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Evaluation Of Surface Characteristics For Three Milled CAD/CAM Monolithic Ceramic Restorations

Fahad Algahtani
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EVALUATION OF SURFACE CHARACTERISTICS FOR THREE MILLED
CAD/CAM MONOLITHIC CERAMIC RESTORATIONS

A Thesis presented

By

FAHAD ALGAHTANI, B.D.S

A Thesis Presented to the Faculty of the College of Dental Medicine of Nova
Southeastern University in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE IN DENTISTRY

May 2018

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CAD/CAM MONOLITHIC CERAMIC RESTORATIONS.

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FAHAD ALGAHTANI, B.D.S

A thesis submitted to the College of Dental Medicine of Nova Southeastern
University in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

Cariology and Restorative Dentistry

College of Dental Medicine

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May 2018

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DATE SUBMITTED: May, 2018

I certify that I am the sole author of this thesis, and that any assistance I received in its preparation has been fully acknowledged and disclosed in the thesis. I have cited any sources from which I used ideas, data, or words, and labeled as quotations any directly quoted phrases or passages, as well as providing proper documentation and citations. This thesis was prepared by me, specifically for the M.S. degree and for this assignment.

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Date

DEDICATION

This thesis work is dedicated to my wife, Meaad, who has been a constant source of support, encouragement and inspiration during the challenges of life and postgraduate school. To my lovely little boy yesterday, my friend today and my son everyday Yosif. This thesis is also dedicated to my parents, Saeed and Norah Algahtani, who have always loved me unconditionally, encouraged and supported me with my education. With your wisdom, support and guidance I have been able to achieve my goals.

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ABSTRACT

EVALUATION OF SURFACE CHARACTERISTICS FOR THREE MILLED CAD/CAM MONOLITHIC CERAMIC RESTORATIONS.

DEGREE DATE: May 2018

FAHAD ALGAHTANI, B.D.S.

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Background: Dental ceramics have been chosen as the material of choice by patients and clinicians because of their aesthetics, color stability, and low thermal conductivity. Clinically, almost all restorations need some adjustments to allow adequate occlusion and contacts. However, these adjustments will create rough surfaces. Therefore, different surface treatments have been developed to improve surface smoothness and gloss of dental ceramics. **Objective:** To evaluate the average surface roughness (Ra) and gloss (GU) of three different monolithic ceramics: Lithium Disilicate (IPS e.max CAD), Leucite reinforced (IPS Empress CAD) and Feldspathic (Vitablocs Mark II) subjected to two different surface treatments (mechanical polishing vs reglazing firing procedure).

Material and methods: Ten disc-shaped samples (10-mm diameter and 2-mm thick) of each ceramic were prepared, for a total of 60 samples. Ceramics were CAD designed by E4D Technologies and milled to size specification. Then, specimens were glazed following manufacturer's recommendation and fired in a furnace. A fully adjustable device was used to hold the hand piece to have a standardized pressure. After adjustment, specimens were randomly assigned to one of the surface treatment options: mechanical finishing and polishing by Dialite LD System; or reglazing firing procedure using Enamelite Low-Fusing Ceramic Spray Glaze. A surface profiler was used to assess the surface roughness and gloss values were measured using a gloss meter. **Results:** Post-hoc tests were conducted using a Bonferroni adjustment. R Studio and R 3.2.2 was used for all statistical analysis, and significance was accepted at $p < 0.05$. Post-hoc Tukey results indicate mechanical polishing had a significantly lower Ra average than reglazing firing procedure (difference = 1.51, 95% CI:1.27,1.75]. Post-hoc Tukey results indicate reglazing firing procedure had a significantly higher GU average than mechanical polishing (difference = 15.01, 95% CI:14.04,15.96]. **Conclusion:** All tested CAD/CAM monolithic ceramics presented smoother surfaces and higher gloss at baseline than after subjected to adjustment and surface treatments.

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Chapter 1

Introduction

1.1 Dental ceramics:

1.1.1 Overview:

The word ceramic comes from the Greek word “Keramos”, which means "potter's clay". The basic component was clay that was heated to form pottery (Edward A, 2009). Once humans realized that clay could be dug up and formed into objects by first mixing with water and then firing, the industry was born. As early as 24,000 BC, animal and human figurines were made from clay and other materials, then fired in furnaces partially dug into the ground (Guire, 2014).

Ceramics are nonmetallic inorganic materials and apply to various materials, including metal oxides, carbides, nitrides, and borides, as well as combinations of these materials. Their structure is crystalline, demonstrating a regular periodic arrangement of the component atoms, and may reveal ionic or covalent bonding (Shenoy & Shenoy, 2010).

Ceramics are extremely brittle and will tragically fail after minor flexure; on the contrary, they can also be very strong. Therefore, dental ceramics are strong in compression but weak in tension (Pilathadka & Vahalova, 2007).

Dental ceramic materials have been elected as the material of choice by patients and clinicians and have been extensively used for both anterior and posterior restorations to replace damaged or lost teeth because of their aesthetics, color stability, biocompatibility, and low thermal conductivity (Al-Shammery, Bubb, Youngson, Fasbinder, & Wood, 2007; Asai, Kazama, Fukushima, & Okiji, 2010; Dalkiz, Sipahi, &

Beydemir, 2009; Karan & Toroglu, 2008; Lohbauer, Muller, & Petschelt, 2008; Nakamura, Sato, Ohtsuka, & Hojo, 2010). Recently, there have been enormous improvements in the mechanical properties and methods of fabrication of dental ceramics, resulting in dozens of products for dentists to choose from (McLean, 2001). Traditionally, ceramic restorations have been restricted to the anterior area of the dental arch until recently with the introduction of monolithic lithium dioxide and zirconia restorations (Hamza & Sherif, 2017).

1.1.2 Classification:

Ideally, a classification system for dental ceramics should be helpful in providing clinically important information about where to use the them (anterior versus posterior), type of restoration (partial versus full, short versus long-span), and how to deliverer it (traditionally versus adhesively) (Gracis, Thompson, Ferencz, Silva, & Bonfante, 2015).

Different classification systems have been suggested based on microstructure components, processing techniques and clinical indications to explain different ceramics uses and properties, and, provide the clinicians with a better understanding of ceramics (Giordano & McLaren, 2010).

1.1.2.1 Microstructural classification:

Composition category 1: Glass-based systems, amorphous glass:

They are known as feldspars. Mechanical properties are low with flexural strength ranging from 60 MPa to 70 MPa. They are mainly used for veneer materials for metal or ceramic substructures.

Composition category 2: Glass-based systems with crystalline second phase, porcelain:

This material is the most successfully documented machinable glass for the fabrication of inlays and onlays with all studies showing a less than 1% per year failure rate compared to metal-ceramic survival data (Berg & Derand, 1997; Heymann, Bayne, Sturdevant, Wilder, & Roberson, 1996; Otto, 1995; Reiss & Walther, 2000).

Subcategory 2.1 Low-to-moderate leucite-containing feldspathic glass:

The typical powder/liquid materials used for veneer core systems and are mainly used for porcelain veneers. These materials are less abrasive and have much higher flexural strengths.

Subcategory 2.2 High-leucite (approximately 50%) containing glass, glass-ceramics:

The most commonly used is the pressable ceramic Empress (Ivoclar Vivadent, Amherst, NY). A machinable version of Empress CAD (Ivoclar Vivadent, Amherst, NY) has performed well clinically when used for posterior inlays and onlays, as well as anterior veneer and crown restorations.

Subcategory 2.3 Lithium-disilicate glass-ceramics:

IPS e.max (Ivoclar Vivadent, Amherst, NY) pressable and machinable ceramics. Flexural strength is 360 MPa. The material is translucent (due to the relatively low refractive index of the lithium disilicate crystals) used for the highest esthetics.

Composition Category 3: Interpenetrating phase ceramics:

In-Ceram Spinell (Ivoclar Vivadent, Amherst, NY) (alumina and magnesia matrix) with flexural strength of 350 MPa is the most translucent and used for anterior crowns. In-Ceram Alumina (Ivoclar Vivadent, Amherst) (alumina matrix) with flexural strength of 450 MPa has a moderate translucency and is mainly used for anterior and posterior crowns.

In-Ceram Zirconia (Ivoclar Vivadent, Amherst, NY) (alumina and zirconia matrix) with flexural strength of 650 MPa has lower translucency and used primarily for three-unit posterior bridges.

Composition category 4: Polycrystalline solids:

It is a partially stabilized zirconia by the addition of small amounts of other metal oxides. It is used for high stress areas. Flexural strength ranges from 900 MPa to 1100 MPa. Another important physical property is fracture toughness (a measure of a materials ability to resist crack growth). Clinical reports on zirconia have not demonstrated a problem with the zirconia framework but the problem has been with chipping and cracking of porcelain (Raigrodski et al., 2006; Sailer et al., 2007). Zirconia may be in the form of porous or dense blocks that are milled to create the frameworks or, more recently, full contour single unit restorations.

According to the microstructural classification, glass-based systems (Category 1 and Category 2) are etchable and thus easily bondable. Crystalline-based systems (Category 3 and Category 4) are not etchable and much more difficult to bond. Categories 1 to 3 can exist in a powdered form or blocks that can be pressed or machined. As a general rule, powder/liquid systems have much lower strength than pre-manufactured blocks due to a much larger amount of bubbles and flaws.

1.1.2.2 Classification based on processing technique

1. Powder/liquid

IA. Conventional:

Typically, these materials are hand-mixed and built up by hand and vibrated to remove water and air then fired in a vacuum. Because these restorations are handmade, voids are often present.

IB. Slip casting:

The "slip" is a homogenous dispersion of ceramic powder in water, then water pH is often adjusted to create a charge on the ceramic particles, and the ceramic powder is coated with a polymer to cause the particles to be evenly suspended in the water. The original In-Ceram and some partially stabilized zirconia blocks are fabricated based on slip casting.

2. Pressable

Materials are heated to allow the material to flow under pressure into a mold formed using a conventional lost-wax technique. Alternatively, a coping may be molded on which porcelain is added to achieve the restorations final shape and shade.

3. CAD/CAM

3A. Subtractive removal of excess material to fabricate the restoration (milling):

Full-Contour:

In general, these blocks are fabricated from starting powders mixed with a binder and then pressed into a block form. Then, transferred to a furnace to remove the binder and sinter to full density

Glass/Crystal

The blocks are available as monochromatic, polychromatic with stacked shades. It has an excellent history of clinical success for inlays, onlays, and anterior and posterior crowns such as Vitablocs and Sirona CEREC Blocs (Ivoclar Vivadent, Amherst, NY).

Glass/Leucite

Empress CAD is available in monochromatic and polychromatic stacked shades. Strength properties are similar to Vitablocs.

Lithium Disilicate

The IPS e.max block is not initially fully crystallized, which improves milling time and decreases chipping risk, then the milled restoration is heat-treated (20-30 minutes) to crystallize the glass and produce the final shade and mechanical properties of the restoration. This crystallization changes the restoration from blue to a tooth shade.

Framework

Alumina: Interpenetrating phase/glass-infused:

Porous blocks of In-Ceram materials are milled to produce a framework. The blocks are then infused with a glass in different shades to produce a 100% dense material, which is then veneered with porcelain.

Alumina: Porous:

Alumina frameworks may be fabricated from porous blocks of material. The frameworks are milled from the blocks and then sintered to full density at approximately 1500°C for 4 to 6 hours.

Partially stabilized zirconia: Porous:

Zirconia frameworks milled from porous blocks are fabricated similarly to alumina blocks. The milled zirconia framework shrinks about 25% after a 4- to 6-hour cycle at approximately 1300°C to 1500°C.

Partially stabilized zirconia: "HIP" blocks:

Flexural strength values of approximately 1200 MPa to 1400 MPa. However, it requires extended milling to produce the framework.

3B. Additive electrodeposition

This approach is efficient for single units but becomes cumbersome and potentially unreliable for multiple-unit frameworks.

1.2 CAD/CAM technology:

Computer-aided design (CAD) and computer-aided manufacturing (CAM) were developed in the 1960s for utilization in the automotive and aircraft industries and then applied to dentistry a decade later (Davidowitz & Kotick, 2011).

The first CAD/CAM restoration was produced in 1983 by Dr. Duret and he demonstrated his system at the French Dental Association's international congress in November 1985 by fabricating a posterior crown restoration for his wife in less than an hour (Preston & Duret, 1997).

The use of computer-aided design (CAD) and computer aided manufacturing (CAM) has clearly increased in the past several years. CAD/CAM technology can be used in both the dental office and the dental laboratory to fabricate veneers, inlays/onlays, crowns, fixed partial dentures, and even full mouth construction(Li, Chow, & Matinlinna,

2014). CAD/CAM technology was developed to overcome three obstacles. The first obstacle was to guarantee sufficient strength of the restoration. The second obstacle was to fabricate a natural apparent restoration. The third obstacle was to make the restoration more accurate, easier, and faster to be provided to the patient on the same day (Davidowitz & Kotick, 2011).

1.3 Surface treatment:

Numerous surface systems available on the market have been used by clinicians to improve surface smoothness and gloss of dental ceramic restorations, these include polishing kits, disks, and cleaning-prophylaxis paste materials.

Also, another consideration is that the application of glaze improves surface smoothness (Schneider, Dias Frota, Passos, Santiago, & Freitas Pontes, 2013).

It has been shown that polishing kits and disks are more effective than polishing paste alone or in combination with disks (Sarıkaya & Guler, 2010).

Another study evaluated the surface roughness of different dental ceramics treated with different surface treatments, and concluded that surface smoothness could be achieved by glazing and paste methods (Yilmaz & Ozkan, 2010b).

1.4 Surface roughness and gloss:

Roughness and gloss are two essential elements for evaluating the surface properties of dental materials after finishing and polishing. In spite of a strong association between the two parameters, they are considered as two different surface properties (Covey, Barnes, Watanabe, & Johnson, 2011; Heintze, Forjanic, & Rousson, 2006; Ohara et al., 2009).

Roughness (Ra) is a high-frequency, short-wavelength component of a measured surface and refers to the fine irregularity of surfaces, measured in micrometers and it is a dimensional evaluation of the surface topography that could be described by several linear (Ra, Rq, Rz) or three-dimensional (Sa, Sq, Sz) parameters (Whitehead, Shearer, Watts, & Wilson, 1995).

Gloss (GU) is an optical phenomena that is defined as the property of a surface that involves specular reflection and is responsible for a lustrous or mirror-like appearance and it is calculated by comparing the magnitude of incident light traveling toward a surface at a 60° angle to the magnitude traveling away from the surface at an equal and opposite angle (Lawson & Burgess, 2016).

The controversy concerning the effectiveness of surface treatment to obtain an acceptable smooth ceramic surface is still being debated (A. al-Wahadni & Martin, 1998; Patterson, McLundie, Stirrups, & Taylor, 1992; Raimondo, Richardson, & Wiedner, 1990; Schneider et al., 2013).

The main controversy is that, on one hand, in order to achieve minimal bacterial retention, the average values of (Ra) should be less than $0.2 \mu\text{m}$ (Bollen, Lambrechts, & Quirynen, 1997). On the other hand, the Ra values of intact human enamel are generally between 0.45 and $0.65 \mu\text{m}$ and are also reported to be a guideline parameter (Botta, Duarte, Paulin Filho, Gheno, & Powers, 2009; Willems, Lambrechts, Braem, Vuylsteke-Wauters, & Vanherle, 1991). However, other studies have shown higher values, which ranged from 0.2 to $3 \mu\text{m}$ after different surface treatment protocols (Sarac, Sarac, Yuzbasioglu, & Bal, 2006; Scurria & Powers, 1994)

Unlike for roughness, a clinically accepted value for gloss has not been established. However, natural enamel gloss is reported to range between 40 and 52 GU (Barucci-Pfister & Gohring, 2009; Mormann et al., 2013). The factors that have been reported to affect gloss include refractive index of the material, angle of incident light, and surface topography (Jain, Platt, Moore, Spohr, & Borges, 2013).

Smoothness of dental ceramics is obtained by applying a surface glaze method which is a thin layer of colorless glass powder applied on the surface of the restoration and heated to the appropriate temperature (Brewer, Garlapo, Chipps, & Tedesco, 1990; Cook, Griswold, & Post, 1984). A smooth surface of dental ceramics is required not only for aesthetics but also for biological reasons; to reduce plaque accumulation, enhance the strength of the restoration, reduce the amount of wear of the opposing teeth and increase longevity of the restoration (Bollen et al., 1997; Williamson, Kovarik, & Mitchell, 1996).

Clinically, almost all dental restorations need some functional adjustments to allow adequate occlusion, eliminate overhanging, and improve aesthetics (Albakry, Guazzato, & Swain, 2004; Wright et al., 2004). However, these adjustments remove the natural glaze layer of the restoration, which leads to a rough surface (A. M. Al-Wahadni & Martin, 1999; Sarac et al., 2006). The presence of rough surface restorations resulting from poor surface treatment procedures can cause problems such as staining, gingival irritation, plaque accumulation and recurrent caries, thereby affecting the clinical performance of the restorations (Akar, Pekkan, Cal, Eskitascioglu, & Ozcan, 2014; Silva, Salvia, Carvalho, Silva, & Pagani, 2015).

Several studies have shown that inadequate surface treatments of dental ceramics lead to smaller resistance to cracks propagation, unsatisfactory esthetics and weariness of

the opposing teeth(Flury, Lussi, & Zimmerli, 2010; Sarikaya & Guler, 2010; Sasahara, Ribeiro Fda, Cesar, & Yoshimura, 2006).

1.5 Innovation:

One of the main advantages of using a CAD/CAM system is to deliver the restoration in a single appointment. However, time needed and quality of surface treatment after clinical adjustment is a matter of great importance for both dentist and patient. One study stated that glazing procedures would save 20 % of the clinician's time when compared with mechanical polishing (Reich, Troeltzsch, Denekas, & Wichmann, 2004).

Dental ceramic materials have been continuously developed but the most acceptable surface treatments for the new generation of dental ceramic restorations has yet to be determined. Occasionally, when the restoration is subjected to finishing and adjustments, the procedure would require a considerable delay of cementation or, more likely, a second appointment. Chairside surface treatment is a desirable option and can be performed either mechanically or chemically by furnace glazing.

There is scarce research on the surface characteristics after clinical adjustments and surface treatments for CAD/CAM monolithic ceramic restorations in both polished and glazed forms. Therefore, this study is innovative in that two different surface treatments for CAD/CAM monolithic ceramic restorations will be evaluated and compared to help dentists in determining the most acceptable surface treatment protocol, which will potentially save time and efforts for both patients and dentists, as well as prevent or at least decrease rapid wear of the opposing teeth, improve aesthetics and longevity of the restoration.

1.6 Purpose of the study:

The purpose of this study is to evaluate the surface characteristics of three types of monolithic ceramic restorations subjected to two different surface treatments (mechanical polishing vs reglazing firing procedure).

1.7 Specific aims and hypothesis:

1- To evaluate the average surface roughness (Ra) of three types of monolithic ceramic restorations subjected to two different surface treatments (mechanical polishing vs reglazing firing procedure).

2- To evaluate the gloss values (GU) of three types of monolithic ceramic restorations subjected to two different surface treatments (mechanical polishing vs reglazing firing procedure).

The first null hypothesis tested is that there will be no difference in the surface roughness (Ra) of monolithic ceramic restorations when subjected to different surface treatments.

The second null hypothesis tested is that there will be no difference in the gloss (GU) of monolithic ceramic restorations when subjected to different surface treatments.

1.8 Location of study:

Bioscience Research Center, Room 7356

Nova Southeastern University, Health Professional Division, College of Dental Medicine

3200 South University Drive

Fort Lauderdale, Florida 33328-2018

Chapter 2

Materials and Methods

2.1 Experimental design:

The study was divided into two stages based on the objectives.

Stage 1: To measure of the average surface roughness (Ra) of three types of monolithic ceramic restorations subjected to two different surface treatments.

Stage 2: To measure the gloss values (GU) of three types of monolithic ceramic restorations subjected to two different surface treatments.

2.1.1 Sample size calculation:

The sample size for the average Ra using Profilometer was determined based on a similar study conducted by Amaya-Pajares et al 2016 (Amaya-Pajares et al., 2016). This study compared Ra of different monolithic ceramics resulting from different polishing systems. The study concluded that the smoothest surface of monolithic ceramics was recorded at the baseline before any adjustment and polishing.

Accordingly, it was decided that the number for each study group will be $n= 10$ per monolithic ceramics.

2.1.2 Pilot study:

A pilot study was conducted using one sample for each research group. All equipment was calibrated and techniques were reviewed.

2.1.3 Sample preparation:

Three commercial brands of monolithic ceramic blocks (Table 1) and two surface treatments (Table 2) were selected for this study.

| CERAMIC TYPE | COMPOSITION CATEGORY | FLEXURAL STRENGTH | SHADE | BLOCK SIZE | MANUFACTURER |
|-------------------|---|-------------------|-------|------------|---|
| IPS e-max CAD | Lithium-Disilicate Glass-Ceramics | 360 ± 60 MPa | A2 | 12 LT | Ivoclar-Vivadent, Schaan, Leichtenstein, Germany |
| IPS Empress CAD | High-Leucite (Approximately 50%) Containing Glass, Glass-Ceramics | 160 MPa | A2 | 12 LT | Ivoclar-Vivadent, Schaan, Leichtenstein, Germany |
| Vitabloes Mark II | Low-to-Moderate Leucite-Containing Feldspathic Glass | 154 ± 15 MPa | A2C | 14 | VITA Zahnfabrik H. Rauter GmbH & Co. KG Spitalgasse 3, D-79713 Bad Säckingen, Germany |

Table 1:Types of monolithic ceramics, composition, flexural strength, shades, size and manufacturer

| SURFACE TREATMENT | PRODUCT NAME | MANUFACTURER |
|----------------------------|---|--|
| Mechanical polishing | Dialite LD Finishing & Polishing System. | Brasseler USA, Savannah, GA, USA |
| Reglazing firing procedure | Glazing material: Enamelite Low-Fusing Ceramic Spray Glaze. Furnace: Programat CS2 | Keystone Industries, Singen, Germany Ivoclar-Vivadent, Schaan, Leichtenstein, Germany |

Table 2: Surface treatments and manufacturers

Block shade A2 was selected for standardization purposes. Ten disc-shaped samples (10-mm diameter and 2-mm thick) of each type of ceramic were prepared in accordance with the manufacturer's instructions, for a total of 30 discs (Figure 1).



Figure 1: Disc-shaped samples (10-mm diameter and 2-mm thick) of each type of CAD/CAM Monolithic Ceramic

Vitablocs Mark II, IPS e.max CAD and IPS Empress CAD were CAD designed by E4D Technologies (Richardson, TX, USA) to the requested dimensions, and the design was created as a STereoLithography (STL) file. Disc geometry was uploaded to CEREC MC XL milling unit (DENTSPLY Sirona, York, Pennsylvania).

IPS e.max CAD, IPS Empress CAD and Vitablocs Mark II blocks were placed into the milling chamber and milled to size specification, using 50 micro grit size diamond bur. The IPS e.max CAD specimens were crystallized to achieve final strength and shade.

IPS e.max CAD and IPS Empress CAD specimens were glazed with IPS Ivocolor Glaze powder according to the manufacturer's instructions (Ivoclar-Vivadent, Schaan, Leichtenstein, Germany), while Vitablocs Mark II specimens were glazed using VITA Akzent 26 Finishing Agent VITA following the manufacturer's instructions (Zahnfabrik H. Rauter GmbH & Co. KG, Spitalgasse 3, D-79713 Bad Säckingen, Germany) (Figure 2).



Figure 2: IPS e.max CAD, IPS Empress CAD and Vitablocs Mark II blocks glazing systems

Then, the specimens were fired in a furnace (Dekema Austromat D4, Wieland Dental + Technik GmbH & Co. KG, Lindenstraße 2, 75175 Pforzheim, Germany) and the firing was scheduled as follows:

IPS e.max CAD: pre-dry 1 minute, start temperature 403 °C, firing rate 30 °C per minute, high temperature 760 °C, no vacuum and a holding time of 1 minute.

IPS Empress CAD: pre-dry 1 minute, start temperature 403 °C, firing rate 45 °C per minute, high temperature 810 °C, no vacuum and a holding time of 1 minute.

Vitablocs Mark II: pre-dry 1 minute, start temperature 500 °C, firing rate 45 °C per minute, high temperature 910 °C, no vacuum and a holding time of 1 minute.

All discs were milled and glazed by VM Lab (VM Lab Technologies Inc., Miami, FL, USA).

2.1.4 Sample distribution:

Six groups of monolithic ceramic materials were included in this study, $n= 10$ specimens per group of milled discs made of three different monolithic ceramic materials (Figure 3).

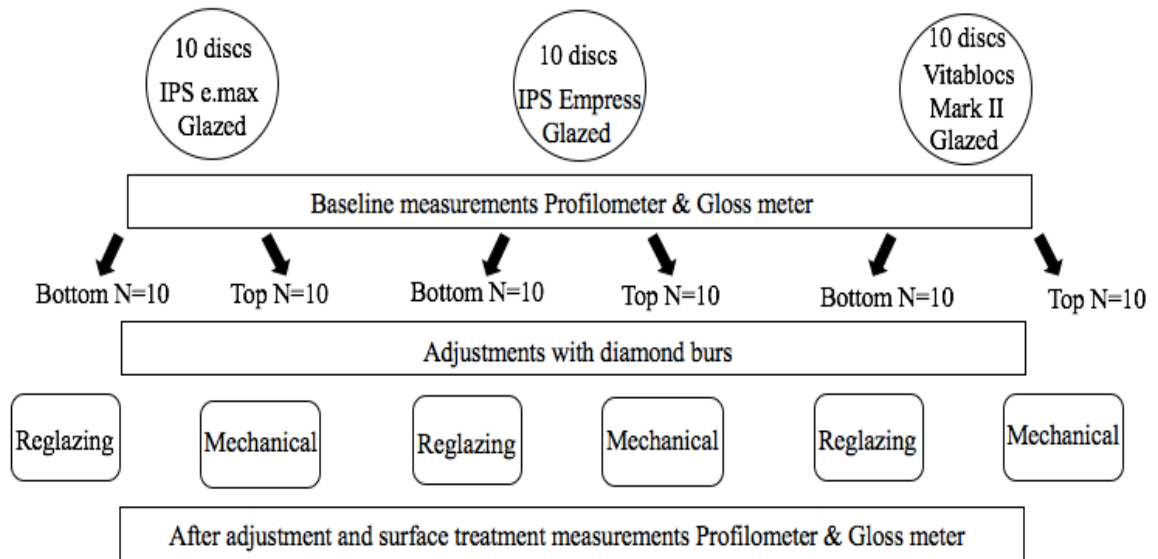


Figure 3:Flowchart of group distribution, surface roughness and gloss recordings

The top surface of each disc was marked with a black dot, while the bottom surface was marked with a red dot (Figure 4).



Figure 4:Black and red dots to differentiate between the top and the bottom sides

All specimens were randomly assigned to each one of the surface treatment composing the following group:

Group 1: Ten discs of glazed IPS e.max CAD:

The top side was treated with mechanical polishing using Dialite LD Finishing & Polishing System (Brasseler USA, Savannah, GA, USA).

Group 2: Ten discs of glazed IPS Empress CAD:

The top side was treated with mechanical polishing using Dialite LD Finishing & Polishing System (Brasseler USA, Savannah, GA, USA).

Group 3: Ten discs of glazed Vitablocs Mark II:

The top side was treated with mechanical polishing using Dialite LD Finishing & Polishing System (Brasseler USA, Savannah, GA, USA).

Group 4: Ten discs of glazed IPS e.max CAD:

Opposing surface (bottom side) was treated with reglazing firing procedure using Enamelite Nova Universal Low-Fusing Ceramic Spray Glaze (Keystone Industries, Singen, Germany).

Group 5: Ten discs of glazed IPS Empress CAD:

Opposing surface (bottom side) was treated with reglazing firing procedure using Enamelite Nova Universal Low-Fusing Ceramic Spray Glaze (Keystone Industries, Singen, Germany).

Group 6: Ten discs of glazed Vitablocs Mark II:

Opposing surface (bottom side) was treated with reglazing firing procedure using Enamelite Nova Universal Low-Fusing Ceramic Spray Glaze (Keystone Industries, Singen, Germany).

2.2 Protocol for adjustments and mechanical polishing:

A fully adjustable device (Manufactured by Mr. Arthur Zielinski for Bioscience Research Center, College of Dental Medicine, Nova Southeastern University) was used to hold the hand piece to have a standardized pressure during adjustments and polishing the specimens (Figure 5).

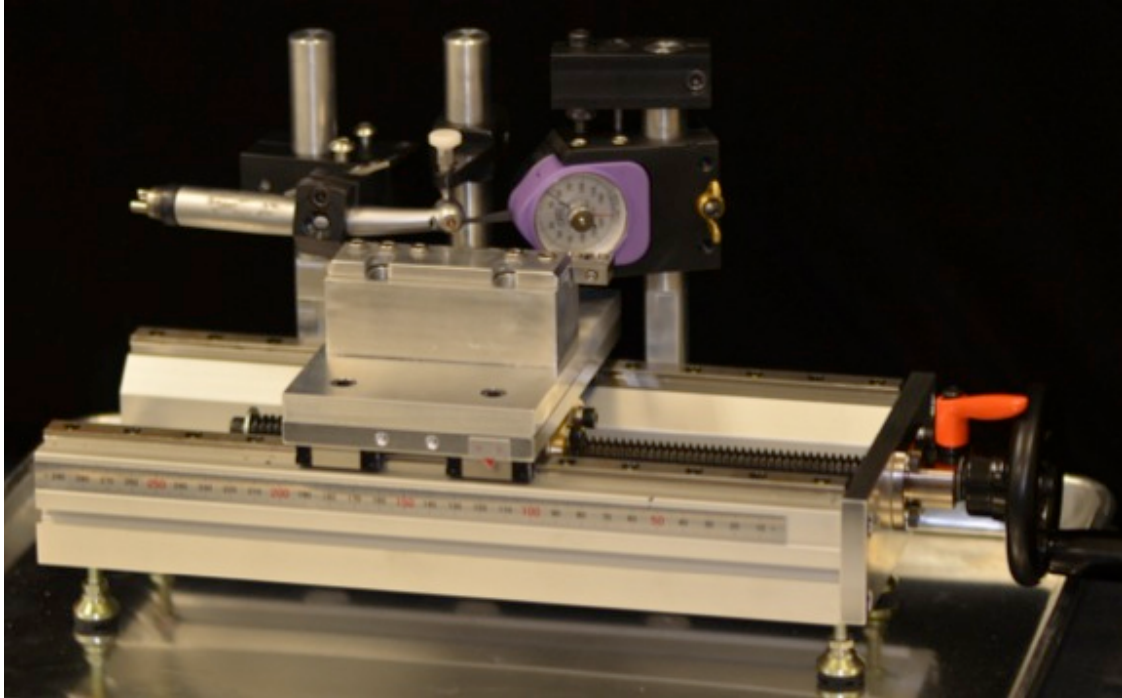


Figure 5: The device used for adjustment and polishing the samples

This device has a scale to measure the applied force during adjustments and polishing the specimens that can be modified to the desired force (Figure 6).

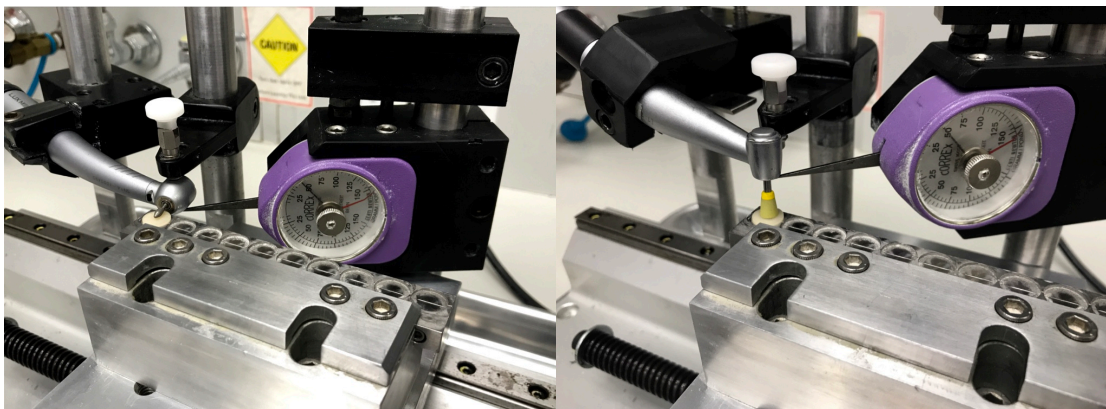


Figure 6: The scale in the device used to have a standardized pressure during polishing

The specimens were placed in 10mm x 2mm split mold that manually moves so that each specimen would receive the same time and force of polishing (Figure 7).



Figure 7: Monolithic ceramic specimens placed in a 10mmx2mm split mold

A fine diamond bur (8369DF.31.025 FG fine football-shape Dialite finishing bur, Brasseler USA, Savannah, GA, USA) and an extra-fine diamond bur (369DEF.31.025 FG extra-fine football-shape Dialite finishing bur, Brasseler USA, Savannah, GA, USA) were used on both surfaces of each disc (the top and bottom sides) for adjustment. Both diamond burs were single used for 5 seconds each at 0.5N of pressure.

After the adjustment procedure completion, the polishing was performed only on the top surface of each disc with the following instruments (W17MLD.RA Dialite LD red medium cup, Brasseler USA, Savannah, GA, USA), (W17FLD.RA Dialite LD yellow fine cup, Brasseler USA, Savannah, GA, USA) Both cups were single used for 40 seconds at 1.5N of pressure (Figure 8).

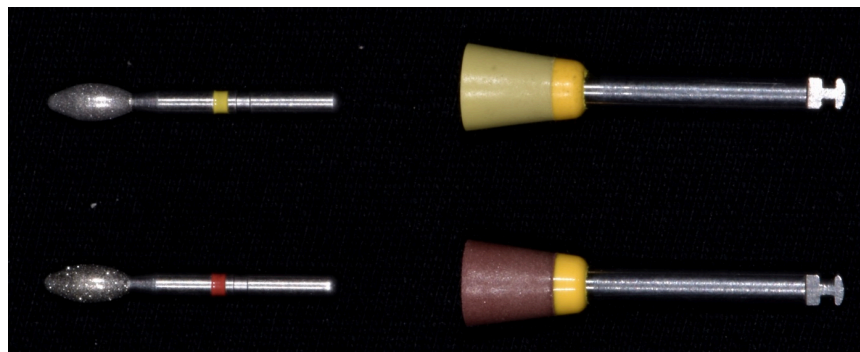


Figure 8: Dialite LD Finishing & Polishing Burs and Cups

2.3 Protocol for reglazing firing procedure:

Only the bottom surface of each disc was subjected to the reglazing firing procedure using Enamelite Nova Universal Low-Fusing Ceramic Spray Glaze following the manufacturer's instructions (Keystone Industries, Singen, Germany) and no polishing burs were used.

All specimens were placed on ceramic tray and held by boxing wax (figure 9).



Figure 9: Enamelite Nova Universal Low-Fusing Ceramic Spray Glaze

The glaze container was shaken well until mixing ball moved freely inside. The glaze was sprayed approximately 10 inches from the specimens using short bursts while agitating the can between sprays on bottom surface of each disc. Then, the specimens were fired in a furnace (IPS e.max Programat CS2, Ivoclar Vivadent, Schaan, Leichtenstein, Germany).

The firing was: pre-dry 1 minute, start temperature 510 °C, firing rate 56 °C per minute, high temperature 871 °C, no vacuum and a holding time of 15 seconds.

2.4 Stage 1: Average surface roughness (Ra) assessment:

After completion of the adjustment and polishing procedures, the samples were rinsed in tap water for 5 seconds, cleaned in an ultrasonic cleaner for 3 minutes, air-dried and stored in distilled water for 48 hours (Figure 10).

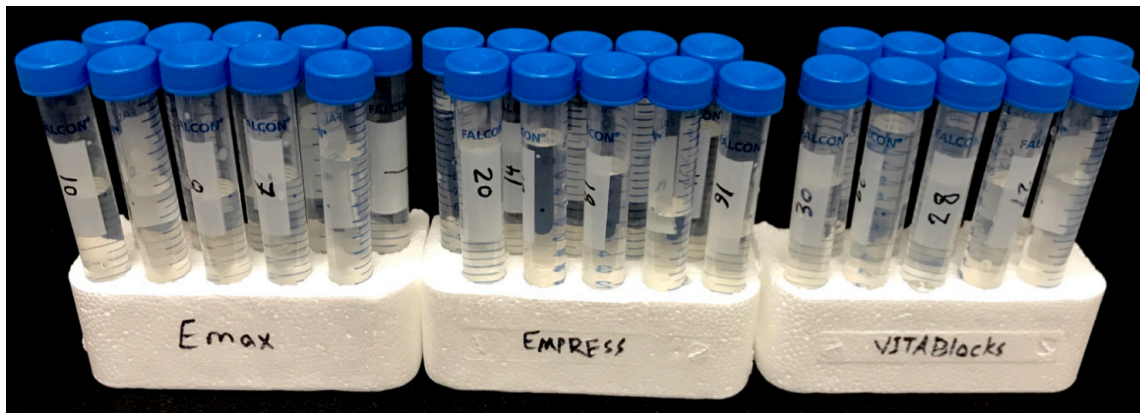


Figure 10: All Samples were stored in distilled water for 48 hours after polishing

A Profilometer (Veeco Dektak 150 profilometer, Veeco Corporate Headquarters, Plainview, NY, USA) was used to measure the average surface roughness of the specimens before and after the adjustment and surface treatment with mechanical polishing and re-glazing procedure (Figure 11). To measure the roughness profile value in micrometers, a diamond stylus (tip radius, 12.5 μm) was moved across the surface under a constant force of 3 mg with a duration of 15 seconds, length of 2 mm and measurement range of 524 μm ("The European Standard (2004) EN 623-624 Advanced technical ceramics. Monolithic ceramics. General and textures properties. Part 4: Determination of surface roughness. Brussels (B): European Committee for Standardization,") (Figure 12).

Three traces were recorded for each specimen at 3 different locations. The mean surface roughness measurement was calculated for each specimen.

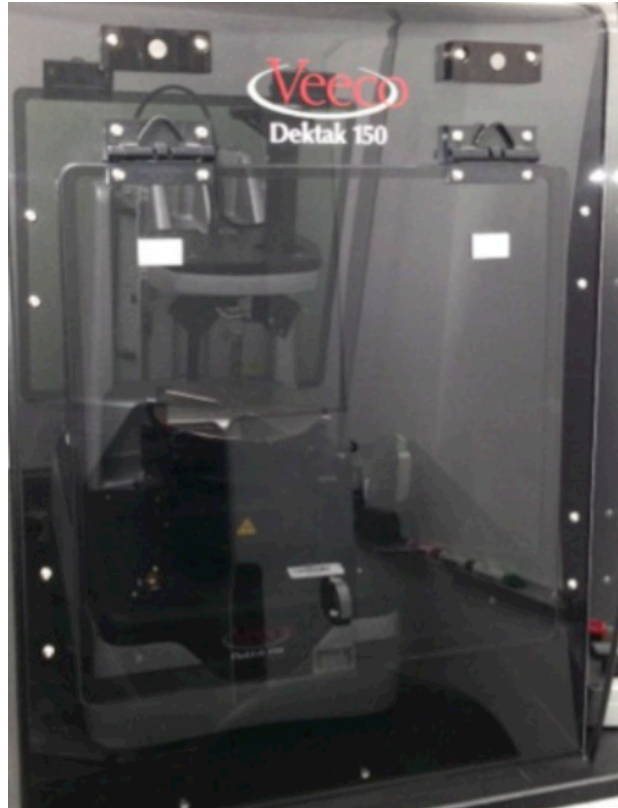


Figure 11: Veeco Dektak 150 profilometer

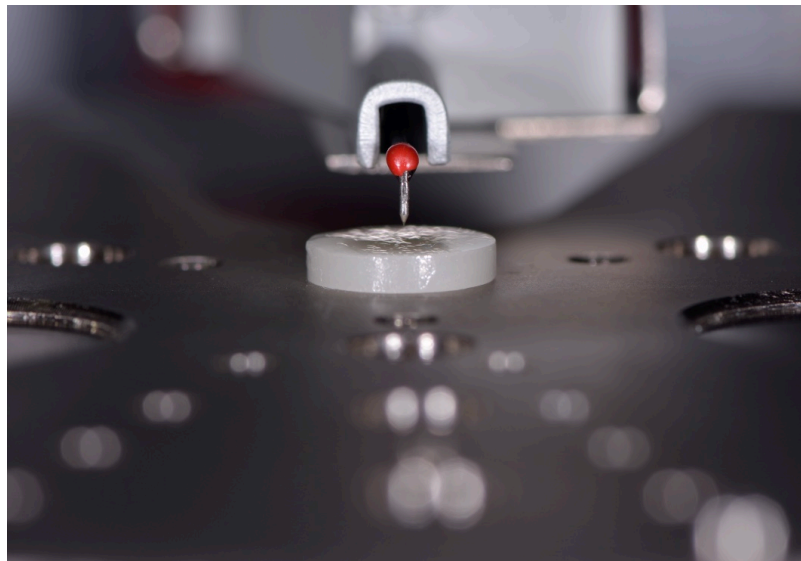


Figure 12: Diamond stylus with 12.5 μm tip radius

2.5 Stage 2: Gloss values (GU) assessment:

A glossmeter (Novo-Curve, Rhopoint Instruments Ltd, Bexhill-on-Sea, UK) with a 60° angle was used for the gloss evaluation (Figure 13).



Figure 13:Novo-Curve Gloss Meter

ISO 2813 specifications for ceramic materials were followed and gloss units (GU) were recorded ("ISO-Standards(1999) EN ISO 2813. Specular gloss. ed. Specular gloss Geneve: International Organization for Standardization.,"). Three measurements were recorded for each specimen at 3 different locations. The average gloss value was calculated for each specimen. All specimens were covered during measuring to avoid any ambient light.

Chapter 3

Results

Frequencies and percentages were calculated for all categorical variables, and means and standard deviations were calculated for continuous measures. To compare differences between the groups, a general linear model was created. The fixed factors were material (E. Max vs. Empress vs. Vita) and treatment (Mechanical vs Reglazing). Post-hoc tests were conducted using a Bonferroni adjustment. R Studio and R 3.2.2 was used for all statistical analysis and significance was accepted at $p < 0.05$. Results are presented below and in Table 3.

3.1 Average surface roughness Ra (Profilometer):

3.1.1 Ra average (Baseline):

There was no significant difference in the measurement of material [F (2,54) = 1.42, $p = 0.249$, $\eta^2 = 31\%$], treatment [F (1,54) = 0.56, $p = 0.457$, $\eta^2 = 31\%$], and the interaction of material by treatment [F (2,54) = 0.07, $p = 0.929$, $\eta^2 = 31\%$]. (Figure14)

3.1.2 Ra average (After treatment):

There was no significant difference in the measurement of material [F (2,54) = 0.49, $p = 0.611$, $\eta^2 = 0.1\%$], but we found a significant treatment effect [F (1,54) = 159.26, $p < 0.001$, $\eta^2 = 74.1\%$], but no interaction effect of material by treatment [F (2,54) = 0.32, $p = 0.722$, $\eta^2 = 0.01\%$]. Post-hoc Tukey results indicate reglazing had a significantly higher Ra average than mechanical (difference = 1.51, 95% CI:1.27,1.75]. (Figure 15)

3.2 Average gloss values GU (Glossmeter):

3.2.1 GU Average (Baseline):

There was no significant difference in the measurement of material [F (2,54) = 0.09, p = 0.909, $\eta^2 = 0.3\%$], treatment effect [F (1,54) = 0.91, p = 0.346, $\eta^2 = 1.5\%$], or the interaction of material by treatment [F (2,54) = 1.29, p = 0.283, $\eta^2 = 4.4\%$]. (Figure 16)

3.2.2 GU Average (After treatment):

There was no significant difference in the measurement of material [F (2,54) = 1.81, p = 0.172, $\eta^2 = 0.1\%$], but we found a significant treatment effect [F (1,54) = 987.34, p < 0.001, $\eta^2 = 94.4\%$], but no interaction effect of material by treatment [F (2,54) = 0.10, p = 0.899, $\eta^2 = 0.01\%$]. Post-hoc Tukey results indicate reglazing had a significantly higher GU average than mechanical (difference = 15.01, 95% CI:14.04,15.96]. (Figure 17)

Table 3: Descriptive Statistics for Measures

| | Material | Treatment | N | M | SD | Min | Max |
|-------------------------------------|----------|------------|----|-------|------|-------|-------|
| Ra Average Baseline | E. Max | Mechanical | 10 | 1.36 | 0.39 | 0.88 | 1.94 |
| | Empress | Mechanical | 10 | 1.39 | 0.26 | 0.99 | 1.71 |
| | Vita | Mechanical | 10 | 1.26 | 0.34 | 0.69 | 1.83 |
| | E. Max | Reglazing | 10 | 1.41 | 0.29 | 0.96 | 1.79 |
| | Empress | Reglazing | 10 | 1.49 | 0.33 | 0.98 | 1.98 |
| | Vita | Reglazing | 10 | 1.29 | 0.31 | 0.86 | 1.76 |
| Ra Average After Treatment | Material | Treatment | N | M | SD | Min | Max |
| | E. Max | Mechanical | 10 | 1.61 | 0.4 | 1.02 | 2.05 |
| | Empress | Mechanical | 10 | 1.65 | 0.35 | 1.08 | 1.93 |
| | Vita | Mechanical | 10 | 1.63 | 0.5 | 1.05 | 2.68 |
| | E. Max | Reglazing | 10 | 3.14 | 0.66 | 2.45 | 4.2 |
| | Empress | Reglazing | 10 | 3.28 | 0.36 | 2.62 | 4 |
| | Vita | Reglazing | 10 | 3.02 | 0.45 | 2.44 | 3.71 |
| GU Average Baseline | Material | Treatment | N | M | SD | Min | Max |
| | E. Max | Mechanical | 10 | 76.57 | 2.22 | 70.93 | 78.7 |
| | Empress | Mechanical | 10 | 77.57 | 4.68 | 69.37 | 84.8 |
| | Vita | Mechanical | 10 | 77.83 | 3.03 | 72.27 | 81.33 |
| | E. Max | Reglazing | 10 | 77.71 | 3.02 | 73.67 | 82.4 |
| | Empress | Reglazing | 10 | 75.79 | 3.17 | 72.3 | 80.8 |
| | Vita | Reglazing | 10 | 76.01 | 3.47 | 71.13 | 81.9 |
| GU Average After Treatment | Material | Treatment | N | M | SD | Min | Max |
| | E. Max | Mechanical | 10 | 43.2 | 1.78 | 40.93 | 46.43 |
| | Empress | Mechanical | 10 | 42.39 | 1.55 | 40.27 | 44.97 |
| | Vita | Mechanical | 10 | 43.08 | 1.33 | 40.83 | 45.3 |
| | E. Max | Reglazing | 10 | 58.49 | 0.96 | 56.1 | 59.9 |
| | Empress | Reglazing | 10 | 57.15 | 3.01 | 52.5 | 59.7 |
| | Vita | Reglazing | 10 | 58.05 | 1.78 | 54.3 | 59.87 |

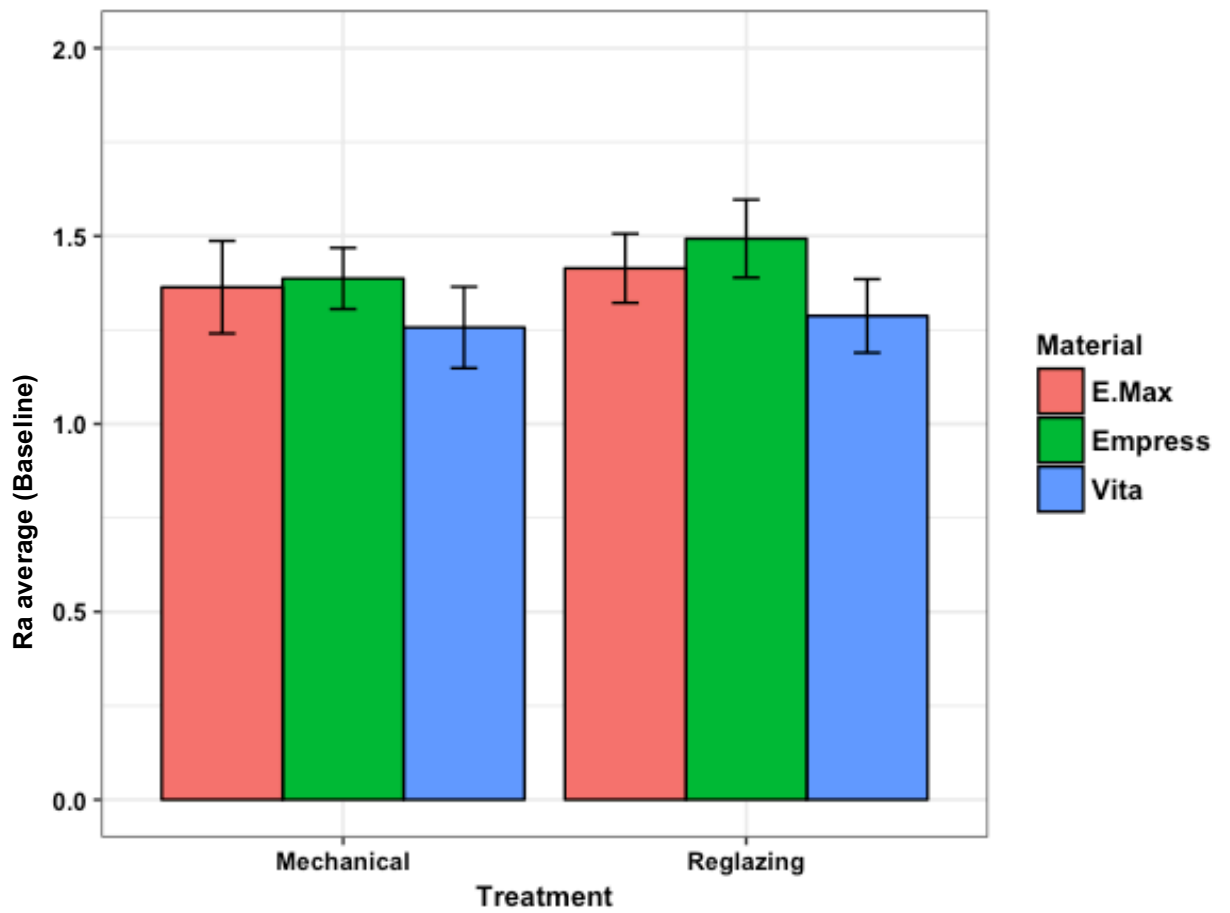


Figure 14: Barplot with 95% standard error bars for Ra Average (Baseline)

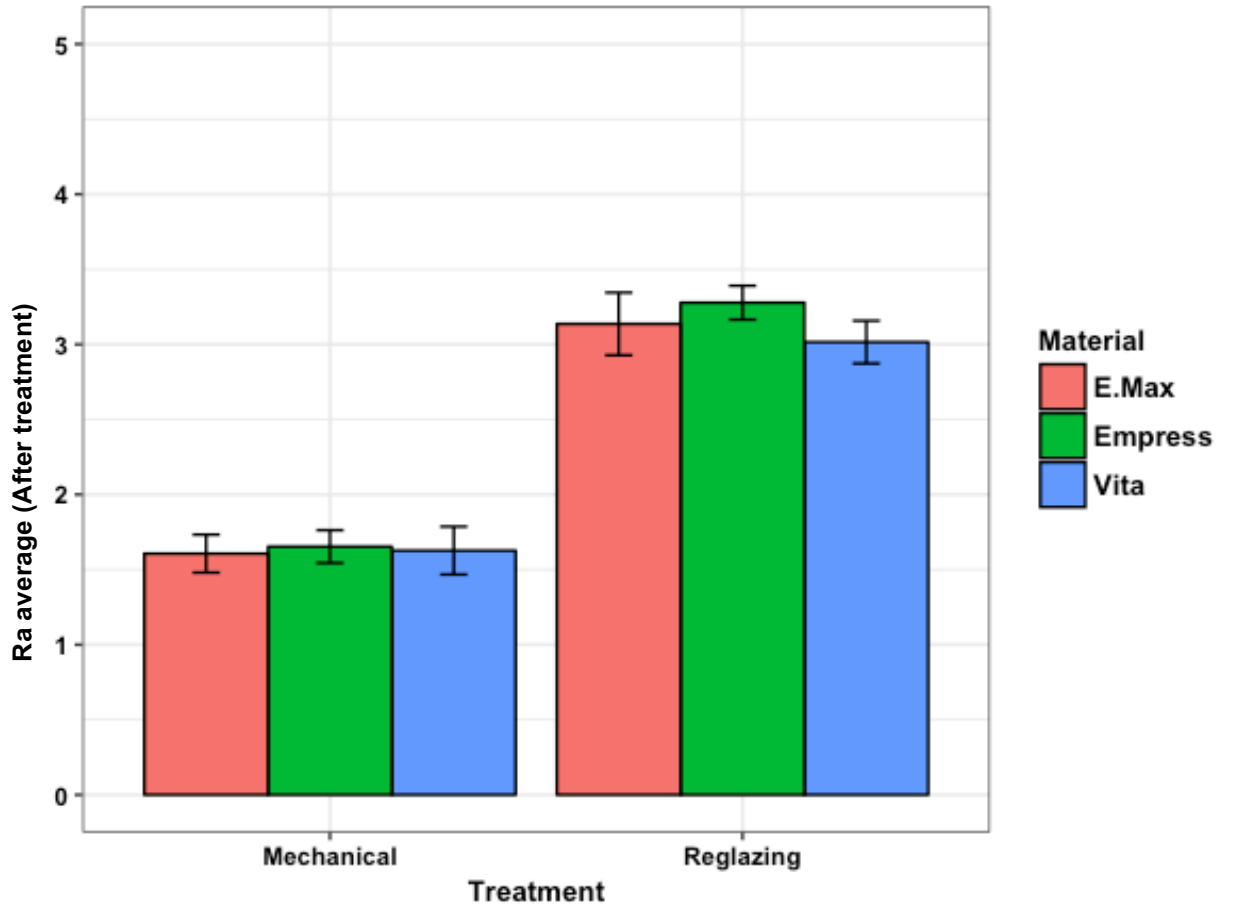


Figure 15: Barplot with 95% standard error bars for Ra Average (After Treatment)

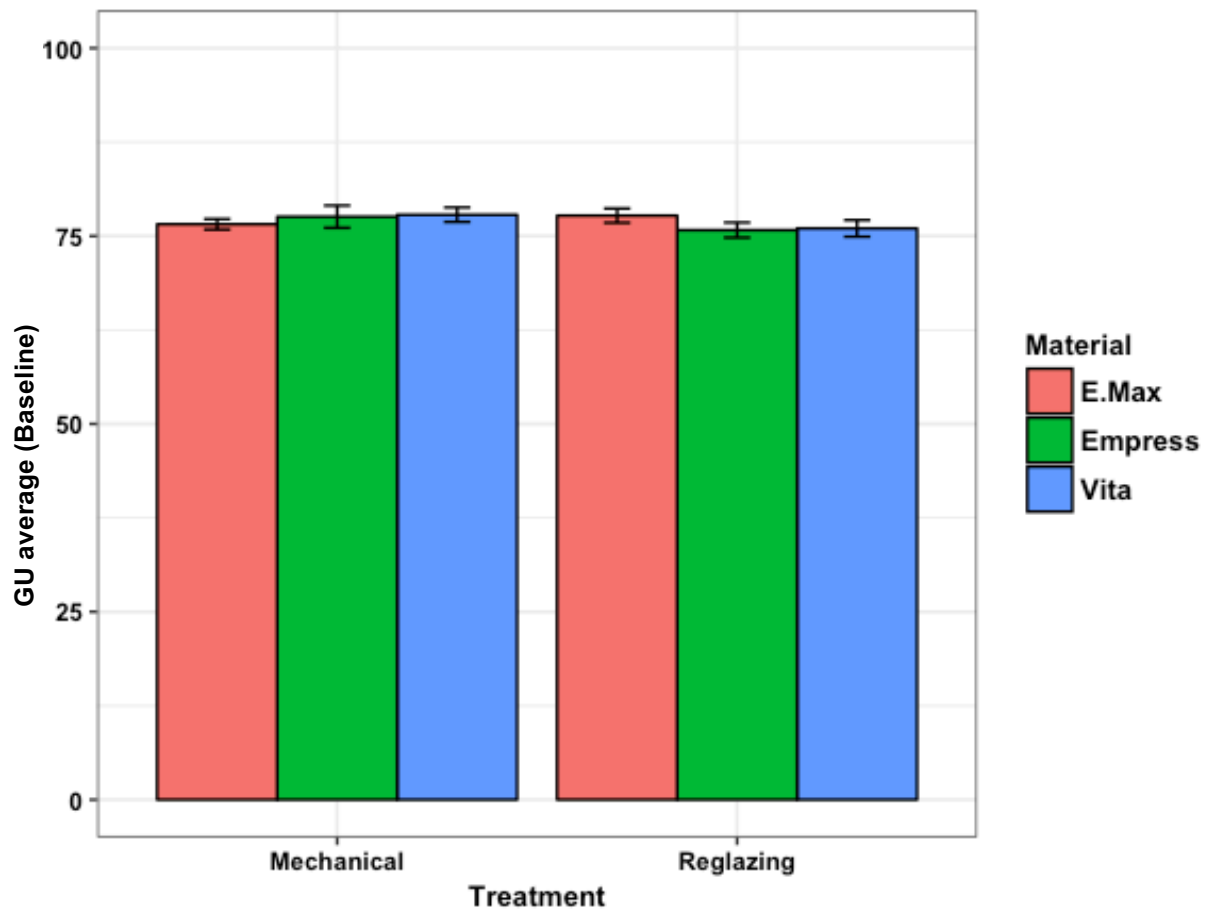


Figure 16: Barplot with 95% standard error bars for GU Average (Baseline)

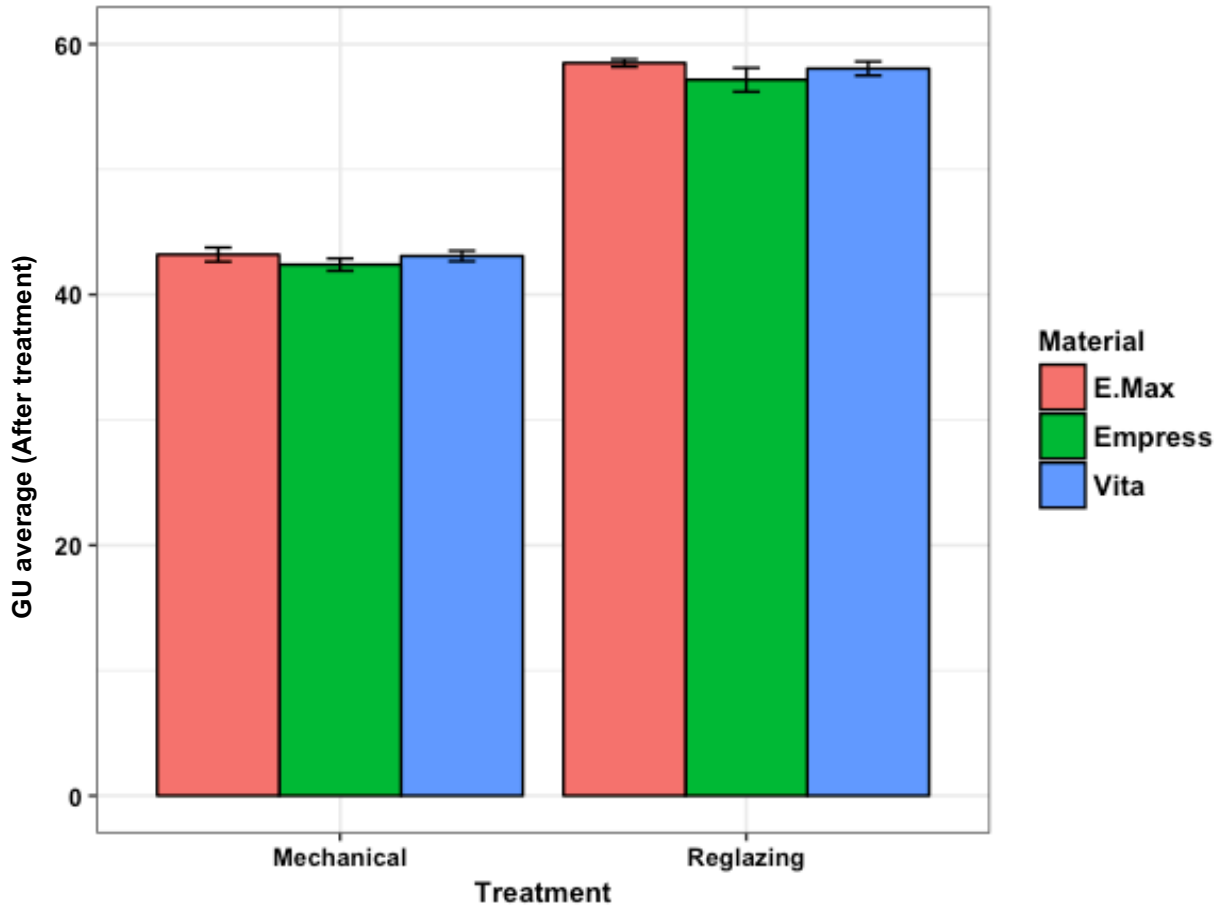


Figure 17: Barplot with 95% standard error bars for GU Average (After treatment)

Chapter 4

Discussion

Based on the results of this study, the null hypotheses were rejected as no difference was found in surface roughness (Ra) and gloss (GU) of monolithic ceramic restorations when subjected to different surface treatments.

The adjustment of occlusion and contacts at the time of delivering a ceramic restoration is crucial since a rough surface is more susceptible to staining and plaque accumulation, thus increasing the likelihood for gingivitis and tooth decay(Quirynen & Bollen, 1995). In addition, rough ceramic restorations are abrasive and can lead to greater wear of opposing teeth or restorations(Heintze, Cavalleri, Forjanic, Zellweger, & Rousson, 2008).

Roughness can also affect the strength of ceramics, thereby causing cracking, chipping, and fracture and the studies showed a significant correlation between the surface roughness and the biaxial flexural strength(de Jager, Feilzer, & Davidson, 2000; Yilmaz & Ozkan, 2010a). The smoother the surface, the stronger the ceramic restoration.

Achieving a smooth ceramic surface is important for a variety of reasons, including esthetics, patient comfort, and biological aspect(Quirynen & Bollen, 1995)

Numerous studies have shown that reglazing firing procedures provide a smooth surface while others have concluded that mechanical polishing may provide a surface showing characteristics more similar to the natural tooth and they prefer the polishing procedures because their higher level of control during the polishing of the final surface (Brewer et al., 1990; Rosenstiel, Baiker, & Johnston, 1989). However, there is not a literature consensus on the ideal surface treatments protocol and there is no publication that

compares all of the surface treatments (Amaya-Pajares et al., 2016; Kim, Lee, Lim, & Kim, 2003).

Ceramics have exhibited the least bacterial and glucan adhesion when compared to other dental restorations (Eick, Glockmann, Brandl, & Pfister, 2004). The controversy regarding the average value of surface roughness (Ra) to achieve the minimal bacterial retention is still current (Carrabba et al., 2017).

The ideal threshold for Ra value is still controversial. Certain *in vivo* studies (Bollen & others, 1997) suggest an ideal value surface roughness for bacterial retention (Ra = 0.2 μm). Enamel roughness was also reported to be a standard parameter (0.45 to 0.65 μm) (Botta et al., 2009; Willems et al., 1991), but it depends on the tooth type and location in the oral cavity. However, other studies reported higher Ra values up to 3 μm resulted from different surface treatments.(Sarac et al., 2006; Scurria & Powers, 1994). None of the surface treatments tested in the present study were able to achieve an Ra < 0.2 μm .

The use of profilometer and the mean roughness value (Ra) measurements are the most common combination used by authors for evaluating surface roughness in dentistry (Sarıkaya & Guler, 2010; Sasahara et al., 2006; Yilmaz & Ozkan, 2010b). The Ra describes the texture of a surface and it can be defined as the mean arithmetical value of all the absolute distances of the roughness profile to the intermediate height along the measured length (Whitehead et al., 1995).

In the present study, the surfaces obtained with reglazing firing procedure were rougher compared with the surfaces finished through using mechanical polishing. This finding is in agreement with previous reports investigating the effects of different surface treatment on the surface roughness of ceramics (Bollen et al., 1997; Jagger & Harrison,

1994; Sarac et al., 2006; Sarikaya & Guler, 2010; Sasahara et al., 2006; Schneider et al., 2013; Wright et al., 2004). However, the results of other studies were contrary to the finding of this present study (A. M. Al-Wahadni & Martin, 1999; Flury et al., 2010; Kawai, Urano, & Ebisu, 2000; Tholt de Vasconcellos, Miranda-Junior, Prioli, Thompson, & Oda, 2006).

In the present study, the angle of incident light was set to 60 degrees, as indicated by ISO 2813 specifications for ceramic materials ("ISO-Standards(1999) EN ISO 2813. Specular gloss. ed. Specular gloss Geneve: International Organization for Standardization.) and the surface topography was considered the main factor influencing the ceramic gloss.

Although the results showed that reglazing firing procedures achieved the highest mean gloss (GU), there was no clinically significant difference when compared to mechanical polishing since a clinically accepted gloss value is reported to range between 40 and 52 GU (Barucci-Pfister & Gohring, 2009; Mormann et al., 2013).

The comparison of the results of this present study with other studies is often challenged by different factors including type of ceramics, the presence of voids and irregularities on a ceramic surface, surface roughness and gloss testing methods. The Ra value measured by the Profilometer is influenced by stylus diameter, scanning speed and length, force and frequency response (Amaya-Pajares et al., 2016).

The limitation of this study include that it is an in vitro study that does not replicate what may happen in the oral cavity such as wear, occlusal forces and presence of the saliva. Also, forces of adjustments and polishing of the samples were standardized which is not the real case in clinical situations.

Further investigations are necessary to evaluate the surface characteristics of other CAD/CAM monolithic ceramic restorations subjected to different surface treatments protocols as well as to simulate other factors present in the oral environment.

Chapter 5

Conclusion

Regardless of the type of ceramic or pre-treatment, any adjusted ceramic restoration should be treated to achieve a clinically accepted smoothness and gloss. None of the tested surface treatments could create a Ra value less than the baseline measurement. Mechanical polishing showed lower Ra and GU values compared to reglazing firing procedure. The large number of variables that effect the ultimate outcome of surface treatment should be considered in future research.

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Appendices

Appendix A: Raw data for surface roughness values (Baseline):

| Group | Sample | Treatment | R1 | R2 | R3 | Average |
|-------|--------|------------|---------|---------|---------|-------------|
| 1 | 1 | Mechanical | 1.25533 | 1.05174 | 1.06199 | 1.12302 |
| 1 | 2 | Mechanical | 0.81997 | 0.96349 | 0.86734 | 0.8836 |
| 1 | 3 | Mechanical | 1.06436 | 0.99408 | 1.06515 | 1.041196667 |
| 1 | 4 | Mechanical | 1.86631 | 1.87595 | 1.88799 | 1.87675 |
| 1 | 5 | Mechanical | 1.13318 | 1.11238 | 1.20234 | 1.1493 |
| 1 | 6 | Mechanical | 1.96691 | 1.97582 | 1.8685 | 1.937076667 |
| 1 | 7 | Mechanical | 1.87478 | 1.78544 | 1.71352 | 1.791246667 |
| 1 | 8 | Mechanical | 1.10557 | 1.15333 | 1.24349 | 1.167463333 |
| 1 | 9 | Mechanical | 1.27814 | 1.5752 | 1.85899 | 1.570776667 |
| 1 | 10 | Mechanical | 1.1112 | 1.06032 | 1.12312 | 1.098213333 |
| 2 | 11 | Mechanical | 1.44342 | 1.53058 | 1.41575 | 1.46325 |
| 2 | 12 | Mechanical | 1.20697 | 1.10218 | 1.11259 | 1.14058 |
| 2 | 13 | Mechanical | 1.10499 | 1.65368 | 1.31951 | 1.359393333 |
| 2 | 14 | Mechanical | 0.82903 | 1.01935 | 1.10926 | 0.98588 |
| 2 | 15 | Mechanical | 1.41063 | 1.38213 | 1.43858 | 1.410446667 |
| 2 | 16 | Mechanical | 1.02236 | 0.98643 | 1.10684 | 1.038543333 |
| 2 | 17 | Mechanical | 1.37816 | 1.58176 | 1.47453 | 1.47815 |
| 2 | 18 | Mechanical | 1.65329 | 1.77818 | 1.58478 | 1.672083333 |
| 2 | 19 | Mechanical | 1.69801 | 1.57419 | 1.5482 | 1.6068 |
| 2 | 20 | Mechanical | 1.69521 | 1.64958 | 1.79858 | 1.714456667 |
| 3 | 21 | Mechanical | 1.06497 | 0.99764 | 1.00239 | 1.021666667 |
| 3 | 22 | Mechanical | 1.80343 | 1.85453 | 1.82602 | 1.827993333 |
| 3 | 23 | Mechanical | 1.23556 | 1.38559 | 1.34485 | 1.322 |
| 3 | 24 | Mechanical | 1.37632 | 1.37026 | 1.31106 | 1.352546667 |
| 3 | 25 | Mechanical | 0.92358 | 1.03183 | 0.98249 | 0.9793 |
| 3 | 26 | Mechanical | 1.09928 | 1.03483 | 1.29584 | 1.143316667 |
| 3 | 27 | Mechanical | 0.99853 | 1.40553 | 1.2333 | 1.212453333 |
| 3 | 28 | Mechanical | 0.78747 | 0.64166 | 0.64812 | 0.692416667 |
| 3 | 29 | Mechanical | 1.72799 | 1.78251 | 1.75662 | 1.755706667 |
| 3 | 30 | Mechanical | 1.17308 | 1.25298 | 1.3523 | 1.259453333 |
| 4 | 31 | Reglazing | 1.7326 | 1.74893 | 1.64145 | 1.70766 |
| 4 | 32 | Reglazing | 1.72429 | 1.6843 | 1.85447 | 1.754353333 |

| | | | | | | |
|---|----|-----------|----------|----------|---------|-------------|
| 4 | 33 | Reglazing | 0.83795 | 0.93118 | 1.09699 | 0.955373333 |
| 4 | 34 | Reglazing | 1.322417 | 1.1375 | 1.18658 | 1.215499 |
| 4 | 35 | Reglazing | 1.3193 | 1.10215 | 1.22427 | 1.21524 |
| 4 | 36 | Reglazing | 1.53711 | 1.76277 | 1.4358 | 1.57856 |
| 4 | 37 | Reglazing | 0.93096 | 1.28987 | 1.11261 | 1.111146667 |
| 4 | 38 | Reglazing | 1.23664 | 1.38414 | 1.36675 | 1.329176667 |
| 4 | 39 | Reglazing | 1.55738 | 1.43653 | 1.46325 | 1.48572 |
| 4 | 40 | Reglazing | 1.41872 | 1.44964 | 2.49876 | 1.78904 |
| 5 | 41 | Reglazing | 1.95433 | 1.98859 | 1.65273 | 1.865216667 |
| 5 | 42 | Reglazing | 1.78999 | 1.11817 | 0.62447 | 1.177543333 |
| 5 | 43 | Reglazing | 1.50965 | 1.36566 | 1.52179 | 1.4657 |
| 5 | 44 | Reglazing | 1.93674 | 1.52363 | 1.68086 | 1.713743333 |
| 5 | 45 | Reglazing | 1.51635 | 1.70324 | 1.50583 | 1.57514 |
| 5 | 46 | Reglazing | 1.1241 | 0.82329 | 1.26705 | 1.07148 |
| 5 | 47 | Reglazing | 0.96085 | 1.05698 | 0.93436 | 0.984063333 |
| 5 | 48 | Reglazing | 1.13903 | 1.7147 | 1.79438 | 1.54937 |
| 5 | 49 | Reglazing | 1.50317 | 1.13603 | 2.01769 | 1.552296667 |
| 5 | 50 | Reglazing | 2.20991 | 1.93647 | 1.78041 | 1.975596667 |
| 6 | 51 | Reglazing | 1.78109 | 1.59294 | 1.45462 | 1.60955 |
| 6 | 52 | Reglazing | 1.59997 | 1.53433 | 1.30277 | 1.479023333 |
| 6 | 53 | Reglazing | 1.01192 | 1.18325 | 1.217 | 1.13739 |
| 6 | 54 | Reglazing | 1.89496 | 1.50162 | 1.4923 | 1.629626667 |
| 6 | 55 | Reglazing | 1.04835 | 1.03152 | 0.99866 | 1.026176667 |
| 6 | 56 | Reglazing | 1.86764 | 1.699679 | 1.72632 | 1.764546333 |
| 6 | 57 | Reglazing | 1.04891 | 1.05705 | 1.49403 | 1.199996667 |
| 6 | 58 | Reglazing | 1.06321 | 0.92505 | 0.98851 | 0.992256667 |
| 6 | 59 | Reglazing | 1.25206 | 1.1569 | 1.10155 | 1.17017 |
| 6 | 60 | Reglazing | 0.62563 | 0.973856 | 0.99423 | 0.864572 |

Appendix B: Raw data for surface roughness values (After treatment):

| Group | Sample | Treatment | R1 | R2 | R3 | Average |
|-------|--------|------------|---------|----------|---------|-------------|
| 1 | 1 | Mechanical | 1.45305 | 1.31985 | 1.21955 | 1.330816667 |
| 1 | 2 | Mechanical | 1.04354 | 1.0479 | 0.96314 | 1.018193333 |
| 1 | 3 | Mechanical | 1.24853 | 1.01718 | 1.09313 | 1.119613333 |
| 1 | 4 | Mechanical | 2.1824 | 1.98511 | 1.96987 | 2.045793333 |
| 1 | 5 | Mechanical | 1.3656 | 2.02082 | 2.52891 | 1.971776667 |
| 1 | 6 | Mechanical | 2.07752 | 2.06347 | 1.90477 | 2.015253333 |
| 1 | 7 | Mechanical | 1.97421 | 1.9224 | 2.08445 | 1.993686667 |
| 1 | 8 | Mechanical | 1.33885 | 1.21031 | 1.58258 | 1.377246667 |
| 1 | 9 | Mechanical | 1.74743 | 1.6637 | 1.97299 | 1.794706667 |
| 1 | 10 | Mechanical | 1.43971 | 1.33095 | 1.42021 | 1.396956667 |
| 2 | 11 | Mechanical | 1.94711 | 1.97174 | 1.8332 | 1.91735 |
| 2 | 12 | Mechanical | 1.28709 | 1.28201 | 1.29946 | 1.28952 |
| 2 | 13 | Mechanical | 1.8691 | 1.894296 | 1.9052 | 1.889532 |
| 2 | 14 | Mechanical | 0.93145 | 1.12833 | 1.1849 | 1.08156 |
| 2 | 15 | Mechanical | 1.75651 | 1.79231 | 1.78965 | 1.77949 |
| 2 | 16 | Mechanical | 0.99862 | 1.19162 | 1.21337 | 1.134536667 |
| 2 | 17 | Mechanical | 1.89914 | 1.52875 | 1.69797 | 1.70862 |
| 2 | 18 | Mechanical | 1.99915 | 1.91369 | 1.8475 | 1.920113333 |
| 2 | 19 | Mechanical | 1.86135 | 1.79408 | 1.97816 | 1.877863333 |
| 2 | 20 | Mechanical | 1.94968 | 1.97872 | 1.87183 | 1.93341 |
| 3 | 21 | Mechanical | 1.19098 | 1.11351 | 1.31214 | 1.205543333 |
| 3 | 22 | Mechanical | 1.97852 | 1.99284 | 1.98743 | 1.986263333 |
| 3 | 23 | Mechanical | 1.83235 | 1.75786 | 1.89857 | 1.829593333 |
| 3 | 24 | Mechanical | 1.5267 | 1.48657 | 1.65362 | 1.55563 |
| 3 | 25 | Mechanical | 0.98742 | 1.19071 | 0.9752 | 1.05111 |
| 3 | 26 | Mechanical | 1.38763 | 1.37501 | 1.3022 | 1.354946667 |
| 3 | 27 | Mechanical | 1.62567 | 1.51539 | 1.68093 | 1.60733 |
| 3 | 28 | Mechanical | 1.13835 | 0.95276 | 1.09006 | 1.06039 |
| 3 | 29 | Mechanical | 1.99144 | 1.93154 | 1.89267 | 1.93855 |
| 3 | 30 | Mechanical | 2.41313 | 2.57572 | 3.04764 | 2.67883 |
| 4 | 31 | Reglazing | 3.34564 | 3.3983 | 3.28623 | 3.34339 |
| 4 | 32 | Reglazing | 4.54728 | 4.2058 | 3.84626 | 4.19978 |
| 4 | 33 | Reglazing | 2.48831 | 2.53991 | 2.32951 | 2.452576667 |

| | | | | | | |
|---|----|-----------|---------|---------|----------|-------------|
| 4 | 34 | Reglazing | 2.82456 | 3.27053 | 4.87442 | 3.656503333 |
| 4 | 35 | Reglazing | 3.06574 | 2.25721 | 3.46551 | 2.929486667 |
| 4 | 36 | Reglazing | 3.94524 | 4.39552 | 4.02883 | 4.123196667 |
| 4 | 37 | Reglazing | 2.69043 | 2.79896 | 2.27757 | 2.588986667 |
| 4 | 38 | Reglazing | 3.83986 | 2.20119 | 2.66836 | 2.903136667 |
| 4 | 39 | Reglazing | 2.2169 | 2.68746 | 3.08062 | 2.66166 |
| 4 | 40 | Reglazing | 2.53295 | 2.43446 | 2.54282 | 2.50341 |
| 5 | 41 | Reglazing | 3.11043 | 3.07925 | 4.20129 | 3.463656667 |
| 5 | 42 | Reglazing | 2.51652 | 3.04262 | 3.68449 | 3.08121 |
| 5 | 43 | Reglazing | 3.42749 | 3.21275 | 3.39692 | 3.34572 |
| 5 | 44 | Reglazing | 2.68584 | 2.60936 | 2.55536 | 2.616853333 |
| 5 | 45 | Reglazing | 3.40589 | 3.32588 | 3.06666 | 3.266143333 |
| 5 | 46 | Reglazing | 2.95948 | 2.94733 | 3.29132 | 3.066043333 |
| 5 | 47 | Reglazing | 3.66224 | 3.57049 | 2.58294 | 3.27189 |
| 5 | 48 | Reglazing | 3.81821 | 3.35117 | 3.33931 | 3.502896667 |
| 5 | 49 | Reglazing | 4.33865 | 3.66974 | 3.98839 | 3.998926667 |
| 5 | 50 | Reglazing | 3.01727 | 3.01512 | 3.45864 | 3.163676667 |
| 6 | 51 | Reglazing | 4.94358 | 2.95131 | 3.2349 | 3.70993 |
| 6 | 52 | Reglazing | 2.667 | 3.31364 | 3.12781 | 3.03615 |
| 6 | 53 | Reglazing | 3.07222 | 3.55327 | 2.11005 | 2.911846667 |
| 6 | 54 | Reglazing | 2.97061 | 3.16454 | 4.016959 | 3.384036333 |
| 6 | 55 | Reglazing | 2.03274 | 2.03385 | 3.24237 | 2.43632 |
| 6 | 56 | Reglazing | 2.41812 | 2.74649 | 2.74163 | 2.635413333 |
| 6 | 57 | Reglazing | 3.57886 | 3.58573 | 3.88622 | 3.683603333 |
| 6 | 58 | Reglazing | 3.0878 | 3.08539 | 2.96698 | 3.046723333 |
| 6 | 59 | Reglazing | 2.30341 | 2.70564 | 3.00963 | 2.672893333 |
| 6 | 60 | Reglazing | 2.49699 | 2.49339 | 2.91395 | 2.634776667 |

Appendix C: Raw data for Gloss values (Baseline):

| Group | Sample | Treatment | R1 | R2 | R3 | Average |
|-------|--------|------------|------|------|------|---------|
| 1 | 1 | Mechanical | 77.1 | 76.9 | 77 | 77.1 |
| 1 | 2 | Mechanical | 76.9 | 77.2 | 79.2 | 76.9 |
| 1 | 3 | Mechanical | 81.5 | 77.2 | 77.4 | 81.5 |
| 1 | 4 | Mechanical | 79.6 | 79.7 | 74.2 | 79.6 |
| 1 | 5 | Mechanical | 77 | 78.6 | 77.7 | 77 |
| 1 | 6 | Mechanical | 75.1 | 78.8 | 79.4 | 75.1 |
| 1 | 7 | Mechanical | 71.4 | 71.6 | 69.8 | 71.4 |
| 1 | 8 | Mechanical | 73.9 | 75.7 | 76.1 | 73.9 |
| 1 | 9 | Mechanical | 79.2 | 70.1 | 79.6 | 79.2 |
| 1 | 10 | Mechanical | 75.7 | 77.1 | 76.4 | 75.7 |
| 2 | 11 | Mechanical | 86.7 | 81.4 | 86.3 | 86.7 |
| 2 | 12 | Mechanical | 80.9 | 79.3 | 78.6 | 80.9 |
| 2 | 13 | Mechanical | 69.3 | 68.9 | 69.9 | 69.3 |
| 2 | 14 | Mechanical | 76.4 | 76.5 | 71.8 | 76.4 |
| 2 | 15 | Mechanical | 77.8 | 74.2 | 74.1 | 77.8 |
| 2 | 16 | Mechanical | 82.5 | 83.3 | 80.7 | 82.5 |
| 2 | 17 | Mechanical | 80.7 | 81.9 | 79.6 | 80.7 |
| 2 | 18 | Mechanical | 77.3 | 79.7 | 83.5 | 77.3 |
| 2 | 19 | Mechanical | 73.5 | 72.5 | 73.5 | 73.5 |
| 2 | 20 | Mechanical | 73.8 | 76.5 | 76.1 | 73.8 |
| 3 | 21 | Mechanical | 80.4 | 81.4 | 79.4 | 80.4 |
| 3 | 22 | Mechanical | 71.6 | 71.1 | 74.1 | 71.6 |
| 3 | 23 | Mechanical | 75.4 | 80.1 | 78.1 | 75.4 |
| 3 | 24 | Mechanical | 70.6 | 74.2 | 74.2 | 70.6 |
| 3 | 25 | Mechanical | 81.1 | 78.8 | 76 | 81.1 |
| 3 | 26 | Mechanical | 81.2 | 82.8 | 80 | 81.2 |
| 3 | 27 | Mechanical | 79.4 | 79.2 | 73.2 | 79.4 |
| 3 | 28 | Mechanical | 79.3 | 79.8 | 80.2 | 79.3 |
| 3 | 29 | Mechanical | 79.1 | 74.3 | 79.9 | 79.1 |
| 3 | 30 | Mechanical | 80.3 | 79.4 | 80.4 | 80.3 |
| 4 | 31 | Reglazing | 78.2 | 74.1 | 74.2 | 78.2 |
| 4 | 32 | Reglazing | 80.5 | 82.6 | 81.1 | 80.5 |
| 4 | 33 | Reglazing | 73.8 | 76.7 | 79.8 | 73.8 |

| | | | | | | |
|---|----|-----------|------|------|------|------|
| 4 | 34 | Reglazing | 75.7 | 73.7 | 71.6 | 75.7 |
| 4 | 35 | Reglazing | 82.4 | 80.3 | 84.5 | 82.4 |
| 4 | 36 | Reglazing | 78.9 | 70.7 | 71.8 | 78.9 |
| 4 | 37 | Reglazing | 79.8 | 78.5 | 79.1 | 79.8 |
| 4 | 38 | Reglazing | 75.4 | 78.8 | 77.4 | 75.4 |
| 4 | 39 | Reglazing | 79.5 | 80.2 | 80.9 | 79.5 |
| 4 | 40 | Reglazing | 79.8 | 71.5 | 79.9 | 79.8 |
| 5 | 41 | Reglazing | 78.5 | 80.4 | 76.6 | 78.5 |
| 5 | 42 | Reglazing | 79.8 | 78.7 | 79.2 | 79.8 |
| 5 | 43 | Reglazing | 72.2 | 72.1 | 75.3 | 72.2 |
| 5 | 44 | Reglazing | 73.4 | 73.3 | 70.2 | 73.4 |
| 5 | 45 | Reglazing | 77.9 | 75.3 | 82.5 | 77.9 |
| 5 | 46 | Reglazing | 72.2 | 72.3 | 73 | 72.2 |
| 5 | 47 | Reglazing | 81.9 | 81.8 | 78.7 | 81.9 |
| 5 | 48 | Reglazing | 75.3 | 71.9 | 78.2 | 75.3 |
| 5 | 49 | Reglazing | 76.6 | 72.3 | 73.7 | 76.6 |
| 5 | 50 | Reglazing | 71.1 | 69.4 | 79.8 | 71.1 |
| 6 | 51 | Reglazing | 71.6 | 70.3 | 71.5 | 71.6 |
| 6 | 52 | Reglazing | 73.6 | 71.2 | 71.9 | 73.6 |
| 6 | 53 | Reglazing | 71.1 | 78.5 | 79.6 | 71.1 |
| 6 | 54 | Reglazing | 78.7 | 71.2 | 78.2 | 78.7 |
| 6 | 55 | Reglazing | 79.3 | 82.3 | 79.6 | 79.3 |
| 6 | 56 | Reglazing | 76.2 | 75.6 | 79.5 | 76.2 |
| 6 | 57 | Reglazing | 77.8 | 76.5 | 78.3 | 77.8 |
| 6 | 58 | Reglazing | 74.2 | 70.5 | 73.5 | 74.2 |
| 6 | 59 | Reglazing | 82.9 | 83.6 | 79.2 | 82.9 |
| 6 | 60 | Reglazing | 72.2 | 74.6 | 77.1 | 72.2 |

Appendix D: Raw data for Gloss values (After treatment):

| Group | Sample | Treatment | R1 | R2 | R3 | Average |
|-------|--------|------------|------|------|------|---------|
| 1 | 1 | Mechanical | 46.3 | 45.8 | 47.2 | 46.3 |
| 1 | 2 | Mechanical | 44.9 | 42.4 | 43.6 | 44.9 |
| 1 | 3 | Mechanical | 44.7 | 45.2 | 45.8 | 44.7 |
| 1 | 4 | Mechanical | 40.1 | 41.4 | 41.8 | 40.1 |
| 1 | 5 | Mechanical | 41.2 | 40.1 | 42.8 | 41.2 |
| 1 | 6 | Mechanical | 40.7 | 41.3 | 40.8 | 40.7 |
| 1 | 7 | Mechanical | 43.8 | 42.6 | 44.7 | 43.8 |
| 1 | 8 | Mechanical | 44.7 | 43.5 | 43.2 | 44.7 |
| 1 | 9 | Mechanical | 41.1 | 43.9 | 44.6 | 41.1 |
| 1 | 10 | Mechanical | 43.6 | 41.9 | 42.3 | 43.6 |
| 2 | 11 | Mechanical | 44.5 | 44.3 | 45.1 | 44.5 |
| 2 | 12 | Mechanical | 39.8 | 42.2 | 43.8 | 39.8 |
| 2 | 13 | Mechanical | 40.1 | 41.5 | 39.2 | 40.1 |
| 2 | 14 | Mechanical | 39.3 | 42.8 | 41.7 | 39.3 |
| 2 | 15 | Mechanical | 41.7 | 40.6 | 41.3 | 41.7 |
| 2 | 16 | Mechanical | 45.2 | 44.1 | 45.6 | 45.2 |
| 2 | 17 | Mechanical | 43.5 | 44.3 | 42.9 | 43.5 |
| 2 | 18 | Mechanical | 41.8 | 40.1 | 42.7 | 41.8 |
| 2 | 19 | Mechanical | 41.7 | 42.4 | 41.3 | 41.7 |
| 2 | 20 | Mechanical | 43.9 | 43.7 | 40.7 | 43.9 |
| 3 | 21 | Mechanical | 40.1 | 40.6 | 41.8 | 40.1 |
| 3 | 22 | Mechanical | 41.8 | 42.1 | 42.6 | 41.8 |
| 3 | 23 | Mechanical | 41.4 | 43.2 | 42.9 | 41.4 |
| 3 | 24 | Mechanical | 45.8 | 43.2 | 44.6 | 45.8 |
| 3 | 25 | Mechanical | 42.3 | 41.8 | 43.6 | 42.3 |
| 3 | 26 | Mechanical | 44.2 | 45.8 | 43.5 | 44.2 |
| 3 | 27 | Mechanical | 43.2 | 42.3 | 42.1 | 43.2 |
| 3 | 28 | Mechanical | 43.6 | 42.1 | 43.8 | 43.6 |
| 3 | 29 | Mechanical | 42.2 | 44.1 | 41.9 | 42.2 |
| 3 | 30 | Mechanical | 46.1 | 44.3 | 45.5 | 46.1 |
| 4 | 31 | Reglazing | 58.4 | 57.5 | 58.8 | 58.4 |
| 4 | 32 | Reglazing | 56.7 | 58.5 | 60.2 | 56.7 |
| 4 | 33 | Reglazing | 58.2 | 57.3 | 59.3 | 58.2 |

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|---|----|-----------|------|------|------|------|
| 4 | 34 | Reglazing | 59.8 | 57.4 | 58.7 | 59.8 |
| 4 | 35 | Reglazing | 59.6 | 60.2 | 57.3 | 59.6 |
| 4 | 36 | Reglazing | 56.1 | 56.3 | 55.9 | 56.1 |
| 4 | 37 | Reglazing | 58.7 | 59.6 | 58.2 | 58.7 |
| 4 | 38 | Reglazing | 59.9 | 58.6 | 57.8 | 59.9 |
| 4 | 39 | Reglazing | 60.8 | 59.1 | 59.8 | 60.8 |
| 4 | 40 | Reglazing | 57.4 | 59.2 | 59.5 | 57.4 |
| 5 | 41 | Reglazing | 58.9 | 57.4 | 60.2 | 58.9 |
| 5 | 42 | Reglazing | 59.2 | 58.6 | 57.4 | 59.2 |
| 5 | 43 | Reglazing | 52.4 | 51.8 | 53.3 | 52.4 |
| 5 | 44 | Reglazing | 53.1 | 54.3 | 52.2 | 53.1 |
| 5 | 45 | Reglazing | 58.8 | 57.7 | 59.6 | 58.8 |
| 5 | 46 | Reglazing | 52.8 | 54.3 | 51.3 | 52.8 |
| 5 | 47 | Reglazing | 60.7 | 59.9 | 58.4 | 60.7 |
| 5 | 48 | Reglazing | 58.2 | 59.8 | 59.3 | 58.2 |
| 5 | 49 | Reglazing | 56.4 | 59.6 | 59.8 | 56.4 |
| 5 | 50 | Reglazing | 59.2 | 60.4 | 59.5 | 59.2 |
| 6 | 51 | Reglazing | 54.9 | 55.8 | 56.5 | 54.9 |
| 6 | 52 | Reglazing | 59.2 | 58.9 | 59.3 | 59.2 |
| 6 | 53 | Reglazing | 59.4 | 58.7 | 59.3 | 59.4 |
| 6 | 54 | Reglazing | 58.8 | 57.9 | 56.5 | 58.8 |
| 6 | 55 | Reglazing | 60.1 | 59.3 | 59.4 | 60.1 |
| 6 | 56 | Reglazing | 58.3 | 57.5 | 57.4 | 58.3 |
| 6 | 57 | Reglazing | 59.7 | 57.3 | 58.7 | 59.7 |
| 6 | 58 | Reglazing | 54.7 | 53.9 | 54.3 | 54.7 |
| 6 | 59 | Reglazing | 60.3 | 59.5 | 59.8 | 60.3 |
| 6 | 60 | Reglazing | 59.5 | 57.8 | 58.8 | 59.5 |