na qualidade de orientador da estudante do Mestrado em Reabilitação Oral, Elsa Cristina Reis Carneiro, com a dissertação intitulada "Ceramic conditioning protocols for adhesive restorations: a comparative study", apresento o meu parecer sobre a admissibilidade a provas públicas:

1 – O trabalho de investigação foi realizado sob minha coorientação, no decurso do Mestrado em Reabilitação Oral da Faculdade de Medicina Dentária da Universidade do Porto.

2 – A tese cumpre todas as normas científicas e de apresentação escrita, satisfaz os objectivos a que se propôs, apresenta uma introdução completa e atualizada no que diz respeito aos conteúdos apresentados. Os materiais e métodos estão bem definidos e descritos, os resultados corretamente apresentados e discutidos com perspicácia. A bibliografia é recente, completa e adequa-se ao tema da investigação.

3 – Por este motivo sou da opinião de que a candidata reúne as condições necessárias para se submter a provas públicas.

Faculdade de Ciências e Tecnologia da Universidade de Coimbra, 29 de Julho de 2022.

O Coorientador,

Luís Miguel Cardoso Vilhena Pereira da Silva

Investigador Auxiliar

8.4. Data Measurement Tables

Shear bond strength

SBS BEFORE THERMOCYCLING				
		CP	SDBM	MEP
		Shear Bond strength (MPa)	Shear Bond strength (MPa)	Shear Bond strength (MPa)
LDC	test E1	6,66	10,81	11,27
	test E2	12,78	3,21	6,00
	test E3	15,18	3,91	12,77
	test E4	11,29	11,37	13,57
	test E5	10,99	6,28	10,44
	test E6	14,01	10,33	9,96
	test E7	16,02	11,73	-
	test E8	6,74	4,09	10,49
	test E9	5,45	14,02	9,09
	test E10	10,82	15,98	7,66
	Average	10,99	9,17	10,14
	STDEV	3,70	4,50	2,36
FLD	test E1	8,35	8,32	3,84
	test E2	3,60	5,91	6,53
	test E3	6,25	3,67	2,45
	test E4	8,27	5,82	10,60
	test E5	7,42	7,62	3,66
	test E6	7,95	12,52	4,86
	test E7	6,85	4,15	8,51
	test E8	7,11	3,63	7,18
	test E9	5,78	7,52	2,82
	test E10	6,09	15,98	2,06
	Average	6,77	7,51	5,25
	STDEV	1,43	4,00	2,85
HYB	test E1	4,07	3,88	2,01
	test E2	1,66	5,44	2,72
	test E3	7,92	4,84	2,00
	test E4	6,29	4,46	2,87
	test E5	0,87	4,46	3,75
	test E6	0,00	3,40	6,06
	test E7	0,00	5,84	2,46
	test E8	0,00	4,64	0,00
	test E9	0,00	3,22	0,00
	test E10	0,00	0,75	0,00

	Average	4,16	4,09	3,13
	STDEV	2,99	1,43	1,43
LEU	test E1	1,16	6,44	6,44
	test E2	0,94	2,02	2,02
	test E3	4,52	5,23	15,43
	test E4	14,97	15,43	18,23
	test E5	1,97	18,23	9,25
	test E6	7,33	9,25	6,05
	test E7	5,01	6,05	2,68
	test E8	2,68	2,65	3,11
	test E9	2,90	3,11	4,14
	test E10	3,55	4,14	2,05
	Average	4,50	7,26	6,94
	STDEV	4,15	5,51	5,72

SBS AFTER THERMOCYCLING				
		CP	SDBM	MEP
		Shear Bond strength (MPa)	Shear Bond strength (MPa)	Shear Bond strength (MPa)
LDC	test E1	6,64	8,50	6,06
	test E2	6,46	2,88	7,38
	test E3	7,57	10,17	9,59
	test E4	4,65	9,38	9,39
	test E5	4,07	7,34	3,84
	test E6	-	5,14	-
	test E7	-	-	-
	test E8	-	-	-
	Average	5,88	7,24	7,25
	STDEV	1,46	2,76	2,40
FLD	test E1	3,85	6,79	9,99
	test E2	5,45	8,34	8,59
	test E3	10,32	9,83	4,21
	test E4	-	5,32	6,65
	test E5	-	9,06	8,29
	test E6	-	7,70	-
	test E7	-	8,34	-
	test E8	-	5,95	-
	Average	6,54	7,79	7,55
HYB	test E1	1,44	6,49	0,22
	test E2	1,91	0,86	0,13
	test E3	1,84	3,86	1,20

	test E4	-	-	-
	test E5	-	-	-
	test E6	-	-	-
	test E7	-	-	-
	test E8	-	-	-
	Average	1,73	3,74	0,52
	STDEV	0,25	2,82	0,59
LEU	test E1	6,05	7,15	4,31
	test E2	5,34	5,57	1,27
	test E3	1,66	5,85	2,93
	test E4	5,38	8,61	6,23
	test E5	0,15	-	3,23
	test E6	6,57	-	2,05
	Average	4,19	6,80	3,34
	STDEV	2,63	1,39	1,76

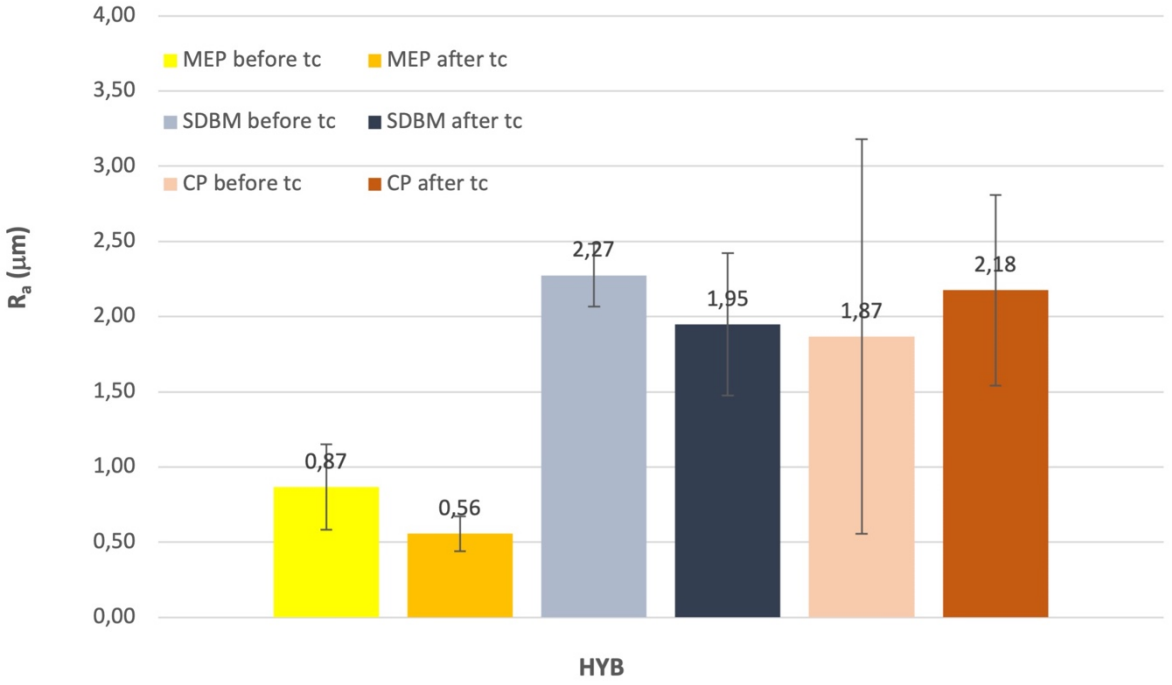
Roughness

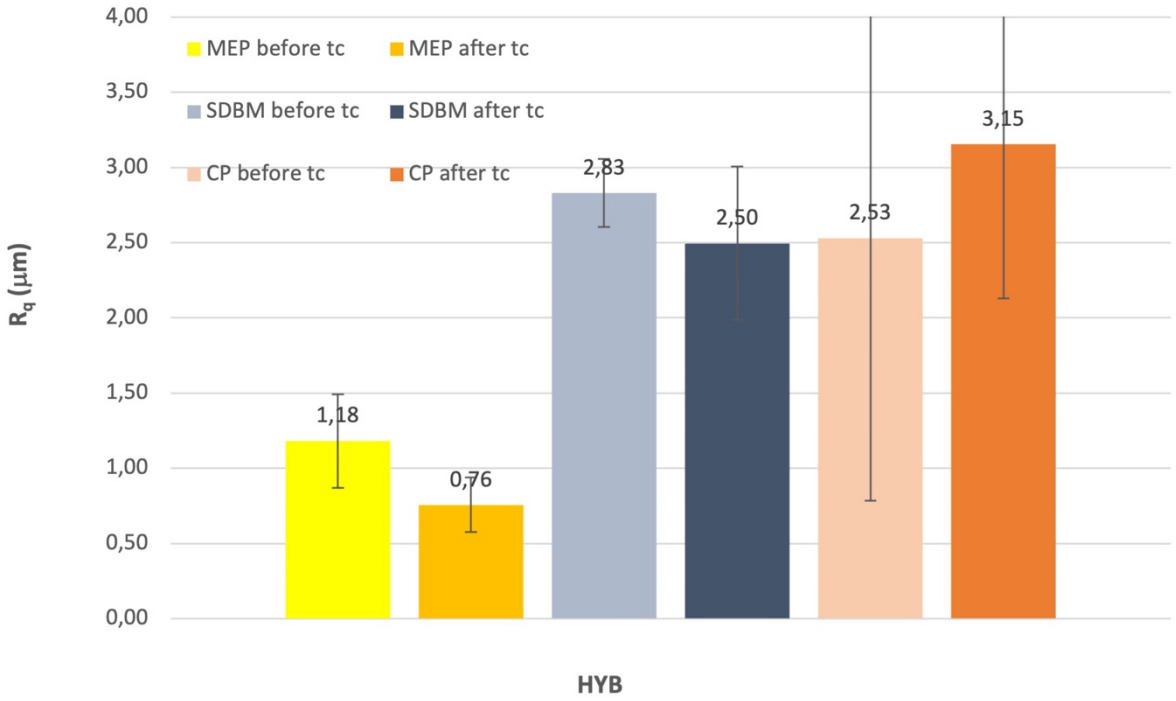
HYB - CONTROL GROUP								
	$R_{a\ Average}$	$R_{a\ STDEV}$	R_q	$R_{q\ Average}$	$R_{q\ STDEV}$	R_{SK}	$R_{SK\ Average}$	$R_{SK\ STDEV}$
µm	**µm**	**µm**	**µm**	**µm**	**µm**	**µm**	**µm**	**µm**
1,2366	1,30	0,11	1,5492	1,63	0,16	-0,1157	-0,23	0,20
1,2785			1,5578			0,0458		
1,3279			1,6606			-0,3995		
1,2011			1,4976			-0,2434		
1,4782			1,897			-0,419		

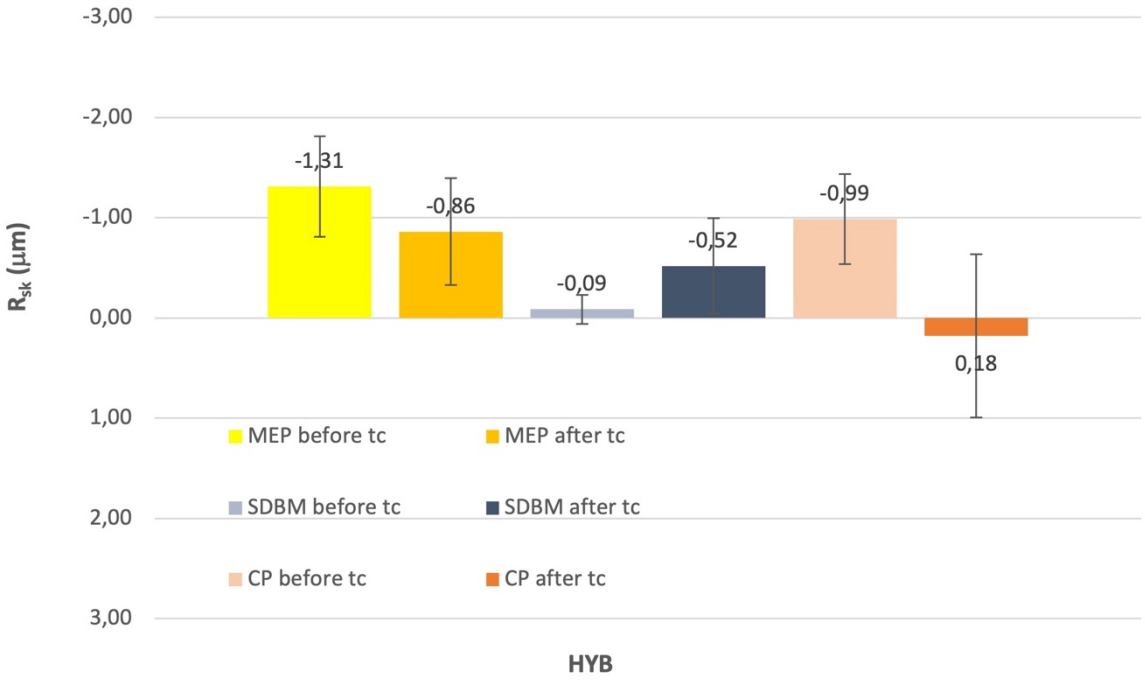
HYB – BEFORE THERMOCYCLING									
	R_a			R_q			R_{SK}		
		$R_{a\ Average}$	$R_{a\ STDEV}$	R_q	$R_{q\ Average}$	$R_{q\ STDEV}$	R_{SK}	$R_{SK\ Average}$	$R_{SK\ STDEV}$
	µm	**µm**	**µm**	**µm**	**µm**	**µm**	**µm**	**µm**	**µm**
MEP	0,8711	0,87	0,28	0,9555	1,18	0,31	-0,8963	-1,31	0,50
	1,3314			0,6999			-1,2873		
	0,5703			0,9306			-1,2728		
	0,7397			0,6552			-0,8805		

	0,823			0,5384			0,0235		
SDBS	2,3476	2,27	0,21	2,113	2,83	0,23	-0,1094	-0,09	0,15
	2,1505			2,5845			-0,8513		
	2,3745			2,3123			-0,7127		
	2,5171			3,3404			0,0722		
	1,984			2,1287			-1,0039		
CP	0,8738	1,87	1,31	1,5856	2,53	1,75	-0,5563	0,18	0,81
	2,9395			4,08			0,5806		
	3,5362			3,309			0,6279		
	0,5262			2,7879			-0,8143		
	1,4645			4,007			1,0492		

HYB – AFTER THERMOCYCLING									
	R_a			R_q			R_{SK}		
		$R_{a\ Average}$	$R_{a\ STDEV}$	R_q	$R_{q\ Average}$	$R_{q\ STDEV}$	R_{SK}	$R_{SK\ Average}$	$R_{SK\ STDEV}$
	µm	**µm**	**µm**	**µm**	**µm**	**µm**	**µm**	**µm**	**µm**
MEP	0,6368	0,56	0,12	0,9555	0,76	0,18	-0,8963	-0,86	0,53
	0,5185			0,6999			-1,2873		
	0,7093			0,9306			-1,2728		
	0,4939			0,6552			-0,8805		
	0,4213			0,5384			0,0235		
SDBS	1,5996	1,95	0,47	2,113	0,76	0,18	-0,1094	-0,52	0,47
	1,9348			2,5845			-0,8513		
	1,8611			2,3123			-0,7127		
	2,7473			3,3404			0,0722		
	1,5993			2,1287			-1,0039		
CP	1,2534	2,18	0,64	1,5856	3,15	1,03	-0,5563	0,18	0,81
	2,8238			4,08			0,5806		
	2,0264			3,309			0,6279		
	2,0451			2,7879			-0,8143		
	2,7265			4,007			1,0492		


4,00
3,50
3,00
2,50
2,00
1,50
1,00
0,50
0,00
MEP before tc
MEP after tc
SDBM before tc
SDBM after tc
CP before tc
CP after tc
0,87
0,56
2,27
1,95
1,87
2,18
Ra (µm)
HYB

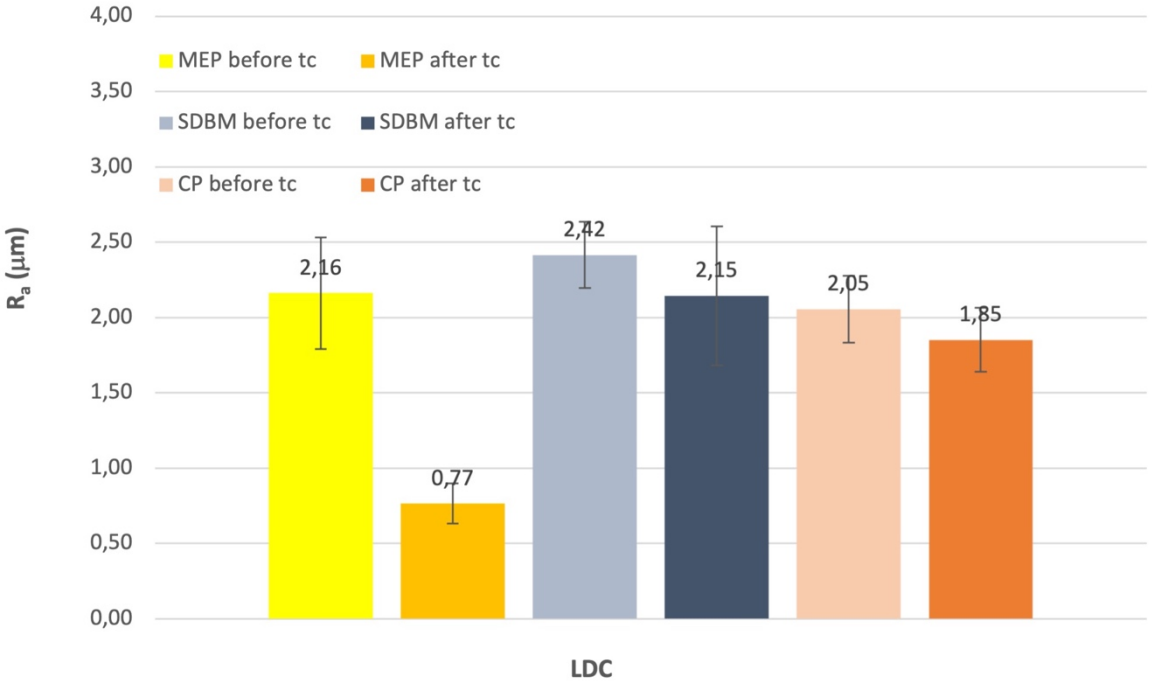

4,00
3,50
3,00
2,50
2,00
1,50
1,00
0,50
0,00
MEP before tc
MEP after tc
SDBM before tc
SDBM after tc
CP before tc
CP after tc
1,18
0,76
2,83
2,50
2,53
3,15
Rq (µm)
HYB

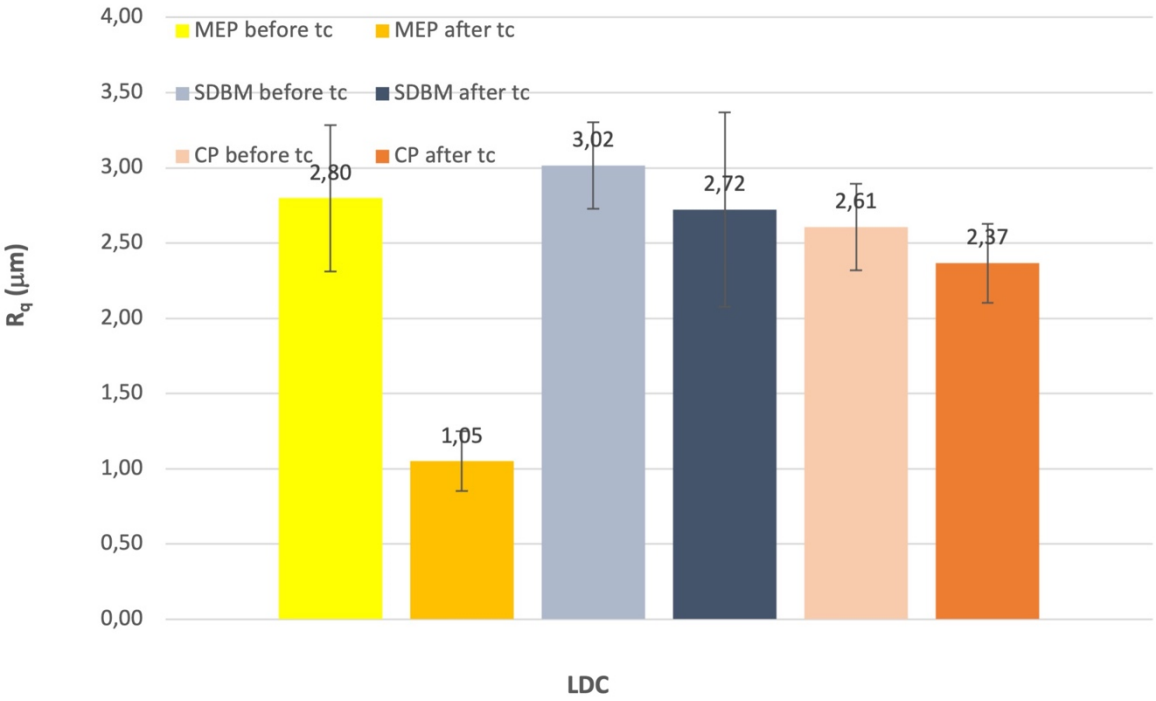


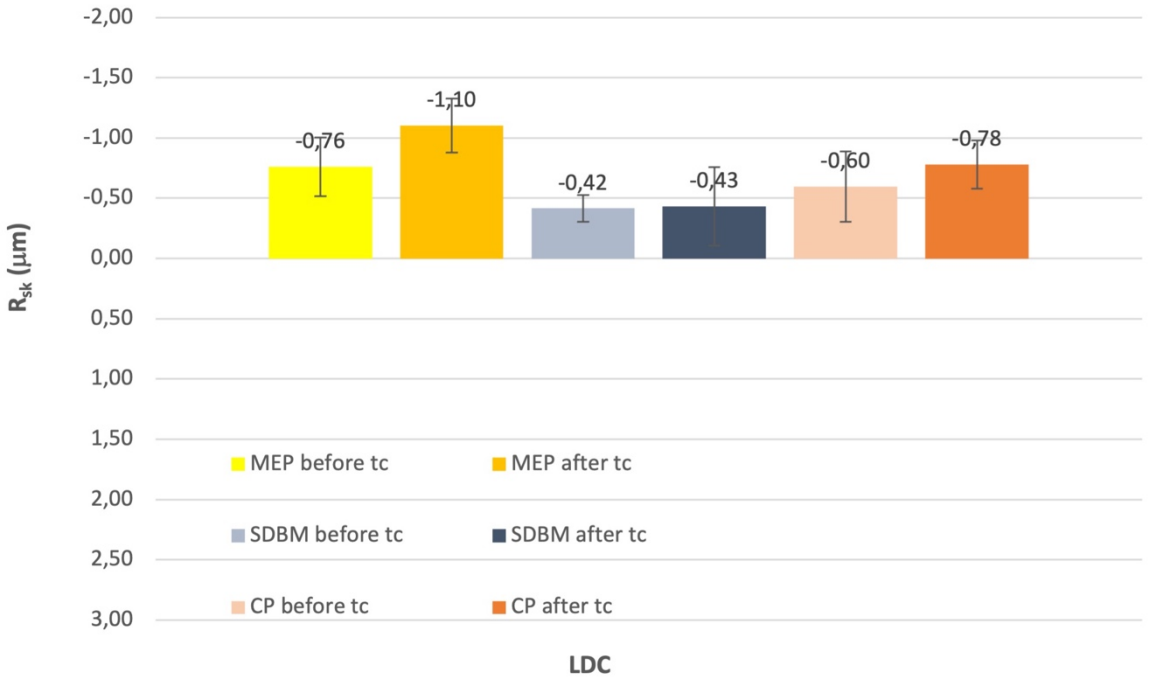
LDC – CONTROL GROUP								
	$R_{a\ Average}$	$R_{a\ STDEV}$	R_q	$R_{q\ Average}$	$R_{q\ STDEV}$	R_{SK}	$R_{SK\ Average}$	$R_{SK\ STDEV}$
µm	µm	µm	µm	µm	µm	µm	µm	µm
1,2366	1,30	0,11	1,5492	1,63	0,16	-0,1157	-0,23	0,20
1,2785			1,5578			0,0458		
1,3279			1,6606			-0,3995		
1,2011			1,4976			-0,2434		
1,4782			1,897			-0,419		

LDC– BEFORE THERMOCYCLING									
	R_a			R_q			R_{SK}		
		$R_{a\ Average}$	$R_{a\ STDEV}$	R_q	$R_{q\ Average}$	$R_{q\ STDEV}$	R_{SK}	$R_{SK\ Average}$	$R_{SK\ STDEV}$
	µm	µm	µm	µm	µm	µm	µm	µm	µm
MEP	2,5572	2,16	0,37	3,2261	0,76	0,18	-0,6588	-0,76	0,24
	2,1184			2,7002			-0,8874		
	2,203			2,7968			-0,6115		
	1,5662			2,0408			-0,5215		
	2,3614			3,2256			-1,1259		
SDBM	2,1395	2,41	0,22	2,6426	0,76	0,18	-0,2707	-0,42	0,11
	2,4108			2,9572			-0,3714		
	2,6166			3,2782			-0,4932		
	2,2644			2,8749			-0,3854		
	2,6453			3,325			-0,5576		
CP	2,3188	2,05	0,22	2,8605	2,61	0,29	-0,5969	-0,60	0,29
	2,0875			2,7551			-0,9562		
	1,7048			2,1218			-0,5422		
	2,0409			2,692			-0,7265		
	2,1226			2,6044			-0,1597		

LDC – AFTER THERMOCYCLING									
	R_a			R_q			R_{SK}		
		$R_{a\ Average}$	$R_{a\ STDEV}$	R_q	$R_{q\ Average}$	$R_{q\ STDEV}$	R_{SK}	$R_{SK\ Average}$	$R_{SK\ STDEV}$
	µm	µm	µm	µm	µm	µm	µm	µm	µm
MEP	0,7725	0,77	0,13	1,0922	1,05	0,20	-1,213	-1,10	0,22
	0,6271			0,8311			-0,9001		
	0,9819			1,3671			-1,4376		
	0,7036			0,9712			-0,9311		
	0,7406			0,992			-1,0357		
SDBM	2,954	2,15	0,46	3,8525	2,72	0,65	-0,8744	-0,43	0,33
	1,8212			2,2329			-0,4515		
	2,0813			2,6201			-0,1528		
	1,9123			2,4239			-0,6059		
	1,957			2,4857			-0,0876		
CP	2,0288	1,85	0,21	2,5221	2,37	0,26	-0,4996	-0,78	0,20
	1,8139			2,4532			-1,0397		
	2,1131			2,6739			-0,6878		
	1,6463			2,0834			-0,8655		
	1,6567			2,1024			-0,8044		


4,00
3,50
3,00
2,50
2,00
1,50
1,00
0,50
0,00
MEP before tc
MEP after tc
SDBM before tc
SDBM after tc
CP before tc
CP after tc
2,16
0,77
2,42
2,15
2,05
1,85
Ra (µm)
LDC

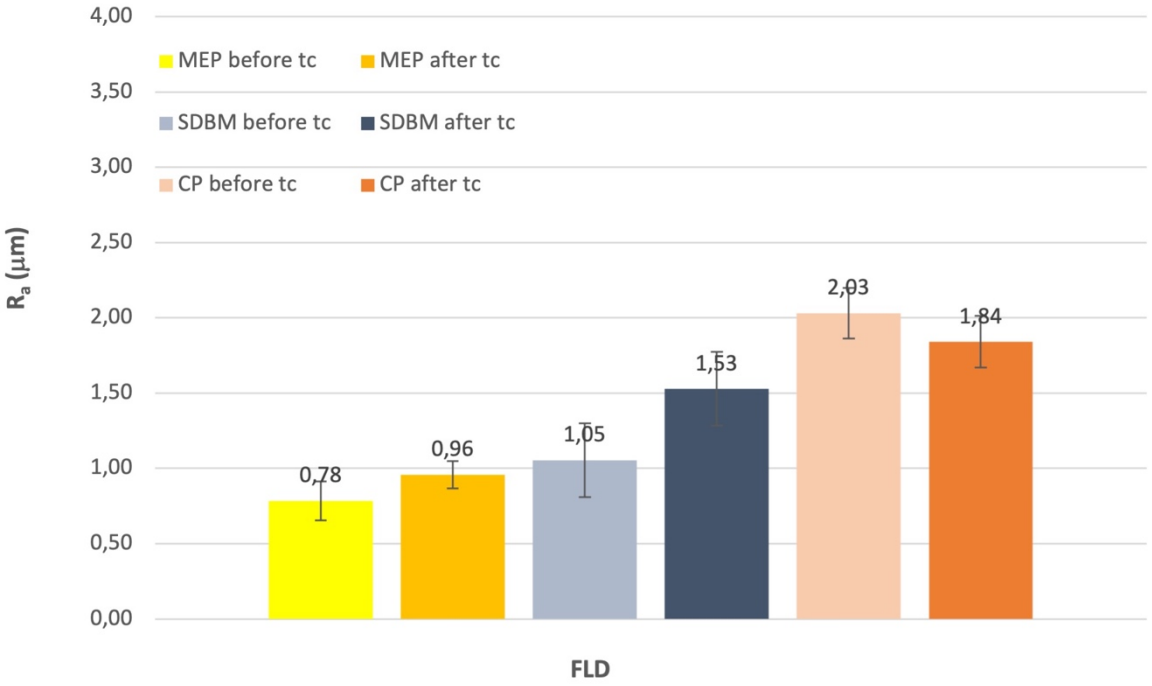

4,00
3,50
3,00
2,50
2,00
1,50
1,00
0,50
0,00
MEP before tc
MEP after tc
SDBM before tc
SDBM after tc
CP before tc
CP after tc
2,80
1,05
3,02
2,72
2,61
2,37
Rq (µm)
LDC

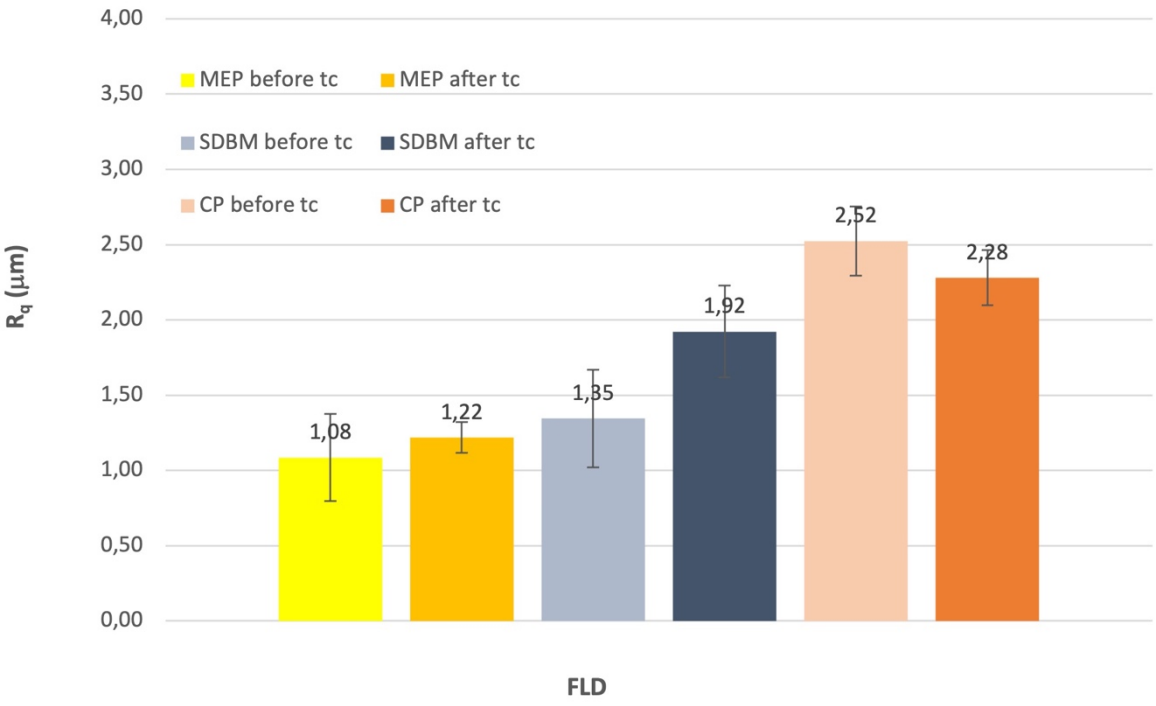


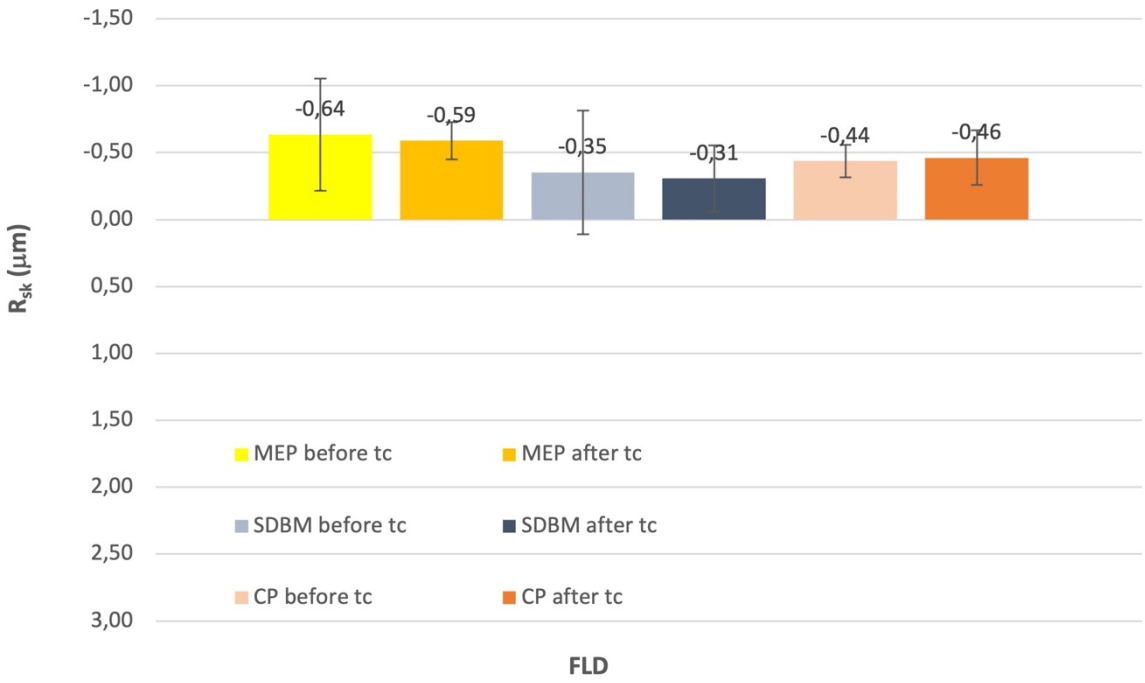
FLD – CONTROL GROUP								
	$R_{a\ Average}$	$R_{a\ STDEV}$	R_q	$R_{q\ Average}$	$R_{q\ STDEV}$	R_{SK}	$R_{SK\ Average}$	$R_{SK\ STDEV}$
µm	µm	µm	µm	µm	µm	µm	µm	µm
1,3636	1,37	0,05	1,7242	1,70	0,07	-0,4422	-0,22	0,19
1,4235			1,7722			-0,1736		
1,3971			1,7166			-0,2048		
1,3557			1,7121			-0,3576		
1,3043			1,5797			0,0579		

FLD – BEFORE THERMOCYCLING									
	R_a			R_q			R_{SK}		
		$R_{a\ Average}$	$R_{a\ STDEV}$	R_q	$R_{q\ Average}$	$R_{q\ STDEV}$	R_{SK}	$R_{SK\ Average}$	$R_{SK\ STDEV}$
	µm	µm	µm	µm	µm	µm	µm	µm	µm
MEP	0,8083	0,78	0,13	1,0564	1,08	0,29	-0,7906	-0,64	0,42
	0,7422			0,9611			-0,8621		
	0,9924			1,589			0,0996		
	0,7182			0,9509			-0,9174		
	0,6597			0,8673			-0,7055		
SDBM	0,8495	1,05	0,25	1,085	1,35	0,32	-0,6779	-0,35	0,46
	0,7326			0,9274			-0,6211		
	1,258			1,6903			0,4496		
	1,252			1,5459			-0,395		
	1,1777			1,4822			-0,5179		
CP	1,9317	2,03	0,17	2,3656	2,52	0,23	-0,3554	-0,44	0,12
	2,1349			2,657			-0,4577		
	2,2653			2,8417			-0,3041		
	1,97			2,4804			-0,6208		
	1,8512			2,275			-0,4532		

FLD – AFTER THERMOCYCLING									
	R_a			R_q			R_{SK}		
		$R_{a\ Average}$	$R_{a\ STDEV}$	R_q	$R_{q\ Average}$	$R_{q\ STDEV}$	R_{SK}	$R_{SK\ Average}$	$R_{SK\ STDEV}$
	µm	µm	µm	µm	µm	µm	µm	µm	µm
MEP	1,1102	0,96	0,09	1,3952	1,22	0,10	-0,5192	-0,59	0,14
	0,8828			1,1496			-0,7554		
	0,9027			1,1484			-0,61		
	0,9709			1,2099			-0,3922		
	0,9245			1,1891			-0,6715		
SDBM	1,4938	1,53	0,24	1,858	1,92	0,30	-0,3564	-0,31	0,25
	1,3442			1,6816			-0,7085		
	1,8493			2,3013			-0,0549		
	1,2568			1,603			-0,2003		
	1,6934			2,1713			-0,2211		
CP	1,5348	1,84	0,17	1,9551	2,28	0,18	-0,6977	-0,46	0,20
	1,9051			2,3216			-0,2971		
	1,9266			2,3464			-0,2779		
	1,9266			2,4042			-0,6656		
	1,9104			2,3724			-0,3688		


4,00
3,50
3,00
2,50
2,00
1,50
1,00
0,50
0,00
MEP before tc
MEP after tc
SDBM before tc
SDBM after tc
CP before tc
CP after tc
Ra (µm)
0,78
0,96
1,05
1,53
2,03
1,84
FLD

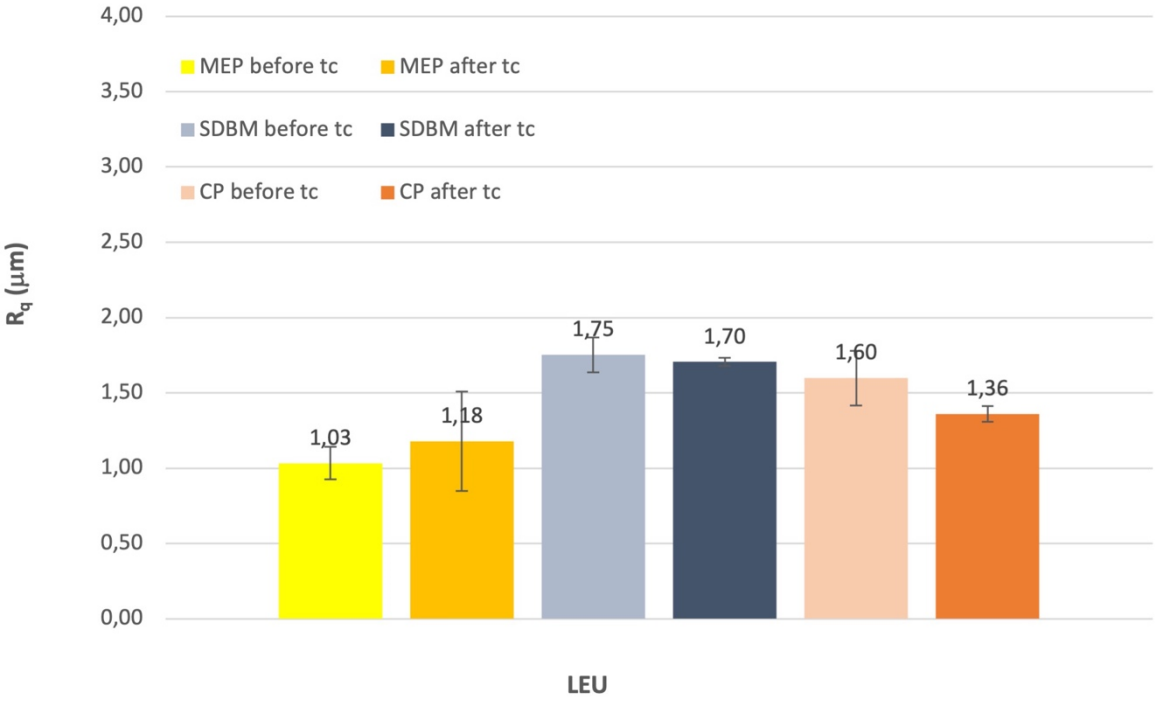

4,00
3,50
3,00
2,50
2,00
1,50
1,00
0,50
0,00
MEP before tc
MEP after tc
SDBM before tc
SDBM after tc
CP before tc
CP after tc
Rq (µm)
1,08
1,22
1,35
1,92
2,52
2,28
FLD

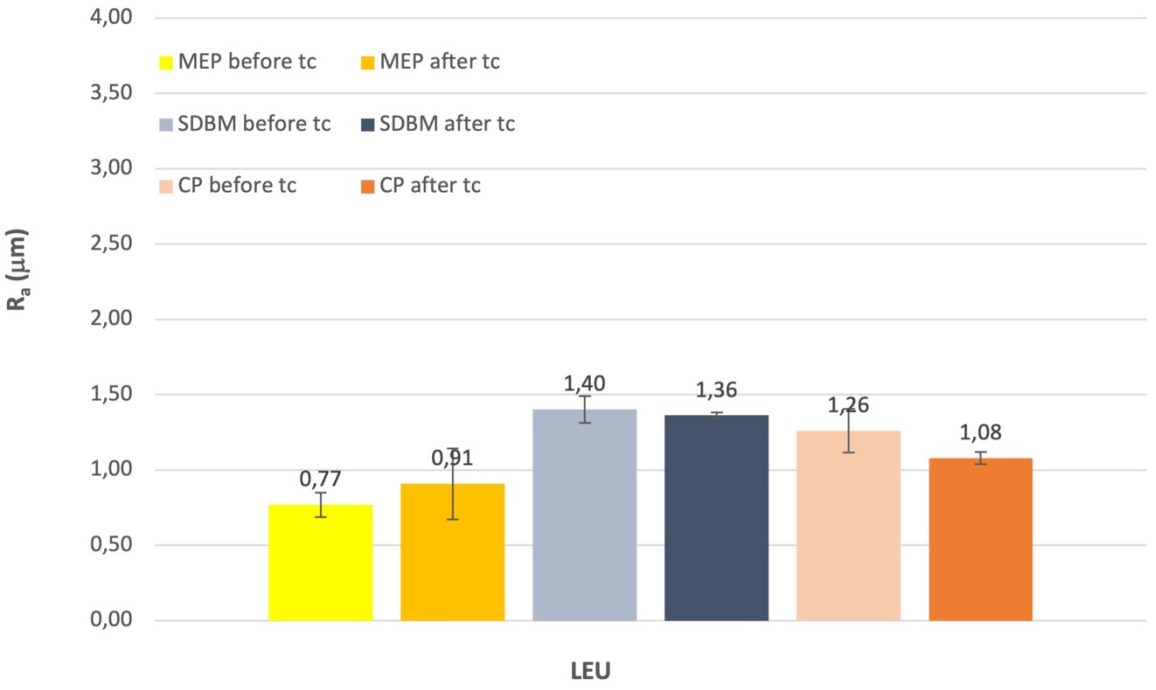


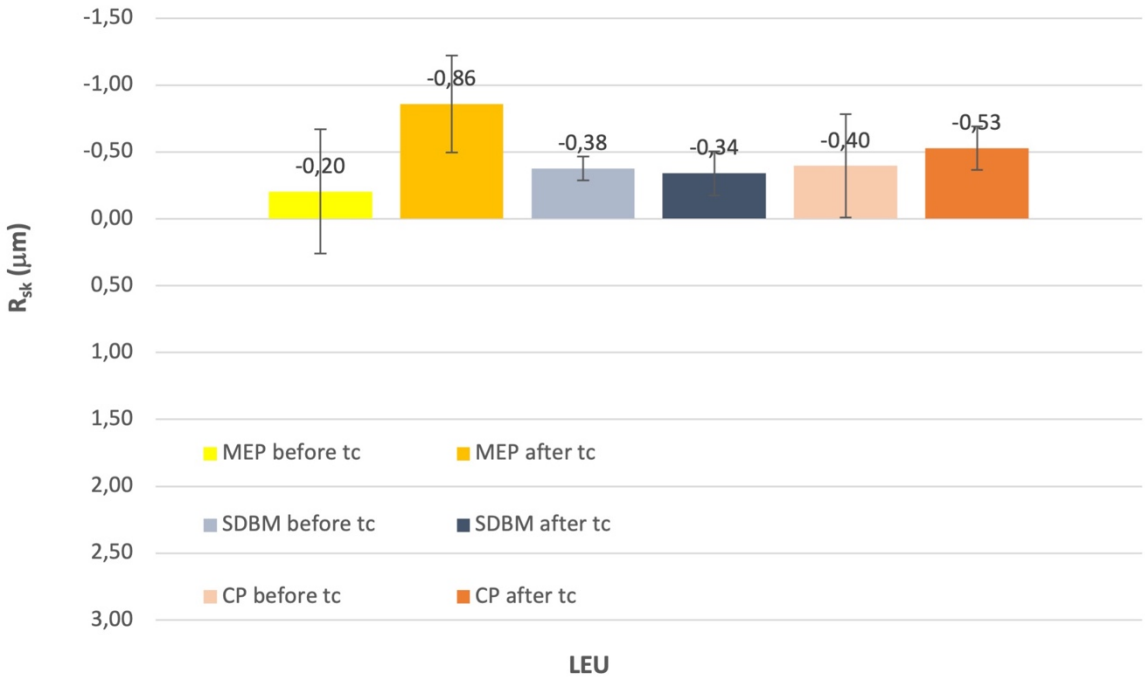
LEU – CONTROL GROUP								
	$R_{a\ Average}$	$R_{a\ STDEV}$	R_q	$R_{q\ Average}$	$R_{q\ STDEV}$	R_{SK}	$R_{SK\ Average}$	$R_{SK\ STDEV}$
µm	µm	µm	µm	µm	µm	µm	µm	µm
1,1357	1,08	0,04	1,4044	1,36	0,05	-0,5633	-0,53	0,16
1,0172			1,2787			-0,4858		
1,0786			1,4001			-0,7872		
1,0885			1,3759			-0,3712		
1,0737			1,3359			-0,4339		

LEU – BEFORE THERMOCYCLING									
	R_a			R_q			R_{SK}		
	R_a	$R_{a\ Average}$	$R_{a\ STDEV}$	R_q	$R_{q\ Average}$	$R_{q\ STDEV}$	R_{SK}	$R_{SK\ Average}$	$R_{SK\ STDEV}$
	µm	µm	µm	µm	µm	µm	µm	µm	µm
MEP	0,6809	0,77	0,08	0,8805	1,03	0,11	-0,4342	-0,20	0,46
	0,8427			1,1351			-0,5969		
	0,7097			1,0006			0,3455		
	0,8649			1,1416			-0,5839		
	0,7477			1,0065			0,2534		
SDBM	1,2844	1,40	0,09	1,6072	1,75	0,12	-0,3661	-0,38	0,09
	1,5085			1,8655			-0,2827		
	1,3651			1,6857			-0,3004		
	1,4708			1,8736			-0,4845		
	1,3755			1,7300			-0,4504		
CP	1,233	1,26	0,15	1,6379	1,60	0,18	-0,9617	-0,40	0,39
	1,1886			1,4853			-0,3894		
	1,2812			1,5852			-0,403		
	1,1067			1,4046			-0,3515		
	1,4945			1,8808			0,1298		

LEU – AFTER THERMOCYCLING									
	R_a			R_q			R_{SK}		
	R_a	$R_{a\ Average}$	$R_{a\ STDEV}$	R_q	$R_{q\ Average}$	$R_{q\ STDEV}$	R_{SK}	$R_{SK\ Average}$	$R_{SK\ STDEV}$
	µm	µm	µm	µm	µm	µm	µm	µm	µm
MEP	1,062	0,91	0,23	1,4319	1,18	0,33	-1,4421	-0,86	0,36
	1,2122			1,5923			-0,7037		
	0,8473			1,0743			-0,9032		
	0,6118			0,7729			-0,4639		
	0,8012			1,0223			-0,7775		
SDBM	1,3339	1,36	0,02	1,6633	1,70	0,03	-0,5409	-0,34	0,17
	1,3632			1,706			-0,21234		
	1,3564			1,7137			-0,3571		
	1,3685			1,7362			-0,4492		
	1,3885			1,7052			-0,1398		
CP	1,1357	1,08	0,04	1,4044	1,36	0,05	-0,5633	-0,53	0,16
	1,0172			1,2787			-0,4858		
	1,0786			1,4001			-0,7872		
	1,0885			1,3759			-0,3712		
	1,0737			1,3359			-0,4339		


4,00
3,50
3,00
2,50
2,00
1,50
1,00
0,50
0,00
MEP before tc
MEP after tc
SDBM before tc
SDBM after tc
CP before tc
CP after tc
Rq (µm)
1,03
1,18
1,75
1,70
1,60
1,36
LEU


4,00
3,50
3,00
2,50
2,00
1,50
1,00
0,50
0,00
MEP before tc
MEP after tc
SDBM before tc
SDBM after tc
CP before tc
CP after tc
Ra (µm)
0,77
0,91
1,40
1,36
1,26
1,08
LEU


-1,50
-1,00
-0,50
0,00
0,50
1,00
1,50
2,00
2,50
3,00
R_{sk} (µm)
-0,20
-0,86
-0,38
-0,34
-0,40
-0,53
MEP before tc
MEP after tc
SDBM before tc
SDBM after tc
CP before tc
CP after tc
LEU

Ceramic conditioning protocols for adhesive restorations: a comparative study

Elsa Cristina Reis Carneiro

FACULTY OF DENTAL MEDICINE, UNIVERSITY OF PORTO