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IMPACT OF GASTRIC ACID INDUCED SURFACE CHANGES ON MECHANICAL BEHAVIOR AND OPTICAL CHARACTERISTICS OF DENTAL CERAMICS

ADITI KULKARNI, 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

July 2016



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By

ADITI KULKARNI, 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 IN DENTISTRY

Department of Prosthodontics

College of Dental Medicine

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July 2016

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DATE SUBMITED: July 22, 2016

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.Sc.D. degree and for this assignment.

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DEDICATION

To my loving husband and family for their endless love and support.



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IMPACT OF GASTRIC ACID INDUCED SURFACE CHANGES ON MECHANICAL BEHAVIOR AND OPTICAL CHARACTERISTICS OF DENTAL CERAMICS

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Objective. This study was conducted to test the impact of exposure to artificial gastric acid combined with toothbrush abrasion on the properties of dental ceramics. Earlier research has indicated that immersion in artificial gastric acid has caused increased surface roughness of dental ceramics. However, the combined effect of acid immersion and toothbrush abrasion and the impact of increased surface roughness on mechanical strength and optical properties has not been studied. **Methods**. Three commercially available ceramics were chosen for this study: feldspathic porcelain, lithium disilicate glass ceramic and monolithic zirconium oxide. The specimens (10×1 mm discs) were cut, thermally treated as required and polished. Each material was divided into four groups (n=8 per group): Control (no exposure), Acid only, Brush only, Acid + Brush. The specimens were immersed in artificial gastric acid (50 ml of 0.2% (w/v) Sodium chloride in 0.7% (v/v) Hydrochloric acid mixed with 0.16g of pepsin powder, pH=2) for 2 minutes



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and rinsed with deionized water for 2 minutes. The procedure was repeated 6 times/day x 9 days and specimens were stored in deionized water at 37°C. Toothbrush abrasion was performed using an ISO/ADA design brushing machine (Sabri Dental Enterprises, Inc.) for 100 cycles/day x 9 days. The Acid + Brush group received both treatments. Specimens were examined under SEM and an optical microscope for morphological changes. Color and translucency were measured using spectrophotometer CIELAB coordinates (L*, a* and b*); surface gloss was measured using a gloss meter. Surface roughness was measured using a stylus profilometer; biaxial strength was measured using an Instron mechanical testing machine. The data was analyzed by one-way ANOVA followed by Tukey's HSD post hoc test (p<0.05). **Results**. Statistically significant changes were found for color, gloss and surface roughness for porcelain and E.max samples. No statistically significant changes were found for any properties of zirconia samples. Conclusion. The acid treatment affected the surface roughness, color and gloss of porcelain and E.max ceramics. The changes in translucency and mechanical strength for all materials were not statistically significant. Zirconia ceramic showed resistance to all treatments.



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CHAPTER 1: INTRODUCTION

1.1 Prosthodontics and restorative dentistry

The specialty of Prosthodontics was second to Oral Surgery to appear as a dental specialty, and is recognized as an adaptable and innovative branch of dentistry, evolved to manage more sophisticated patient needs and embrace new technologies¹. There is a rise in the elderly population in industrialized countries leading to distinct oral health problems, including but not limited to those affecting restorative dentistry such as root caries or dental erosion, of which the prevalence seems to be on the rise²⁻³. In keeping with increasing demands in oral health, dental research has invested a great deal in development of state of the art dental materials. Restorative dentistry has undergone significant advances in therapeutic modalities to manage complex dental conditions⁴. The goal of any dental rehabilitation is to restore health, function and esthetics. In addition, the durability and predictability of interceptive dental treatment is an important factor in the decision making process. There seems to be a paradigm shift towards metal-free ceramic restorations in modern day clinical dentistry due to the significant improvement in the mechanical and optical properties of this class of materials. This has made it possible to accomplish comprehensive dental reconstructions which are esthetically superior and meet functional requirements. As a specialty, Prosthodontics has always been at the cutting edge of technological progressions. In today's world, since patients have easy access to exploring treatment options, there is an increased awareness among the population about seeking dental therapy.



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1.2 Tooth surface loss

Wear is defined as deterioration as a result of use⁵ and the term was first used by Hunter in Dentistry⁶. Tooth surface loss occurs due to a variety of causes listed in the Figure 1.

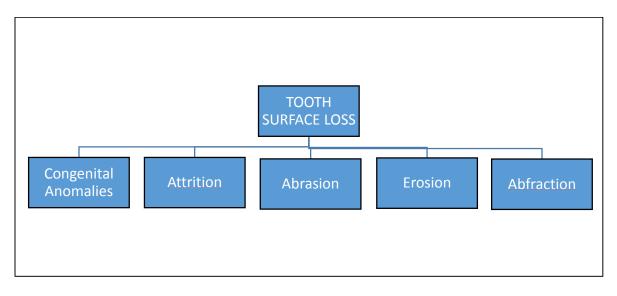


Figure 1. Different causes of tooth surface loss

Tooth wear can also be broadly categorized based on its etiology as occurring due to mechanical, chemical or biomechanical causes.⁷

Attrition is defined as the mechanical wear resulting from mastication or parafunction limited to contacting surfaces of teeth.⁸ Physiologic wear of contacting surfaces of teeth is a normal occurrence during the lifetime of an individual.⁹ Patients often report no symptoms and lesions can be clearly identified on the occluding surfaces of the teeth as small polished facets on the cusp or ridge or a slight flattening of an incisal edge.¹⁰ Reports have indicated that estimated tooth loss due to wear of enamel is approximately 65µm/year.¹¹



Abrasion is defined as the abnormal wearing of tooth substance by causes other than mastication. This type of wear is caused due to foreign objects or substances continuously rubbing against tooth surfaces. For example, aggressive tooth brushing with abrasive dentifrices, damaging habits such as opening hair pins with teeth or biting nails, tacks, pins, thread, a pipe stem, or a wind instrument.¹⁰ Each of the mentioned substances/objects produce a characteristic pattern of wear and is a valuable diagnostic parameter.

Abfraction is the abnormal loss of tooth substance caused by biomechanical loading factors. The lesions present themselves as wedge-shaped defects at the cervical areas of the teeth and their etiopathology remains controversial.¹²⁻¹³ Congenital abnormalities like Amelogenesis Imperfecta and Dentinogenesis Imperfecta can also cause loss of tooth structure with very peculiar clinical presentations. These abnormalities are shown to accelerate other forms of tooth wear due to decreased wear resistance and weakening of the tooth structure. ¹⁴ An understanding of the etiological factors is essential to differentiate these forms of wear from dental erosion, which is caused due to chemical factors.

1.2.1 Dental Erosion

The Glossary of Prosthodontic Terms defines erosion as the progressive loss of tooth substance by chemical processes that do not involve bacterial action producing defects that are sharply defined wedge-shaped depressions, often in facial and cervical areas. There are increasing number of reports in the literature suggesting that dental erosion is a major cause of dental wear, surpassing wear



dueto attrition or abrasion.¹⁵⁻¹⁷ Chemical insults to the teeth seem to cause the most accelerated tooth loss leading to severe damage within a considerably short period of time. There has been growing awareness about this condition during the last few decades.⁷ The clinical presentation of the lesions varies according to the causative factors, which can be either intrinsic or extrinsic. Table 1 summarizes the intrinsic and extrinsic causes of dental erosion. ¹⁸

	EXTRINSIC	INTRINSIC
1	Carbonated beverages, acidic foods, citric lozenges, medications	Bulimia Nervosa
2	Oral hygiene swab sticks, saliva substitutes	Voluntary reflux phenomenon
3	Gas-chlorinated swimming pools	Subclinical regurgitation due to chronic gastritis associated with alcoholism
4	Occupational exposure to corrosive agents	Malabsorption syndrome
5		Chronic vomiting during pregnancy
6		Gastro-esophageal reflux

Table 1. Intrinsic and Extrinsic causes of dental erosion¹⁸

Identification of clinical lesions is imperative to confirmation of the diagnosis of the lesions. Clinically, the enamel exhibits a smooth, glazy appearance, without anatomical ridges or grooves. Lesions involving dentin exhibit a more dull appearance, and in posterior teeth, islands of restorations project toward the occlusal aspect. Incisal edges or anterior teeth and cusps of posterior teeth exhibit



a "cupped" appearance. Extrinsic factors most commonly affect the labial surfaces of anterior teeth and intrinsic factors like chronic vomiting disorders or silent regurgitation affect palatal aspects of maxillary anterior teeth.⁷ The persistent vomiting in anorexia nervosa and bulimia sufferers has the potential to lead to detrimental oral complications, including perimolysis, cervical caries, tooth sensitivity, impairment of mucous membrane, periodontopathy, glossodynia, xerostomia, enlargement of the parotid glands, halitosis, taste impairment, mouth ulcers, and sore throat.¹⁹⁻²¹ Examples of lesions are shown in Figure 2.



Figure 2. Clinical presentations of dental erosion



The diagnostic parameters for different causes of chemical erosion and their possible causes can be summarized in Figure 3 (Adapted from Verrett, 2001).⁷

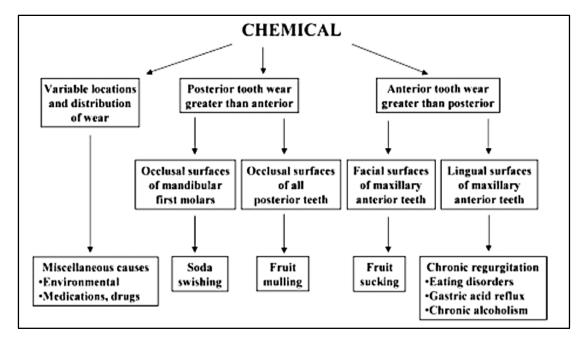


Figure 3. Diagnostic flowchart for dental erosion⁷

Presence of dental erosion is particularly detrimental to the dentition because it is known to potentiate other forms of mechanical wear by weakening the intermolecular bonds of the surface.^{5, 22} The gradient of mineral loss as the result of softening of eroded enamel thins the external layer of the mineral crystal, thus making it more susceptible to mechanical abrasion.^{23, 24} This finding is confirmed by numerous experimental studies. In an attempt to have an effective treatment strategy, various diagnostic indices have been developed in an attempt to quantitatively assess the amount of loss of tooth structure.²⁵ One such example is demonstrated in Table 2, adapted from Eccles and Jenkins.²⁶



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RATING	SEVERITY OF EROSION	
GRADE 0	No involvement of surface	
GRADE 1	Loss of enamel surface; no dentin involvement	
GRADE 2	Exposure of dentin on less than 1/3 of surface	
GRADE 3	Exposure of dentin on more than 1/3 of surface	

Table 2. Eccles and Jenkins Erosion Grading Scale²⁶

It is evident that dental wear seems to be a complex multifactorial process, requiring precise knowledge of the different processes contributing to the etiology to be able to comprehensively manage the conditions and rehabilitate the dentition.



1.3 Gastric reflux disease and Bulimia Nervosa

1.3.1 Introduction and clinical epidemiology

The term gastro-esophageal reflux disease (GERD) is used to describe the symptoms and changes of the esophageal mucosa that result from reflux of stomach contents into the esophagus.²⁷ Gastro-esophageal reflux disease is a fairly common medical condition. 15% individuals experience heartburn once a week; 7-10 % individuals experience heart burn once daily; 25-40% of Americans experience symptomatic GERD at some point; 45%-85% of women during pregnancy experience GERD or heart burn.²⁸⁻²⁹ Bulimia nervosa is an eating disorder characterized by self-induced vomiting³⁰ and is seen to impair physical well-being and psychosocial functioning³¹. Both medical conditions cause gastric acid to accumulate in the oral cavity for variable periods of time, leading to destructive effects on the dentition as described in previous sections. There are many open questions about etiology of GERD.²⁷ Severe exposure of teeth to acid is particularly common in subjects with eating disorders, as vomiting frequencies of 6–10 times per day are often reported from patients, many of whom vomit for several hours each day.³²⁻³³

1.3.2 Dental presentation and implications

The first association between GERD and dental erosion was reported in 1933.³⁴ It has been shown that there is a 31 times higher incidence of acid erosion of teeth in patients with GERD compared to controls.³⁵ A case-control study conducted by Johansson et al ³⁶, showed a significant correlation between eating disorders like



bulimia nervosa and dental erosion. This study also demonstrated erosion was significantly worse in individuals with longer duration of the disease. It has been well established, that presence of acid in the oral cavity has destructive effects on the dentition³⁷ due to its high erosive potential.³⁸ The overall dental considerations of GERD include dysgeusia, dental sensitivity, dental erosion, pulpitis, fibrosis and other mucosal changes.³⁹ The oral manifestations of GERD can be summarized in Table 3. (Adapted from Barron et al).²⁵

ORAL MANIFESTATIONS OF GERD
Burning mouth sensation
Tongue sensitivity
Nonspecific itching/burning
Tooth erosion
Dentinal hypersensitivity
Loss of vertical dimension of occlusion
Esthetic disfigurement

Table 3. Dental manifestations of GERD ²⁵



1.4 Dental ceramics

1.4.1 Evolution of ceramics in dentistry

Research on ceramics began as early as the 1700s⁴⁰ and materials have greatly evolved over the years. Currently available dental ceramics have been classified as predominantly glassy materials, particle-filled glass and polycrystalline ceramics.⁴¹⁻⁴⁴ Each class differs in mechanical properties and optical characteristics and hence have different indications for use. Feldspathic porcelain is a predominantly glassy ceramic which belongs to the family of aluminosilicate glass⁴³ and has an amorphous three-dimensional structure of atoms. This class of ceramics has the closest match to the optical properties of enamel and dentin.⁴¹ The first porcelain crown was introduced by Lang⁴⁵, and has been used with modifications in structure ever since as a veneering material for metal substructures, onlays, inlays and veneers⁴¹. In the 1960s, Mclean initiated the idea of adding aluminum oxide to feldspathic porcelain to improve its mechanical and physical properties.⁴⁶ Modifications included addition of fillers to overcome several deficiencies including low wear resistance and tensile strength, crack propagation, brittleness⁴⁷⁻⁴⁸ These alterations gave birth to the particle-filled glass ceramics which consisted of two or more distinct phases in their structure. Leucite, a crystalline material was the first filler to be added to feldspathic porcelain⁴²⁻⁴³ due to its favorable coefficient of thermal expansion, refractive index which matched that of feldspathic glass and ability to etch it.⁴¹ A higher concentration of leucite was used to obtain a leucite-reinforced glass ceramic (IPS Empress, Ivoclar Vivadent). The successor of this ceramic is a



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lithium disilicate reinforced ceramic (IPS e.max, Ivoclar Vivadent, Schaan, Liechtenstein) which is fabricated by a combination of heat-pressed and lost wax techniques.⁴⁸ It was developed to overcome the low flexural strength of its predecessor, followed by IPS e.max Press (Ivoclar Vivadent) which was introduced in 2005 as an improved press-ceramic material compared to IPS Empress with enhanced optical and physical properties achieved by a modified firing process.⁴⁹ The quest for metal-free restorations with mechanical properties similar to metal lead to the introduction of yttrium-stabilized zirconium oxide in dentistry. Zirconia, which is a crystalline dioxide of zirconium⁵⁰ has a tensile strength of 900-1200 MPa and compressive strength of about 2000 MPa⁵¹.

1.4.2 Desirable properties

Dental ceramics have evolved as the material of choice for restoration of dental hard tissues. They have demonstrated satisfactory clinical longevity ⁵² and esthetic characteristics. Ceramics are being increasingly used for metal-ceramic and all ceramic restorations such as inlays, onlays, veneers, full coverage crowns over teeth⁵³ and implants and implant abutments. They are known to have very favorable characteristics for use in dentistry such as biocompatibility, resistance to wear and excellent esthetics.⁵⁴



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1.4.3 Chemical durability of ceramics in an acidic environment

Ceramics are considered fairly chemically inert biomaterials⁵⁵, however, due to vast differences in the structure and composition of currently available ceramics, this inertness may not be generalized. ⁵⁴ Ceramics used in dentistry are considered chemically stable⁴¹, however there are concerns about degradation of ceramics in the presence of low or high pH in the oral cavity.⁵⁶ Newton in 1985 defined durability as the resistance to the attack of glass by water and aqueous solutions.⁵⁵ The possible chemical degradation of ceramics may have effects such as increased abrasion of opposing dental structures, the release of radioactive components, and increased plaque adhesion as a result of wear and chemical attack.⁵⁶ The effect of different acids on ceramic surfaces have been previously studied.⁵⁴⁻⁵⁶ It has been seen that on immersion of ceramics in acidic agents for 168 hours, there was in increase in the surface roughness of the ceramics. ⁵⁴ There are many implications of increased surface roughness on the mechanical behavior of ceramics,⁵⁷ and corrosion can affect the fracture strength of these materials.⁵⁷-58 Surface roughness may potentially affect the strength of the ceramics by

possibly altering surface flaws.58

Since ceramics are the frontline materials of choice for prosthetic rehabilitations of patients with severe wear, the continued presence of the systemic condition raises concerns about longevity of these materials in patients with chronic GERD. Matsou et al (2011) compared the roughness of three ceramics before and after exposure to simulated vomit solution (SVS) with a pH of 3.8 using a novel peristaltic pump to simulate periodic acid exposure and concluded that no significant surface



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alterations occurred on ceramics on exposure to stimulated gastric acid.⁵⁹ It has been reported that there is accelerated abrasive wear of CAD-CAM machinable ceramics and veneering ceramics in exposure to acids.⁶⁰ A recent study evaluated the effect of gastric acid on monolithic zirconia and found that monolithic zirconia materials show some surface alterations in an acidic environment with minimum effect on their optical properties.⁶¹ There is a growing interest in the research community to test the durability of ceramics in order to predictably meet patient needs. While ceramics have evolved to demonstrate superior optical and functional properties over the last few decades, the question arises about whether these properties are affected by acidic oral environments.

This study is an attempt to test and quantify the actual effect of changes in surfaces of ceramics when exposed to acid on the functional strength and optical properties. The clinician needs to know the clinical significance of these changes to make informed choices for his/her patients. There is limited literature available to examine the effect of gastric acid induced surface changes of dental ceramics. The current study aims to provide a better understanding to make informed choices for material selection for successfully and predictably rehabilitating a patient with damage caused by eating disorders or GERD.



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1.5 Purpose, Specific Aims and Hypothesis

1.5.1. Purpose

The objective of this research study is to understand the behavior of dental ceramics in an acidic environment that may exist in patients with GERD or bulimia nervosa. Few existing studies have examined the effect of gastric acid induced surface changes of dental ceramics. Based on previous studies, it is assumed that the exposure of ceramics to gastric acid (pH <2) causes alterations in their surface texture. However, less is known about the impact of these surface changes on the physical properties of ceramics. The three commercially available dental ceramics that will be used for the study are, feldspathic porcelain, lithium disilicate glass ceramic and monolithic zirconia.

The current study aims to test the hypothesis that exposure to artificial gastric acid combined with toothbrush abrasion will negatively impact the properties of dental ceramics

1.5.2 Specific Aims

- 1) To determine the effect of **abrasive wear** (using toothbrush abrasion) of ceramics after exposure to artificial gastric acid.
- 2) To examine the changes in the optical characteristics (color, translucency parameter and surface gloss) of ceramics due to artificial gastric acid and toothbrush abrasion induced surface changes.
- 3) To examine the **changes in functional strength** (biaxial flexural strength) of ceramics due to gastric acid and toothbrush abrasion induced surface changes.



1.5.3 Hypothesis

H^o- Exposure to gastric acid and toothbrush abrasion will negatively impact the functional strength (biaxial flexural strength) of feldspathic porcelain, lithium disilicate and monolithic zirconia ceramics.

H^o- Gastric acid induced surface change will negatively impact the optical characteristics (color, translucency parameter and surface gloss) of feldspathic porcelain, lithium disilicate and monolithic zirconia ceramics.



2.1 Study

2.1.1 Study protocol

The study protocol was adopted from previous studies on human dentine that studied the cyclic demineralization and remineralization upon exposure to gastric acid and toothbrush abrasion, in an attempt to simulate oral conditions.⁶²⁻⁶³ The protocol was modified to incorporate more variables to test the proposed hypothesis. The protocol for the treatment of specimens with acid and toothbrushing was defined (Table 4) prior to dividing specimens into groups.

PROTOCOL FOR GASTRIC ACID TREATMENT	PROTOCOL FOR TOOTH-BRUSH ABRASION TREATMENT
 Immersion in stimulated artificial gastric acid (pH=2) for 2 minutes Rinsing with distilled water with 2 minutes Storage in distilled water for 2 hours at 37°C 	 Brushing with a standardized tooth-brush and dentifrice slurry using an ISO/ADA design brushing machine Brushing force = 4N
Procedure repeated 6 times a day x 9 days to simulate 108 hours of exposure	Procedure repeated for 100 cycles per day for 9 days

Table 4. Specimen treatment protocol



2.1.2 Material selection

Three types of dental ceramics were selected for this study, as representative of currently used dental ceramics in a clinical setting, (Table 5). The materials were divided into four groups based on the treatments that they received, summarized in Table 6.

CERAMIC TYPE	PRODUCT	MANUFACTURER
Feldspathic porcelain	VITA VMK 95	VITA, Zahnfabrik
Lithium disilicate glass ceramic	IPS e. max	Ivoclar Vivadent, USA
Zirconium oxide	Monolithic zirconia	Dentsply Cercon, USA

Table 5. Materials used for the study

GROUP	A (n=8)	B (n=8)	C (n=8)	D (n=8)
TREATMENT OF SPECIMENS	No exposure	Exposure to acid	Exposure to tooth brush abrasion	Exposure to acid and tooth brush abrasion

Table 6. Gi	roups based	on treatment
-------------	-------------	--------------

2.1.3 Support Acknowledgement

This study was awarded a grant by the Health Professions Division at

Nova Southeastern University (#335954).



2.2 Sample Size Estimate

A power estimate indicated an adequate number of samples per group (alpha = 0.05, power=80%, standardized effect size of 0.50).

2.3 Sample Preparation

Three types of dental ceramics were obtained from the manufacturer (Oral Arts Dental Laboratory, Huntsville, Alabama) as pre-sintered milled rods (diameter of 10mm) using Computer-Aided Design/Computer-Aided Manufacturing (CAD/CAM) technology (Figure 4.a). The rods were sectioned (Figure 4.b) using a precision saw (IsoMet[®] 1000 Precision Cutter; Buehler GmbH, Düsseldorf, Germany) at 400 rotations/minute (rpm) to obtain discs with dimensions of 10 x 1mm (Figure 4.c). A total of forty-eight specimens were obtained from each material, in order to have eight specimens per group. The specimens were polished under running water (Metaserv[®] 2000 Grinder Polisher; Buehler GmbH, Düsseldorf, Germany) at 300 rotations/minute (rpm) using 400, 600 and 800-grit silicon carbide paper (3M ESPE, St. Paul, Minn) for 7 minutes each (Figure 5.a-b). The platen size used was 250 mm. The specimens were then ultrasonically cleaned (PC3; L&R Mfg Co, Kearny, NJ) in distilled water for 15 minutes. Subsequently, the specimens were subjected to heat treatment in a programmed oven (Programat[®] CS; Ivoclar Vivadent Inc., Figure 5.c), as per recommendations of the manufacturer. Finally, the specimens were divided into 8 per group and immersed in distilled water at 37 °C (Thermo Scientific Heratherm General Incubator) for 24 hours, in preparation for exposure.



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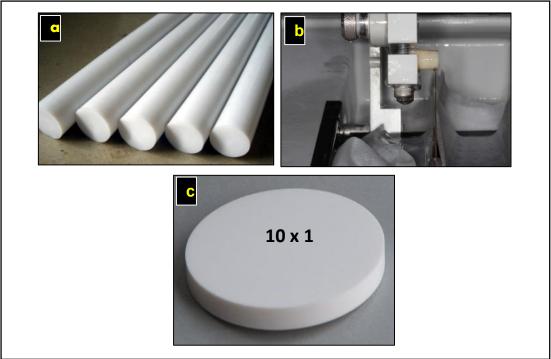


Figure 4.a-c. Sectioning of specimens

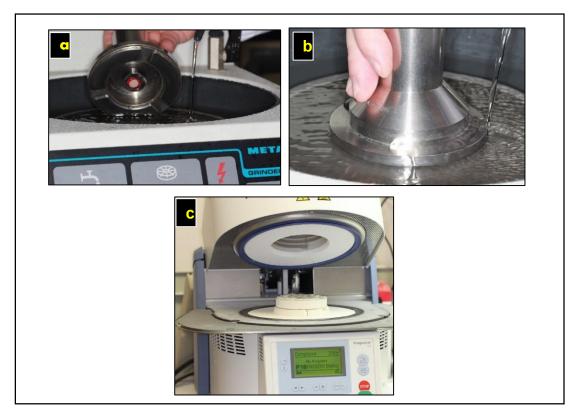


Figure 5.a-c. Preparation of specimens



2.4 Treatment of specimens

2.4.1 Acid treatment

For each treatment time, 50 ml of stimulated artificial gastric acid was prepared fresh prior to the exposure of specimens using the following formula recommended by the manufacturer:

50 ml of 0.2% (w/v) Sodium chloride in 0.7% (v/v) Hydrochloric acid (Ricca Chemical Company, Arlington, TX; Cat # 7108-16) mixed with 0.16g of pepsin powder (Fisher Chemical[™]; Code S25459, Figure 6.a).

The pH of the solution was maintained at 2.0. 50% NaOH was used as a buffer to maintain the pH at 2.0, using a pH measuring probe. (Accumet[™] AB15 Basic pH meter; Fisher Scientific[™], Figure 6.b).



Figure 6.a-b. Preparation of artificial stimulated gastric acid



Glass beakers were used to immerse the specimens in the acid, the unexposed side was marked for identification. The specimens were immersed in the prepared solution for 2 minutes, followed by rinsing with distilled water for 2 minutes. (Figure 7.a-b). Following the acid treatment, the specimens were stored in distilled water at 37 °C. The procedure was repeated for Groups B and D for 6 times / day for 9 days. The specimens from Group D were then cleaned in preparation for tooth-brush abrasion treatment.

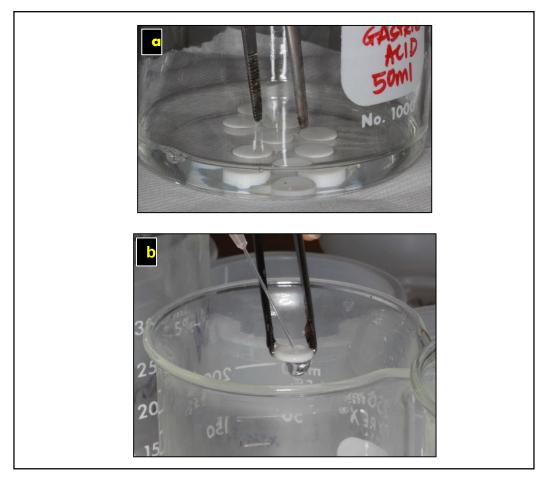


Figure 7.a-b. Acid treatment of the specimens



2.4.2 Tooth brush abrasion

Groups B and D were subjected to tooth brush abrasion using a cross brushing machine as per design specifications of the American Dental Association (V8 Cross Brushing Machine, Sabri Dental Enterprises, Inc.; ISO/DIS standard specification no. 11609, Figure 9.a). Brush heads of standard toothbrushes (Acclean Action Plus, Henry Schein^{®,} Figure 8.a) which follow ADA design specifications were mounted on the machine for every time of exposure. A dentifrice slurry was prepared using a non-abrasive standard dentifrice (Colgate[®] Cavity Protection Toothpaste, Figure 8.b) by diluting the dentifrice in distilled water with a 1:2 ratio (26g of dentifrice : 52ml of distilled water, Figure 9.c). A fresh slurry was made for each day of the experiment. The specimens were cleaned and mounted on resin blocks (Figure 9.b) the machine set at a frequency of 100 cycles / minute for everyday of brushing for 9 days (Figures 9.d-e). The specimens that received both acid and brushing treatments received the acid exposure (6 times/day) first, followed by 100 cycles/minute of brushing.

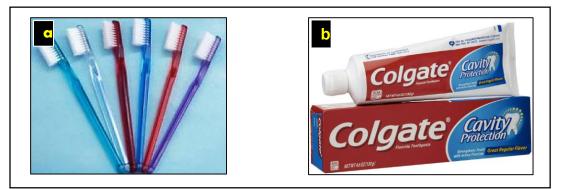


Figure 8.a-b. Tooth brushes and dentifrice as per ADA specifications



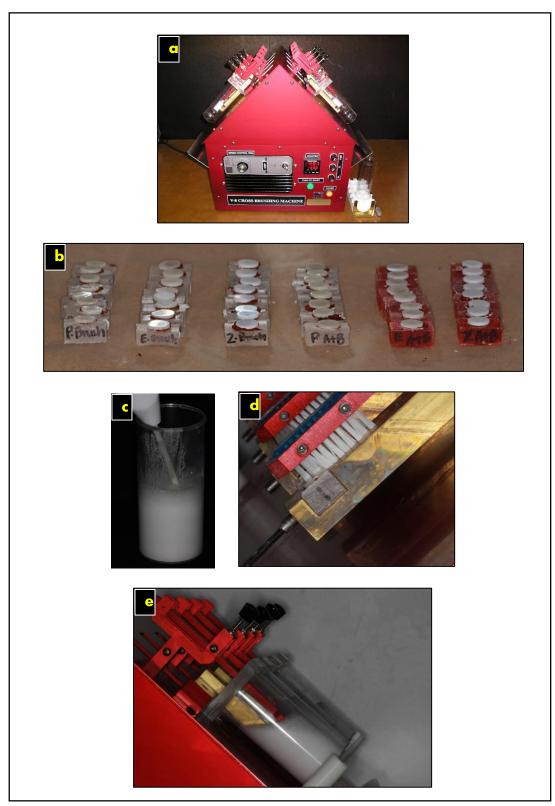


Figure 9 a-e. Tooth brush abrasion of the specimens using the V8 Cross Brushing machine



2.5 Morphological assessment of surface topography

Specimens from each group were randomly selected for morphological assessment using a scanning electron microscope. The selected specimens were thoroughly cleaned, rinsed with distilled water for 5 minutes, dried and fixed onto an aluminum mount and dried. Subsequently, the specimens were lightly sputtered with a gold-palladium alloy (SPI-Module sputter, SPI Supplies, West Chester, PA, USA). The surface topography of the specimens were then using a FEI Quanta 200 Scanning Electron Microscope (FEI, Hillsboro, OR). SEM micrographs of randomly selected specimens of all groups were taken at a 500×, 1000× and 5000× original magnification.

2.6 Testing of specimens

2.6.1 Optical testing

2.6.1.1 Surface gloss measurement

A gloss meter (Novo-Curve[™] Glossmeter, Rhopoint[™] Instruments Ltd, Figure 10.a) was used to measure specular reflection gloss of all groups of ceramics. The machine was calibrated as per the manufacturer's recommendations prior to making any measurements. The specimens were cleaned and dried and placed on the instrument for measurement. The surface gloss measurement was noted (in degrees) as the amount of reflected light at an equal but opposite angle to the projecting beam of light from the specimen. (Figure 10.b).



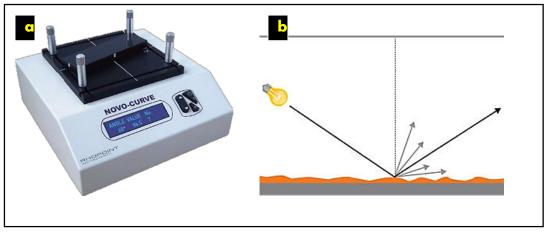


Figure 10.a-b. Surface gloss measurement using glossmeter

2.6.1.2 Color and translucency parameter measurement

A digital spectrophotometer (Gregtag Macbeth[®] Color-Eye 7000 A, Figure 11) was used to record the CIELAB (L*, a*, b*) coordinates of all the ceramic samples. The color measurements were made relative to the CIE standard illuminant D65 (as defined by the International Commission on Illumination) which corresponds to average daylight (including ultraviolet wavelength region with a correlated color temperature of 6504 K). The Specular Component Excluded (SCE) geometry was determined according to the CIE $L^*a^*b^*$ color scale using standard illuminant D65 over a black background, where (SCE) refers to specular component excluded, L^* refers to the lightness, a^* to redness to greenness, and b^* to yellowness to blueness. Additionally, for the translucency parameter (TP) measurements, a standard white background was used. Calibration of the spectrophotometer was executed as per the manufacturer's recommendations before measurement of the specimens.





Figure 11. Spectrophotometer

Color calculations were made using the CIE $L^*a^*b^*$ readings in the following formula:

$$\Delta E = (L^{*2} + a^{*2} + b^{*2})^{1/2}$$

The TP of each specimen was obtained by calculating the color difference between the specimen against the white background (*w*) and against the black background (*b*) using the following equation:

$$TP = \{(Lb* - Lw*)^{2} + (ab* - aw*)^{2} + (bb* - bw*)^{2}\}^{1/2}$$



2.6.2 Surface roughness measurement

A stylus (contact) profilometer (Veeco DEKTAK 150 Profilometer, Bruker Corporation, Figure 12) was used to measure surface roughness values of all the ceramic specimens. The specimens were cleaned, dried and stabilized on a mount for measurements. The profilometer was calibrated as per the recommendations of the manufacturer before measurements of each group. Three roughness measurements (Ra in µm) were made for each specimen. A diamond stylus (NHT-6) of 2 µm radius and 90° stylus angle was crossed over at a constant speed across each of the finished ceramic specimens with a force of 0.7 N. All the measurements were made as close as possible to the center of the specimen as possible. Three measurements were made for each specimen and the mean was calculated to record the surface features. The Ra value obtained represented the mean value for a surface that has been traced by the profilometer.⁶⁴⁻⁶⁵ A lower Ra value is indicative of a smoother surface.⁶⁶

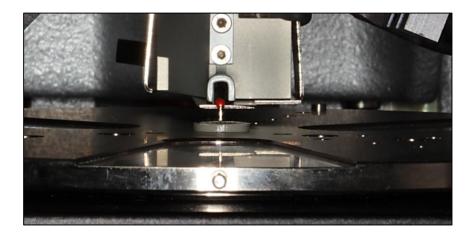


Figure 12. Stylus profilometer



2.6.3 Biaxial flexural strength measurement

Specimens from all the groups were subjected to strength measurements. The specimens were cleaned and dried prior to testing. The piston-on-three-ball test as per ASTM Standard F394-78⁶⁷ was used to determine the biaxial flexural strength (BFS). The diameter of the mount was 10 mm to accommodate the 10 mm diameter ceramic specimens. This dimension was used for the specimens to more closely simulate dental restorations and have a more realistic depiction of strength values.

The thickness of the specimen center was measured with a digital micrometer (Digital Caliper-Deluxe Model, RSR Electronics, Inc) before testing. Three steel spheres (1.6 mm diameter) placed on a circle (8 mm diameter) arranged 120° apart were used to center and support the disk specimens.

The load was applied to the specimen center by a right circular cylinder of hardened steel having a diameter of 0.72 mm with the flat end perpendicular to the axis at a crosshead speed of 1 mm/min until fracture. The testing was performed at room conditions using a universal testing machine (Instron 8871, Instron[®], Figure 13) The maximum tensile stress (MPa), which corresponded to the biaxial flexure strength, was calculated according to the equation suggested by the test standard (ASTM F394-78) as follows:

$S = -0.2387P(X - Y)/d^2$

- S- Maximum tensile stress (MPa)
- P-Load at fracture (N)
- d- Specimen thickness (mm) at fracture origin



 $X = (1+v) \ln (B/C)^2 + [(1-v)/2] (B/C)^2$

$$Y = (1+v) [1 + ln (A/C)^{2}] + (1 - v) (A/C)^{2}$$

v- Poisson's ratio

- A- Radius of the support circle (mm)
- B- Radius of the tip of the piston (mm)
- C- Radius of the specimen (mm).

The Poisson's ratio was assumed to be 0.25 for feldspathic porcelain and IPS e.max ceramics and 0.30 for monolithic zirconia.



Figure 13. Instron 8871 universal testing machine



2.7 Statistical analysis

Descriptive statistics were used on all the raw data obtained from different testing. A one-way between groups analysis of variance (ANOVA) was conducted to compare the effect of different treatments (Acid only, Brush only, Acid + Brush) on optical characteristics (surface gloss, color and translucency) and mechanical behavior (biaxial flexural strength) on the three types of ceramics. A Tukey HSD post hoc test was used for all post-hoc comparisons.



3.1 Surface gloss results

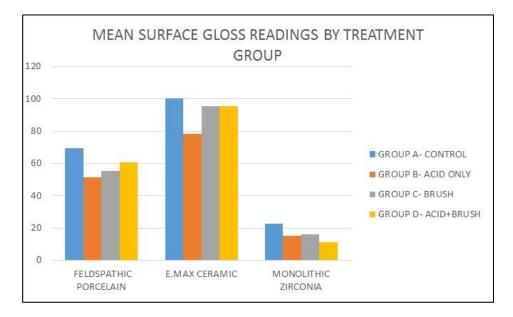
A one-way between groups ANOVA was conducted to compare the effect of acid environment and tooth brushing on the surface gloss in three types of ceramics. A TukeyHSD post hoc test was used for all post-hoc comparisons. Descriptive statistics are presented in Table 7. Results obtained are as follows:

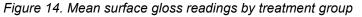
- There was no significant effect of acid and brushing treatment on gloss at the p < 0.05 level for IPS e.max ceramics [F (3, 28) = 2.69, p = 0.065].
- There was a significant effect of acid and brushing treatment on gloss at the *p* < 0.05 level for feldspathic porcelain [F (3, 28) = 4.25, *p* = 0.013, η²= 0.31]. We see a significant difference between the control group A vs. acid only group B [difference = 18.21, 95% CI (3.45, 32.96), *p* = 0.011].
- There was no significant effect of acid and brushing treatment on gloss at the p < 0.05 level for zirconia ceramic [F (3, 28) = 1.91, p = 0.150].



GROUP	PARAMETERS	IPS e.max	PORCELAIN	ZIRCONIA
GROUP A- CONTROL	Ν	8	8	8
	Mean	22.81	69.54	100.13
	SD	13.50	11.63	12.65
	Min	11.30	46.90	78.80
	Max	53.50	79.50	120.80
GROUP B- ACID ONLY	Ν	8	8	8
	Mean	15.08	51.33	78.31
	SD	6.23	9.36	31.34
	Min	7.50	35.80	7.10
	Max	24.00	62.00	98.90
GROUP C- BRUSH	Ν	8	8	8
	Mean	16.05	55.30	95.39
	SD	5.45	13.99	7.74
	Min	6.10	37.40	83.50
	Max	22.50	79.50	105.30
GROUP D- ACID+BRUSH	Ν	8	8	8
	Mean	10.99	60.60	95.68
	SD	5.93	6.98	18.61
	Min	3.60	50.30	60.90
	Max	22.90	70.70	116.30

Table 7. Descriptive Statistics for Surface gloss







3.2 Color results

A one-way between groups ANOVA was conducted to compare the effect of acid environment on color in three types of ceramics. A TukeyHSD post hoc test was used for all post-hoc comparisons. Descriptive statistics are presented in Table 8. Results are as follows:

- There was a significant effect of acid and brushing treatment on color at the *p* < 0.05 level for IPS e.max [F (3, 28) = 3.09, p = 0.043, η²= 0.25]. We see a significant difference between the control group A vs. brush only group C [difference = 3.43, 95% CI (0.19, 5.67), *p* = 0.035].
- There was no significant effect of acid and brushing treatment on color at the p
 < 0.05 level for Porcelain [F (3, 28) = 1.77, p = 0.176].
- There was no significant effect of acid and brushing treatment on color at the p
 < 0.05 level for Zirconia [F (3, 28) = 1.94, p = 0.255].



GROUP	PARAMETER	IPS e.max	PORCELAIN	ZIRCONIA
GROUP A- CONTROL	Ν	8	8	8
	Mean	56.82	58.13	72.21
	SD	1.43	3.04	1.85
	Min	55.13	50.93	69.40
	Max	59.58	60.66	74.68
GROUP B- ACID ONLY	Ν	8	8	8
	Mean	57.76	58.79	73.40
	SD	1.08	0.97	0.70
	Min	56.45	57.38	72.70
	Max	59.47	60.65	74.66
GROUP C- BRUSH	Ν	8	8	8
	Mean	60.36	59.15	72.81
	SD	4.19	1.11	0.93
	Min	56.83	58.04	71.28
	Max	69.76	61.59	74.23
GROUP D- ACID+BRUSH	Ν	8	8	8
	Mean	57.97	60.05	72.99
	SD	1.35	0.46	0.82
	Min	56.12	59.27	71.68
	Max	59.97	60.56	74.21

Table 8. Descriptive Statistics for Color

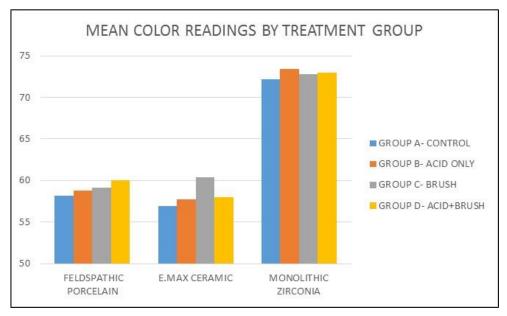


Figure 15. Mean color readings by treatment group



3.3 Translucency parameter results

A one-way between groups ANOVA was conducted to compare the effect of acid and brushing treatment on the translucency parameter on three types of ceramics. A TukeyHSD post hoc test was used for all post-hoc comparisons. Descriptive statistics are presented in Table 9. Results are as follows:

- There was no significant effect of acid and brushing treatment on translucency at the *p* < 0.05 level for IPS e.max [F (3, 28) = 0.48, *p* = 0.700].
- There was no significant effect of acid and brushing treatment on translucency at the *p* < 0.05 level for Porcelain [F (3, 28) = 0.18, p = 0.9119].
- There was no significant effect of acid and brushing treatment on translucency at the *p* < 0.05 level for Zirconia [F (3, 28) = 0.50, p = 0.685].



GROUP	PARAMETER	IPS e.max	PORCELAIN	ZIRCONIA
	N	8	8	8
GROUP A- CONTROL	Max	10.22	12.03	4.44
	Mean	8.49	7.37	3.92
	SD	1.01	0.58	0.58
	Min	6.34	6.76	2.76
GROUP B- ACID ONLY	Mean	8.49	7.37	3.92
	SD	1.01	0.58	0.58
	Min	6.34	6.76	2.76
	Max	9.69	8.52	4.63
GROUP C- BRUSH ONLY	Mean	8.13	7.59	3.29
	SD	1.10	0.97	1.33
	Min	6.69	5.82	0.77
	Max	9.72	8.91	4.61
GROUP D- ACID+BRUSH	Mean	7.91	7.42	3.49
	SD	1.18	0.54	1.11
	Min	6.05	6.94	1.36
	Max	9.12	8.70	4.35

Table 9. Descriptive statistics for translucency parameter

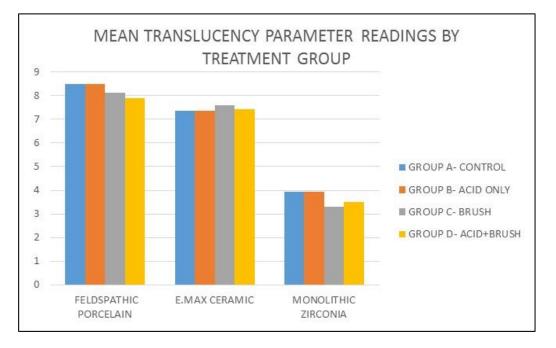


Figure 16. Mean translucency parameter readings by treatment group



3.4 Surface roughness results

A one-way between groups ANOVA was conducted to compare the effect of acid and brushing treatment on surface roughness in three types of ceramics. A TukeyHSD post hoc test was used for all post-hoc comparisons. Descriptive statistics are presented in Table 10. Results are as follows:

- There was a significant effect of acid and brushing treatment on surface roughness at the *p* < 0.05 level for IPS e.max [F (3, 92) = 3.44, p = 0.020, η²= 0.11]. We see a significant difference between the control group A vs. acid + brush group D [difference = 0.17, 95% CI(0.01,0.33)]
- There was a significant effect of acid and brushing treatment on surface roughness at the *p* < 0.05 level for Porcelain [F (3, 28) = 15.52, *p* < 0.000, η²= 0.27].]. We see the following significant differences:
 - Brush vs. Acid [difference = 0.59, 95% CI (0.12, 1.05), *p* = 0.007].
 - Control vs. Acid [difference = 1.18, 95% CI (0.71, 1.64), *p* < 0.000].
 - Brush vs. Acid+Brush [difference = 0.80, 95% CI (0.34, 1.26), *p* < 0.000].
 - Control vs. Brush [difference = 0.59, 95% CI (0.13, 1.05), *p* < 0.006].
- There was no significant effect of acid and brushing treatment on surface roughness at the *p* < 0.05 level for Zirconia [F (3, 28) = 11.19, *p* < 0.000].
 - Control vs. Acid [difference = 0.19, 95% CI (0.05, 0.33), *p* = 0.002].
 - Control vs. Acid+Brush [difference = 0.28, 95% CI (0.14, 0.41), *p* < 0.000].
 - Control vs. Brush [difference = 0.23, 95% CI (0.09, 0.37), *p* < 0.000].



GROUP	PARAMETER	IPS e.max	PORCELAIN	ZIRCONIA
GROUP A- CONTROL	Ν	24	24	24
	Mean	0.36	0.47	0.19
	SD	0.08	0.25	0.16
	Min	0.22	0.21	0.07
	Max	0.51	1.13	0.69
GROUP B- ACID ONLY	Ν	24	24	24
	Mean	0.43	1.66	0.39
	SD	0.17	0.83	0.13
	Min	0.23	0.61	0.21
	Max	1.11	4.21	0.70
GROUP C- BRUSH	N	24	24	24
	Mean	0.52	1.06	0.43
	SD	0.26	0.60	0.21
	Min	0.24	0.31	0.20
	Max	1.24	2.49	1.20
GROUP D- ACID+BRUSH	Ν	24	24	24
	Mean	0.53	1.27	0.47
	SD	0.29	0.63	0.21
	Min	0.21	0.43	0.22
	Max	1.39	2.40	1.01

Table 10.	Descriptive	statistics	for surface	roughness
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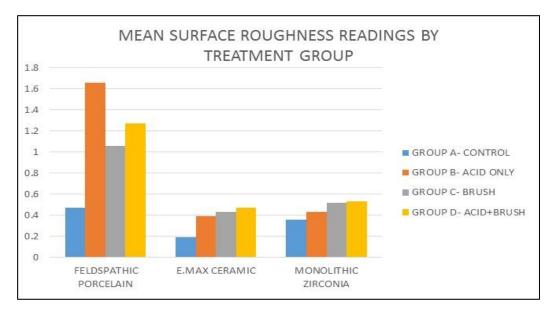


Figure 17. Mean surface roughness readings by treatment group



3.5 Biaxial Flexural Strength results

A one-way between groups ANOVA was conducted to compare the effect of acid and brushing treatment on the biaxial flexural strength in three types of ceramics. A TukeyHSD post hoc test was used for all post-hoc comparisons. Descriptive statistics are presented in Table 11. Results are as follows:

- There was no significant effect of acid and brushing treatment on flexural strength at the *p* < 0.05 level for IPS e.max [F (3, 28) = 1.59, p = 0.214].
- There was no significant effect of acid and brushing treatment on flexural strength at the *p* < 0.05 level for Porcelain [F (3, 28) = 0.05, *p* = 0.983].
- There was no significant effect of acid and brushing treatment on flexural strength at the *p* < 0.05 level for Zirconia [F (3, 28) = 1.05, *p* = 0.386].



GROUP	PARAMETER	IPS e.max	PORCELAIN	ZIRCONIA
GROUP A- CONTROL	Ν	8	8	8
	Mean	297.16	120.93	1093.35
	SD	51.81	56.93	139.91
	Min	209.38	0.00	886.33
	Max	369.60	177.04	1337.47
GROUP B- ACID	Ν	8	8	8
	Mean	342.75	125.10	1057.57
	SD	64.38	32.55	165.52
	Min	265.49	80.97	790.07
	Max	449.82	174.19	1323.90
GROUP C- BRUSH	Ν	8	8	8
	Mean	293.76	120.61	1086.24
	SD	81.63	26.39	96.48
	Min	142.92	87.39	938.72
	Max	429.70	165.64	1207.38
GROUP D- ACID+BRUSH	Ν	8	8	8
	Mean	342.54	117.44	971.97
	SD	38.89	31.75	195.99
	Min	278.96	53.60	713.32
	Max	391.38	146.12	1369.80

Table 11. Descriptive statistics for flexural strength	Table 11.	Descriptive	statistics for	or flexural	strength
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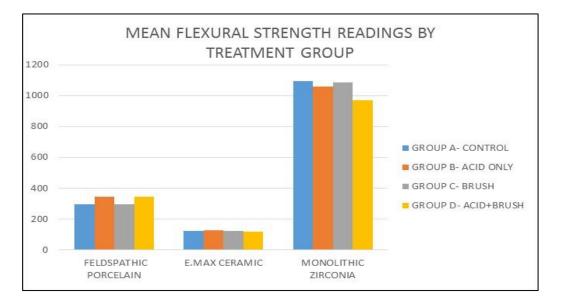


Figure 18. Mean flexural strength readings by treatment group



CHAPTER 4: DISCUSSION

This study was conducted to compare the effects of gastric acid induced surface changes, with and without tooth brush abrasion on the optical characteristics and mechanical properties of currently used dental ceramics. Three types of dental ceramics were selected for this study, as representative of currently used dental ceramics in a clinical setting, (Table 5). The ceramics were prepared as 10 x 1mm discs, the size is assumed to more closely mimic dimensions of dental ceramic restorations. Each specimen received the same timed treatment of polishing and heat treatment (as recommended by the manufacturer) to ensure uniformity. The materials were divided into four groups based on the treatments that they received, summarized in Table 6. The protocol was adopted from previous studies and modified to test the proposed hypothesis. Regimens for gastric acid treatment and tooth brush abrasion (summarized in Table 5) were developed and applied to the respective groups. All specimens were stored in distilled water at 37°C in an incubator (Thermo Scientific Heratherm General Incubator). Specimens from Group A (Control) received no treatments. Following the 9 day regime of gastric acid treatment and/or toothbrush abrasion, the specimens were subjected to optical and mechanical testing using standardized testing protocols. Following testing, statistical analysis was performed to analyze the results and to determine whether the proposed hypothesis was supported or rejected, discussed in the forthcoming sections.



4.1 Study aftermath

4.1.1 Changes in functional strength

The results of the study have rejected the first null hypothesis proposed, i.e. exposure to gastric acid and toothbrush abrasion will negatively impact the functional strength (biaxial flexural strength) of feldspathic porcelain, lithium disilicate and monolithic zirconia ceramics. It was observed in the present study, that the biaxial flexural strength comparisons between groups for all three ceramics used were not statistically significant, (figure 18) indicating that the functional strength of ceramics was not negatively impacted by the changes induced by the acid treatment.

The International Standard Organization (ISO)⁶⁸ recommends biaxial flexural strength testing as a reliable method to study the functional strength of brittle materials⁶⁹ as the maximum tensile stress occurs within the central loading zone and edge failures are removed.⁷⁰ The optimum strength of ceramic materials depends on the fabrication procedures and presence and location of flaws.⁷¹ The biaxial flexural strength test used in this study is dependent on the surface finish of the specimens⁷² and for this purpose, the surface finish, thickness and diameter of the specimens was controlled and uniform in the current study.

Functional strength is a significantly desirable property of dental ceramics, especially because increasing numbers of ceramic restorations are being used in the posterior region, where occlusal forces range from 150 to 665 N.⁷³⁻⁷⁴ Dental ceramics are brittle materials with high elastic moduli and are more sensitive to



tensile stresses as compared to compressive stresses produced during mastication.⁷⁵ The paradigm shift towards metal-free restorations in clinical dentistry has given birth to a host of ceramic materials with excellent esthetic and mechanical properties. Although a good deal of research in recent years has focused on enhancing the mechanical properties of ceramics to meet clinical objectives, there are no studies, to the current knowledge of the authors that have examined the effect of a corrosive environment on the functional strength of these materials. It has been seen that on immersion of ceramics in acidic agents for 168 hours, there was in increase in the surface roughness of the ceramics.⁵⁴ There are many implications of increased surface roughness on the mechanical behavior of ceramics⁵⁷ and corrosion can affect the fracture strength of these materials.⁵⁷⁻⁵⁸ Surface roughness may potentially impact the strength of ceramics by possibly altering surface flaws.⁵⁸ It has been demonstrated that certain surface treatments which increase the surface irregularities of ceramics⁷⁶ might pose a risk of future failure and negatively impact the fracture resistance of the ceramic restorations.⁷⁷

In the current study, although surface alterations were observed upon acid treatment on the feldspathic porcelain and IPS e.max ceramic specimens, the strength values between the four groups were not statistically significant. Some reduction in strength was noted after acid and brushing treatment for zirconia specimens, however the values were not meaningful. These findings may be attributed to a relatively small sample size per group. Although this finding is favorable, results could differ under dynamic loading conditions. This is because



dynamic loading lowers the strength of the material, and presence of water and temperature changes can have an effect on strength values.⁷⁸

4.1.2 Changes in optical characteristics

The results of the current study have supported the second null hypothesis that gastric acid induced surface change will negatively impact the color of IPS e.max ceramics and surface gloss of feldspathic porcelain. Additionally, results of the current study have rejected the second null hypothesis that gastric acid induced surface change will negatively impact the optical characteristics (color, translucency parameter and surface gloss) of monolithic zirconia ceramics.

The demand for esthetics is only rising, as patients desire "natural" looking restorations. Restorative dentistry, the world over, is on the lookout for the most esthetic ceramic material to satisfy increasing patient demands. One of the most challenging tasks for a restorative dentist is to esthetically match natural teeth and surrounding tissues, due to so many variations in color and shape of natural teeth.⁷⁹ As described by Albert Munsell in 1921, the color of an object is a three dimensional phenomenon, described as hue, value (brightness), and chroma (saturation).⁷⁹ In addition to these dimensions, the perceived shade of an object is also influenced by phenomenon including fluorescence, opalescence, texture, shape and refractive index. Certain opacifiers and oxides have been incorporated by manufacturers in currently used ceramics to aid in adding depth for matching desirable shades. In general, ceramics with increased proportion of glassy matrix



like feldspathic porcelain and IPS e.max ceramics have superior esthetic properties compared to more opaque ceramics like zirconia. In keeping with technological advances and clinical goals, compositions of currently used ceramics are evolving every day and while most ceramic materials today perform satisfactorily, their longevity in an acidic environment is a matter of concern and remains to be studied. When restoring worn dentitions with ceramic materials in patients with gastric reflux or eating disorders that cause accumulation of highly acidic fluids (pH=1-2) in the oral cavity, the clinicians should be mindful of the effect of these acids on the various components of esthetics.

Several *in-vitro* investigations have demonstrated that exposure of ceramics to corrosive acids affect the surface roughness of ceramic restorations. Figure 17 shows the differences in the surface roughness values (Ra in μ m) as measured by a stylus profilometer after various treatments on the three ceramics in the current study. All three ceramics displayed an increase in surface roughness values. The felspathic porcelain group demonstrated the most significant difference between groups, as follows:

- Brush vs. Acid [difference = 0.59, 95% CI (0.12, 1.05), *p* = 0.007].
- Control vs. Acid [difference = 1.18, 95% CI (0.71, 1.64), *p* < 0.000].
- Brush vs. Acid+Brush [difference = 0.80, 95% CI (0.34, 1.26), *p* < 0.000].
- Control vs. Brush [difference = 0.59, 95% CI (0.13, 1.05), *p* < 0.006].



The IPS e.max samples demonstrated a significant difference in surface roughness as well, especially between control group A vs. acid + brush group D [difference = 0.17, 95% CI (0.01, 0.33)].

This increase in surface roughness of ceramics has the potential to affect light reflection and color perception co-ordinates due to changes in surface topography. A recent *in-vitro* study by Sulaiman *et al* demonstrated that translucency parameter significantly increased (p<0.05) for partially stabilized zirconia, translucent zirconia and IPS e.max ceramics, and the surface gloss significantly increased (p<0.05) for IPS e.max, partially stabilized, and fully stabilized zirconia when immersed in artificial gastric acid for 96h at 37 °C. For our study, a standard color specification system delevoped by the Commission Internationale de l'Eclairage (CIE, International Commission on Illumination) was used. In this system X, Y and Z stimulus values are obtained from the combination of the object's spectral power distribution and the spectral power distribution of the selected illuminant.

These values can be transformed to L*, a* and b* values where L* is a measure of lightness similar to Value (V) in the Munsell system and the a* and b* values represent positions on a red/green and yellow/blue axis, respectively. ⁸⁰ The changes in optical characteristics in the present study are further elaborated.



4.1.2.1 Translucency parameter

Translucency is the relative amount of light transmitted through the material.⁸¹ The two common parameters used to measure translucency of dental materials are contrast ratio (CR) and translucency parameter (TP). TP is the color difference between a material of uniform thickness over black and a white background directly corresponding to a common visual assessment of translucency.⁸² The CIE recommends calculating color difference (Δ E) based on CIELAB color parameters. The CIELAB is a non-linear transformation of the tristimulus space to agree with Munsell spacing and has been largely used to compare translucency among materials.⁸³ A digital spectrophotometer (Gregtag Macbeth[®] Color-Eye 7000 A) was used to record the CIELAB (L*, a*, b*) coordinates of all the ceramic samples. The TP of each specimen was obtained by calculating the color difference between the specimen against the white background and against the black background using the following equation:

$TP = \{(Lb* - Lw*)^{2} + (ab* - aw*)^{2} + (bb* - bw*)^{2}\}^{1/2}$

The mean values of translucency parameters (TP) for all groups has been graphically demonstrated in Figure 16, in Chapter 3. All treatments showed a slight reduction in the translucency parameter reading for the three ceramics, except that Group C (brushing only) for the IPS e.max demonstrated a slight increase in the translucency parameter as compared to Group A (control). It is well established that translucency of ceramics is affected by ceramic thickness and presence of opacifiers, such as tin oxide, more than by surface topography. The changes in



translucency parameter in the current study are not significant statistically and although present, may not be visually perceptible to the naked eye.

4.1.2.2 Color

The subjectivity in perception of color by an observer results in variations and unpredictable differences in color evaluation and matching among clinicians. ⁸⁴ Spectral distribution of color stimulus, its size, shape and structure, surroundings of the stimulus, state of the observers visual system and the observer's experience affect the visual color evaluation.⁸⁴ These elements can be clinically identified as change in ambient lighting, color of the patient's complexion, make up, clothing, distribution of the appearance of nearby teeth, and the contour and finish of the restoration.⁸⁵

For the purpose of the study, a digital spectrophotometer (Gregtag Macbeth[®] Color-Eye 7000 A) was used to record the CIELAB (L*, a*, b*) coordinates of all the ceramic samples. Color calculations were made using the CIE $L^*a^*b^*$ readings in the following formula:

$\Delta E = (L^{*2} + a^{*2} + b^{*2})^{1/2}$

In the current study, the color differences (ΔE) between groups were not statistically significant (p<0.05) for the feldspathic porcelain and monolithic zirconia specimens. The IPS e.max ceramic specimens however, demonstrated statistical differences as follows: There was a significant effect of acid and brushing treatment on color at the *p* < 0.05 level for IPS e.max [F (3, 28) = 3.09, p = 0.043,



 n^2 = 0.25]. We see a significant difference between the control group A vs. brush only group C [difference = 3.43, 95% CI (0.19, 5.67), p = 0.035]. Figure 15 in chapter 3 is a graphical demonstration of the differences in mean values of color for all groups of IPS e.max specimens. Additionally, Figure 19 demonstrates L*, a* and b* readings for IPS e.max for different groups. All groups demonstrated an increase in L* readings with group C (brush only) showing the largest increase with respect to the control group. The L* coordinate corresponds to the value (lightness or darkness) of an object which is considered to be the most important aspect of color selection in dentistry. The a* and b* readings were relatively constant between control, acid and brush groups but slightly increased for acid+brush group which indicates a shift from redness to greeness as the a* value increases. Similarly, the increase in b* represents a shift from yellowness to blueness. Figure 20 a-h., shows SEM micrographs of IPS e.max specimens after various treatments. It was observed that the ceramic surface exhibits a smoother appearance for groups C (brushing only) and D (acid + brushing treatment) as compared to the control group A. It may be extrapolated then, that the abrasive action of tooth-brushing could have potentially smoothened out some flaws in the surface of the ceramic material. This speculation, however, is not consistent with the surface roughness measurements made using a profilometer, where groups B though D show a rougher surface (increased value of Ra in µm) as compared to the control group. This observation opens up interesting avenues for discussion about the effect of a corrosive environment and tooth brushing on the color stability of IPS e.max. Although statistically significant differences were observed between



groups, it has been accepted that a ΔE value of 3.5 or greater is considered clinically significant.⁸⁶ The groups closest to this value were the control group A and brush only group C, where the color difference (ΔE) was 3.54. Whether these color changes are visually perceptible to the observer, can be further explored by more long term clinical studies.

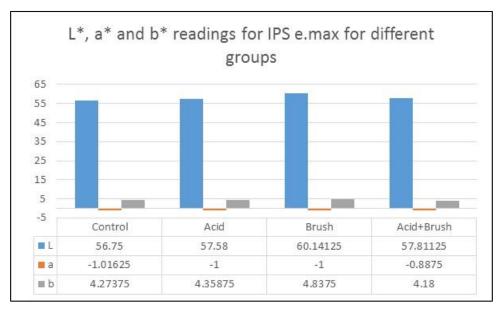


Figure 19. L*, a*, b* readings for IPS e.max for different groups

4.1.2.3 Surface gloss

The surface gloss is an important characteristic of a ceramic restoration. Attempts are made to obtain smooth, glossy surfaces for restorations to enhance esthetic appearance and to obtain surfaces which are unfavorable for plaque retention and wear of the opposing dentition. It has been established from previous studies that the various optical properties of a ceramic are a function of their composition,



surface topography and thickness⁸⁷, as these factors control light reflection and transmittance from the objects. Loss of luster or gloss can lead to a dull looking finish of the restoration and affect the visual color perception, having adverse effects on the esthetic outcome. Most ceramic restorations are glazed prior to insertion into the oral cavity. The goal of glazing is to seal the open pores in the surface via fired glass porcelain to render a smooth surface.⁸⁸ However, some studies have suggested that a polished surface may be as acceptable as a glazed surface,⁸⁹ and preferred by ceramists for better control over the surface luster. For the purpose of this study, ceramics manufactured by CAD/CAM technology were obtained and heat treated per the recommendations of the manufacturer, without adding a glaze layer. The specimens were finished and polished as final restorations would be prepared by a technician for delivery to the clinician. A gloss meter (Novo-Curve[™] Glossmeter, Rhopoint[™] Instruments Ltd, Figure 12.a) was used to measure specular reflection gloss of all groups of ceramics. The surface gloss measurement was collected (in degrees) as the amount of reflected light at an equal but opposite angle to the projecting beam of light from the specimen. (Figure 12.b).

Figure 14 shows a graphically the differences in mean values of surface gloss for all groups of ceramic specimens. There was a significant effect of acid and brushing treatment on gloss at the *p* < 0.05 level for feldspathic porcelain [F (3, 28) = 4.25, *p* = 0.013, η^2 = 0.31]. We see a significant difference between the control group A vs. acid only group B [difference = 18.21, 95% CI (3.45, 32.96), *p* = 0.011].



These findings are correlated with the changes in surface roughness (Ra in µm) of porcelain specimens (Figure 17). Increase in surface roughness caused the surface gloss to decrease. For groups C and D of the porcelain specimens, it is interesting to note that the increase in surface roughness and decrease in surface gloss is not as significant. This may lead to the speculation that the brushing treatment had a tendency to smooth or seal the surface flaws created by the acid treatment, and this can further be explored in subsequent studies. Of the three materials chosen for this study, feldspathic porcelain is the softest material, which explain why the changes in surface roughness were the most noteworthy in these specimens.

4.1.3 Study design

To date, there is no consensus in the dental literature as to the method of gastric acid simulation and exposure time for an in-vitro study model. The ISO testing standard⁹⁰ for dental ceramics for solubility testing recommends the use of 4% acetic acid for 16h at 80°C, which corresponds to an *in-vivo* relevance of 2 years, based on the work of De Rijk.⁹¹ Hunt andMcIntyre (1985)⁹² developed a model to simulate *in-vivo* acid erosion of teeth which simulates about 2-3 years clinically. Kukiattrakoon *et al* immersed ceramics in acidic solutions for 168 hours, and found an increase in their surface roughness.⁵⁴ These studies, however, aged the specimens and subjected them to a static exposure to corrosive agents which is not clinically relevant. The novelty of the current study was to attempt to emulate



the oral condition in patients with GERD or bulimia nervosa. With regards to exposure of teeth to acid, it has been observed in certain risk populations (subjects with eating disorders) that vomiting frequencies of 6–10 times per day are often reported by patients.⁹³ Based on this finding, some erosion-abrasion studies⁹⁴⁻⁹⁵ have used an erosion cycle of 6 times a day for 2 minutes each for 9 days. These studies were used for enamel and dentine, where the authors studied the demineralization-remineralization The process upon acid exposure. demineralization (acid attack) period of 2 minutes imitated the length of the pH decline in saliva after an acid attack.⁹⁶⁻⁹⁷ Additionally, the specimens were immersed in artificial saliva for 2 hours after every 2 minutes of exposure for the remineralization to occur. This is extrapolated as 108 minutes of acid attack and a total 108 hours of exposure. This was the treatment time adopted for the current study. It was assumed by the authors that immersion of ceramics in artificial saliva for 2 hours after each acid attack will not alter the surface or structure of the ceramics (as opposed to enamel or dentin), and hence the samples were thoroughly rinsed for 2 minutes between acid attacks. The oral environment is a dynamic environment with changes in pH due to the buffering action of saliva, and to the current knowledge of the author, is the only study to have made an attempt to mimic a clinical scenario for acid treatment of dental ceramics.

The component of toothbrush abrasion was incorporated in this current study to try to emulate different clinical scenarios of patient with GERD to see if toothbrush abrasion alone or in combination with acid erosion negatively impacted the



properties of ceramics. A standardized brushing machine was utilized (with a constant load of 400 grams) and the brushing duration per sample (100 seconds per day) was approximately that of average daily habits in which the time spent for a whole quadrant is estimated to be approximately 24 seconds.⁹⁸

The addition of pepsin and acid preparations was carried out as per recommendations of the manufacturer and a pH of 2.0 was maintained for the exposure. Even though gastric acid is a highly acidic acid (ph~1.6), the slightly higher pH for the experiment accounts for the buffering action of salivary components in the oral cavity.



4.2 Morphological assessment of surface characteristics

Figures 20-22 demonstrate SEM images of all treatment groups from randomly selected specimens of all three ceramic materials. The images displayed are of 500x and 5000x magnification.

	500x	5000x
Group A Control		
Group B Acid only		
Group C Brush only		P ************************************
Group D Acid+Brush		

Figure 20. SEM micro graphs of IPS e.max samples of all groups



	500x	5000x
Group A Control		
Group B Acid only		212 Yan 5020 22 II ige koza - 14/ 12 Yan 5020 22 II ige koza - 14/ 12 Yan 5020 22 II ige koza - 14/
Group C Brush only	WU Man Store Startood	W07 M02: Spall VolfAcole 10 V 7 M02: Spall VolfAcole 10
Group D Acid+Brush	9 7 ° m 1020 25 11 mg Hover n (123 M ²	

Figure 21. SEM micro graphs of Feldspathic porcelain samples of all groups



	500x	5000x
Group A Control	The sum (2001) See High Sectors - 20 (12 - sum (2001) See High Sectors - 20 (12 - sum (2001) See High Sectors - 20 (12 - sum (2001) See High Sectors - 20 (12 - sum (2001) Sectors - 2	Por Mark Robert Die Der Kannen Berger
Group B Acid only	Wini Jang Spall VacMont Jug	Will Mus Sort VacAdom Will Yamni Lobik 25 Helphaszum 12540 TRUCEs age of Genis treats re-
Group C Brush only	Wing 15g vir white: 100	
Group D Acid+Brush	VU Roc Son Vadrom 10 Tating OX 20 Parkager 122 XV Hostop 20 April	

Figure 22. SEM micro graphs of Monolithic zirconia samples of all groups



4.3 Limitations, Implications and Future Studies

The major limitations of this study is that it is an in-vitro study and was conducted in a non-blinded manner. It is well established that while in-vitro studies can simulate intra-oral conditions, it is not possible to fully reproduce them. When placed in the oral cavity, ceramic restorations are subject to complex thermal, physical and chemical challenges which are difficult to accurately reproduce under experimental conditions. The non-blinded methodology implemented for this research may have introduced confounding variables which could affect the outcome of the study. Although the ceramic samples were stored in distilled water 24 hours before and during the testing, an ideal environment should have been saliva. If the specimens are not sufficiently hydrated during testing, the effect of different treatments could vary.

Due to vast differences in the compositions of dental ceramics, and variations in testing protocols, previous research has shown conflicting findings about the effect of acid treatment on ceramics. While most results agree that surface roughness is negatively impacted by the acid treatment, a more standardized testing protocol is required for more reproducible results. This study measured the changes in surface roughness (in μ m). Wear resistance of the ceramics before and after acid attack was not evaluated. This can be calculated as the % weight loss from the specimen after acid treatment. This might give a better understanding of the potential of the acid attack to affect the resistance to abrasive wear of the ceramic. Another important concern is potential ion interactions and leaching upon corrosive attack, which can be explored in future research.



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An interesting finding of this research was the impact that the acid treatment, with and without the combination of toothbrush abrasion, had on the color of the IPS e.max ceramics. The brushing treatment produced a significant color difference as compared to the control ($\Delta E = 3.54$) and this incidental finding opens up avenues for further research on the color changes of these ceramics in the oral environment.



CHAPTER 5: CONCLUSIONS

Within the limitations of this in-vitro experimental study, it was found that gastric acid treatment affected the surface roughness of all three ceramics used, where the changes for feldspathic porcelain were most significant, followed by lithium disilicate glass ceramics. The acid treatment with or without tooth brush abrasion negatively impacted the surface gloss of feldspathic porcelain and the color of IPS e.max, where color changes with brushing alone were most significant for IPS e.max specimens. The changes in translucency and mechanical strength for all materials were not statistically significant. Zirconia ceramic showed resistance to all treatments.

In conclusion, in the pursuit of the "latest and greatest" biomaterials, clinicians should always be aware of the potential longevity of any restorative material, especially when treating patients with comorbidities that alter the oral cavity environment. Further research should be carried out to confirm the findings of this present study in a reproducible manner to aid in proper material selection and more predictable treatment outcomes.



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