

# EFFECT OF ARTIFICIAL GASTRIC ACID AND BRUSHING ON THE OPTICAL PROPERTIES AND SURFACE HARDNESS OF COLORED ZIRCONIA

Alofi S. RANEEM<sup>1</sup>, Albani M. RAGAD<sup>2</sup>, Alhaizan A. YASMEEN<sup>2</sup>, Alofi, S. ADEEM<sup>3</sup>

<sup>1</sup>BDS, CAGS, DScD, „King Saud” University, Riyadh, Saudi Arabia

<sup>2</sup>BDS, Dental intern, „King Saud” University, Riyadh, Saudi Arabia

<sup>3</sup>BDS, DScD, „King Saud Bin Abdulaziz” University for Health Sciences, Riyadh, Saudi Arabia

Corresponding author: Raneem S. Alofi; e-mail: ralofi@ksu.edu.sa

## Abstract

**Objectives:** To assess the effect of artificial gastric acid with and without brushing on color stability, surface gloss and surface hardness of colored zirconia. **Materials and methods:** Monochromatic zirconia (A) and zirconia colored before sintering (B) were milled into discs and sintered. Each material was divided into three groups (n=11/group): control (1), acid exposure (2), acid exposure and brushing (3). All groups, the control one excluded, were emerged into artificial gastric acid, rinsed with water, and stored in distilled water. Groups A3 and B3 were subjected to brushing. Changes in color, gloss and hardness were measured. **Results and discussion:** There was a significant difference in color after acid exposure with or without brushing in both materials (p<0.05). Surface hardness was significantly lower when exposed to acid with or without brushing, compared to the control group (p=0.0001). Surface gloss shows no statistical difference comparatively with the control groups (p>0.05). When comparing monochromatic and colored zirconia, no significant difference was found in any parameter. **Conclusions:** zirconia exposure to gastric acid with or without brushing will affect its color and lower its surface hardness, regardless the application of coloring stains.

**Keywords:** dental erosion, gastric acid, zirconia, hardness, color, gloss.

## 1. INTRODUCTION

An individual's smile determines the first impression and plays a major role in one's self-confidence, which increases the demand for aesthetic treatment. Due to this increased demand, dentists are trying to locate the best aesthetic and functional materials to treat their patients. Aesthetic fundamentals, including beauty, naturalness and individuality are idealizing or harmonizing the artificial with the natural aspect. [1]

The oral cavity is subjected to a variety of conditions, depending on individual's health

status. Dental hard and soft tissues may undergo multiple physiological changes. It has been mentioned in the literature that a salivary pH below 5.5 will result in demineralization of the dental hard tissue, which will eventually lead to a loss of tooth structure. Loss of tooth structure due to acid exposure, which results in a physiochemical process of dissolution of the dental hard tissue without bacterial activity, is defined as dental erosion. A previous systematic review established an increase in the prevalence of tooth wear among both deciduous and permanent dentition. [2] Dental erosion has a multifactorial etiology, which could be due to intrinsic or extrinsic factors. [3] Dentists often have to consider two main aspects while planning any restorative procedure: function and aesthetics. Increased awareness has resulted in a shift in trends that demands a more organized and systematic approach towards dental aesthetics, such that the overall health of teeth, the patient remaining the most important factor. [4] Metal-free all-ceramic restorations gained popularity due to their high aesthetics and good biocompatibility. [5] Zirconia is usually provided as monolithic blocks, difficult to match the natural teeth color. Therefore, color matching of zirconia is challenging. To overcome this issue, additional coloring techniques may be used, such as: mixing pigments into zirconia powder, dipping zirconia milled frameworks in dissolved coloring agents, and lining the sintered zirconia framework. Application of these stains to add finer details such as mamelons, fissures, and cervical stains and to customize these restorations aims at achieving best aesthetic results. [6,7] Zirconia offers a good clinical choice as an aesthetic restorative material,

due to the continuous improvements in translucency and to the absence of metal exposure, especially at the restoration margin. [8] Another challenge facing dentists when restoring a patient's aesthetics is his/ her patient medical condition. Regurgitation of the gastric contents to the esophagus occurs physiologically in a healthy individual. When uncomfortable symptoms, such as acid regurgitation and/or heartburn (occurring at least once a week) and esophageal mucosal changes accompany these refluxes, "gastroesophageal reflux disease (GERD)", the most common intrinsic factor of dental erosion, occurs. [9,10] A previous systematic review stated a range of GERD prevalence of 18.1%-27.8% in North America, 23% in South America, 8.8% 25.9% in Europe, 11.6% in Australia, 2.5%-7.8% in East Asia, and 8.7%-33.1% in the Middle East, respectively. [11] Moreover, a study conducted in Saudi Arabia showed a 28.7% prevalence of GERD among the Saudi population, a higher value compared to other countries.[12] As a consequence of erosion dental hypersensitivity, compromised aesthetics and loss of vertical dimension could be reported in patients suffering from gastroesophageal reflux disease.[13] The degree of dental erosion varies from one patient to another, depending on many factors, such as the dental surface contacting the acid and the duration of contact, in addition to the salivary buffering capacity. [14,15] Teeth brushing involves mechanically applying a force for a period of time on teeth surfaces. As dentifrices contain abrasives, they can chemically and physically affect the brushed teeth. Studies have shown that the fluoride from dentifrices reduces the properties of dental ceramics and can therefore alter its optical properties. [8]

A study of Kulkarni *et al.* showed a statistically significant difference in color, surface roughness, and gloss for e-max and porcelain specimens after being subjected to teeth brushing abrasion and gastric acid, in an attempt to stimulate oral conditions. [16] Another study stated that, after acid exposure, smoother surfaces of monolithic zirconia were recognized but, on the other hand, fewer changes in the optical properties of zirconia were observed, comparatively with other types of dental ceramics. [17] The literature reports that microhardness is significantly affected in dental

ceramics after being emerged in different types of acids, the changes varying according to the type of ceramics and of acid. [18]

The present study aims at assessing the effect of gastric acid and brushing on the optical properties and surface hardness of colored zirconia. The null hypothesis is that acid exposure and brushing will not affect the optical properties and surface hardness of colored zirconia.

## 2. MATERIALS AND METHODS

### *Materials*

Presintered Zirconia (Prettau Anterior, Zirkozahn) was the material of choice in this study. (A) Monochromatic zirconia in shade A2 and (B) Monochromatic zirconia colored before sintering with (Colour Liquid Prettau Anterior Aquarell A2, Zirkozahn) as per manufacturer instructions.

### *Specimen Preparation*

Zirconia was milled into 10 mm in diameter and X 2mm thick discs using the computer-aided design/ computer-aided manufacturing (CAD/ CAM) technology. The discs were sintered in (ZIRCONOFEN 600, 1,500°C) for 2 hours, following manufacturer specifications. Finally, they were polished using a (DIAPOL® by EVE) polishing system starting from blue zirconia polisher (coarse grit, 7,000 rpm), red (medium grit, 7,000 rpm), and white (fine grit, 7,000 rpm), for 15 seconds for each polishing step. With a total of 66 specimens, each material was randomly divided into three groups (n = 11/ group) based on the treatment received (Table 1), and stored in distilled water at 37° (Mettmert Universal Oven, Mettmert Edestahl Rost Frei) for 24 hours before exposure.

**Table 1. Experiment groups based on the treatment received**

	(A) monochromatic zirconia	(B) colored zirconia
1	No exposure	
2	Acid exposure only	
3	Acid exposure and brushing	

### Specimen Treatment

Artificial gastric acid (0.1N HCL buffer solution of pH 1.2) was prepared using 8.3 ml hydrochloric acid in 1L of distilled water. The pH was maintained at 1.2 using a pH-measuring probe (HQ411 Laboratory pH Meter; HACH).

Specimens from groups A2, A3, B2, and B3 were immersed in the prepared solution for 2 minutes, rinsed with water, and finally stored in distilled water for at least 30 minutes between acid exposures. The process was repeated 208 times, representing 4 regurgitation episodes per week, for 1 year. [19]

Specimens from groups A3 and B3 were subjected to tooth brushing after acid treatment using Toothbrush simulation ZM-3, SD Mechatronik with 10 linear brushing strokes (1.6N) repeated 730 times, which means 2 brushing sessions per day, for 1 year. [20] The specimens were mounted on the machine and brushed using a fresh dentifrice slurry (Closeup Deep Clean Toothpaste, Unilever).

### Testing and measurements

Changes in color were measured using a digital spectrophotometer (Labscan XE spectrophotometer, Hunterlab) and CIELAB ( $L^*$ ,  $a^*$ ,  $b^*$ ) color coordinates. The spectrophotometer was calibrated as per manufacturer's instructions. Calculation of  $\Delta E$  was made using the CIE  $L^*a^*b^*$  reading, where  $L^*$  refers to lightness,  $a^*$  to redness to greenness, and  $b^*$  to yellowness to blueness, using the following formula:

$$\Delta E = \sqrt{\Delta L^2 + \Delta a^2 + \Delta b^2}$$

Surface gloss was measured using a glossmeter (Novo-Curve Glossmeter, Rhopoint Instruments) after being calibrated as per manufacturer's recommendations, using a 60-degree angle and 2 readings for each specimen. Surface hardness was tested using NOVA 130 Micro Vickers Hardness Tester, Innovatest, at room temperature, with an indent load of 200 g and dwell time of 15 seconds, using the following formula:

$$\text{Vickers hardness} = 0.0102 \times ((2F \sin 136^\circ/2)/d^2) \\ \approx 0.1891 \times F/d^2$$

where  $F$  is the force in Newtons and  $d$  is the mean length of two diagonals of the indentation in mm.

### Statistical Analysis

Descriptive statistics for each group was reported as mean and standard deviations. The two sample T-test was used to compare the effect of acid, with or without brushing, on the surface characteristics between the two types of ceramics. Analysis of variance (ANOVA) was used to compare surface characteristics (change in color, gloss, and hardness) between the three groups (control, acid, acid + brushing). A Tukey HSD *post hoc* test was used for multiple groups comparison at a level of  $\alpha = 0.05$ . Data analysis was performed using SAS software (SAS Institute Inc., Cary, NC) version 9.4.

## 3. RESULTS

In both types of zirconia, surface exposure to acid with or without brushing had a higher value of  $\Delta E$  compared to controls. This difference was statistically significant ( $p < 0.05$ ). No statistically significant difference in  $\Delta E$  was seen when comparing acid and acid with brushing ( $p > 0.05$ ).

Exposure to acid with or without brushing showed no statistically significant difference in surface gloss, compared to the control ( $p > 0.05$ ).

Surface hardness was significantly lower in specimens exposed to acid with or without brushing, compared to the control group ( $p = 0.0001$ ). This difference was not statistically significant when comparing the effect of acid alone to acid with brushing ( $p > 0.05$ ).

When comparing monochromatic zirconia and colored zirconia, the data showed no statistically significant difference as to the effect of acid treatment on  $\Delta E$ , gloss, and hardness (P values = 0.77, 0.65, 0.36), respectively. Acid and brushing showed no statistically significant difference in  $\Delta E$ , gloss, and hardness (P values = 0.1, 0.74, 0.45) between the two types of zirconia.



Table 2. Mean (standard deviation) of surface characteristics for zirconia ceramics

	Control		Acid		Acid and brushing	
	Mono-chromatic zirconia	Colored-zirconia	Mono-chromatic zirconia	Colored-zirconia	Mono-chromatic zirconia	Colored-zirconia
	N=11	N=11	N=11	N=11	N=11	N=11
$\Delta E$	0.12±0.04 <sup>1</sup>	0.12±0.06 <sup>1</sup>	2.91±1.79 <sup>2</sup>	2.72±1.09 <sup>2</sup>	3.38±2.30 <sup>2</sup>	2.01±1.33 <sup>2</sup>
<b>Hardness</b>	1544.54±3.19 <sup>1</sup>	1543.86±2.25 <sup>1</sup>	1474.21±1.53 <sup>2</sup>	1473.58±1.62 <sup>2</sup>	1474.44±2.16 <sup>2</sup>	1473.84±1.4 <sup>2</sup>
<b>Gloss</b>	175.83±7.32 <sup>1</sup>	178.39±5.93 <sup>1</sup>	185.21±11.26 <sup>1</sup>	183.49±5.2 <sup>1</sup>	181.23±10.35 <sup>1</sup>	182.35±4.33 <sup>1</sup>

\* Different numbers represent statistically significant differences within each row (P<.05).

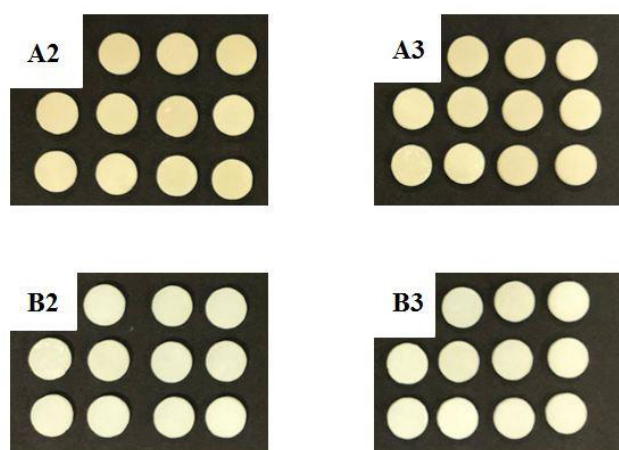


Fig. 1. Laboratory specimens. A2-monochromatic zirconia with acid exposure only. A3-monochromatic zirconia with acid exposure and brushing. B2-colored zirconia with acid exposure only. B3-colored zirconia with acid exposure and brushing

#### 4. DISCUSSION

This study evaluates the artificial gastric acid effect on the color, gloss, and surface hardness of colored zirconia. The results lead to partially rejecting the first hypothesis: exposure of colored zirconia to gastric acid and teeth brushing abrasion negatively affect its surface color but not its surface gloss. The second hypothesis was rejected: exposure of colored zirconia to gastric acid and teeth brushing abrasion negatively affect its surface hardness. Acidic exposure of

zirconia specimens will affect some of its properties, on the other hand, teeth brushing abrasion do not affect specimens. Staining of zirconia has no impact.

Recently, the dramatic increase in aesthetic dentistry raised a concern about the use of liquid stains to simulate natural teeth color and stability in a variety of conditions affecting the oral environment. The stability of these stains in an acidic environment is not yet investigated. Many studies focused on improving the mechanical properties of zirconia, but only a few examined the effect of acidic environment on zirconia color stability, especially when using intrinsic stains. The novelty of this study is that it investigates the gastric acid effect either with or without teeth brushing abrasion on the optical properties and surface hardness of zirconia colored before sintering.

Color perception varies from one clinician to another. [21] Many factors may affect color evaluation, such as adjacent natural teeth, ambient light, clothing, and makeup. [22] A spectrophotometer is an accurate device that eliminates any subjective evaluation and provides good reproducibility. [23]

In this study, for both monochromatic and colored zirconia, a significant color change was found, which is a clinically unfavorable finding, meaning that the color in zirconia is unstable in an acidic environment. A previously published study stated that the color of zirconia reacts

differently when subjected to an acidic environment, a finding agreeing with the present study, [17] unlike a previous study of Kulkarni *et al.*, who found no significant change in the color of zirconia when subjected to an acidic attack. This could be due to the difference in the acidic immersing protocol between the two studies. [16] In Kulkarni *et al.* study, both sodium chloride and hydrochloric acids were used with pepsin powder to create the artificial gastric acid while, in the present study, only hydrochloric acid was used. In the study conducted by Kulkarni, acid exposure was repeated 54 times and specimens were stored in deionized water at 37°C while, in our study, the procedure was repeated 208 times and the specimens were stored in distilled water for 30 minutes between each acid exposure. The color change could be due to zirconia degradation, possibly accelerated in the presence of water, leading to a change in grains size and increase in volume, resulting in microcracks development. [24] Discoloration and change could result from acid leaking within these microcracks.

Gloss is an important parameter in the optical properties of zirconia, determining surface light reflection, which will affect the visual appearance of the material. It has been observed that zirconia will undergo some topographic changes in the surface after being exposed to acidic attack which, as a result, could affect light reflection. [17,18] In this study, increase in the surface gloss could be due to the smoother surface of the material after acidic exposure, even if it was not statistically significant; increasing the sample size may lead to a significant increase in the surface gloss of both monolithic and colored zirconia, with or without teeth brushing abrasion.

Surface hardness is one of the important mechanical properties in dentistry, being defined as the resistance of the material to indentation. [25] In this experiment, a significant decrease in surface hardness was observed between the control groups of both materials and all other groups. Similarly, a previous study stated that the value of microhardness of different dental ceramics decreased significantly. [18] This may be related to the aging phenomenon of zirconia, due to the tetragonal-monoclinic phase transformation after acid exposure, which could

affect the longevity of this type of material and be related to grain size, residual stress, and stabilizer type and content. [24]

## 5. CONCLUSIONS

---

Within the limitation of this study, zirconia exposure to gastric acid with or without brushing will affect its color and reduce its surface hardness, regardless of the application of coloring stains. However, since the study was conducted in a controlled laboratory setting, results may vary when zirconia is exposed to different environmental conditions in the oral cavity. The effect of moisture on aging and fatigue of zirconia was not considered. Lastly, the effect of toothpaste mixed with saliva was not investigated.

**Acknowledgements.** *The authors extend their appreciation to the Deanship of Scientific Research at "King Saud" University for funding this work through the undergraduate research support program, project no. (URSP-4-19-106). The authors also would like to thank Dr. Mohammed Alsenaidy for his help in artificial acid preparation.*

## References

---

1. Bolla SC, Gantha NS, Sheik RB. Review of History in the Development of Esthetics in Dentistry. IOSR-JDMS; 2014;13(6):31-5.
2. Kreulen CM, Van't Spijker A, Rodriguez JM, Bronkhorst EM, Creugers NHJ, Bartlett DW. Systematic review of the prevalence of tooth wear in children and adolescents. Caries Res. 2010;44:151-9. <https://doi.org/10.1159/000308567>.
3. Picos A, Chisnoiu A, Dumitrasc DL. Dental erosion in patients with gastroesophageal reflux disease. Adv Clin Exp Med. 2013;22(3):303-7.
4. Spear FM, Kokich VG, Mathews DP. Interdisciplinary management of anterior dental esthetics. J Am Dent Assoc. 2006;137(2):160-9.
5. Beuer F, Stimmelmayer M, Guetha JF, Edelhoff D, Naumann M. In vitro performance of full-contour zirconia single crowns. Dent Mater. 2012;28(4):449-56.
6. Daou EE. The Zirconia Ceramic: Strengths and Weaknesses. Open Dent J. 2014;8:33-42. doi: 10.2174/1874210601408010033.
7. Ahangari HA, Ardakani TK, Mahdavi F, Ardakani TM. The Effect of two Shading Techniques on Value of Zirconia-Based Crowns. J Dent (Shiraz). 2015;16(2):129-33.

8. Lee JH, Kim SH, Han JS. Optical and Surface Properties of Monolithic Zirconia after Simulated Tooth brushing. *Materials (Basel)*. 2019; 12(7):1158.
9. Bredenoord AJ, Pandolfino JE, Smout AJPM. Gastro-oesophageal reflux disease. *Lancet*. 2013;381(9881):1933-42.
10. Ramachandran A, Raja Khan SI, Vaitheeswaran N. Incidence and Pattern of Dental Erosion in Gastroesophageal Reflux Disease Patients. *J Pharm Bioallied Sci*. 2017;9(Suppl 1):S138-41.
11. El-Serag HB, Sweet S, Winchester CC, Dent J. Update on the epidemiology of gastro-oesophageal reflux disease: a systematic review. *Gut*. 2014;63(6):871-80.
12. Alsuwat OB, Alzahrani AA, Alzhrani MA, Alkathami AM, Mahfouz MEM. Prevalence of Gastroesophageal Reflux Disease in Saudi Arabia. *J Clin Med Res*. 2018;10(3):221-5.
13. Ali DA, Brown RS, Rodriguez LO, Moody EL, Nasr MF. Dental erosion caused by silent gastroesophageal reflux disease. *J Am Dent Assoc*. 2002;133(6):734-7.
14. Lazarchik DA, Filler SJ. Effects of gastroesophageal reflux on the oral cavity. *Am J Med*. 1997;103(5A):107S-13S.
15. Valena V, Young WG. Dental erosion patterns from intrinsic acid regurgitation and vomiting. *Aust Dent J*. 2002;47(2):106-15.
16. Kulkarni A, Rothrock J, Thompson J. Impact of Gastric Acid Induced Surface Changes on Mechanical Behavior and Optical Characteristics of Dental Ceramics. *J Prosthodont*. 2020;29(3):207-18.
17. Sulaiman TA, Abdulmajeed AA, Shahramian K, Hupa L, Donovan TE, Vallittu P, Närhi TO. Impact of gastric acidic challenge on surface topography and optical properties of monolithic zirconia. *Dent Mater*. 2015;31(12):1445-52.
18. Kukiattrakoon B, Hengtrakool C, Kedjarune-Leggat U. Chemical durability and microhardness of dental ceramics immersed in acidic agents. *Acta Odontol Scand*. 2010;68(1):1-10.
19. Wiegand A, Attin T. Design of erosion/abrasion studies--insights and rational concepts. *Caries Res*. 2011;45 Suppl 1:53-9. doi: 10.1159/000325946.
20. Wiegand A, Burkhard JPM, Eggmann F, Attin T. Brushing force of manual and sonic toothbrushes affects dental hard tissue abrasion. *Clin Oral Investig*. 2013;17(3):815-22.
21. Ishikawa-Nagai S, Yoshida A, Sakai M, Kristiansen J, Da Silva JD. Clinical evaluation of perceptibility of color differences between natural teeth and all-ceramic crowns. *J Dent*. 2009;37 Suppl 1:e57-63. doi: 10.1016/j.jdent.2009.04.004.
22. Johnston WM, Kao EC. Assessment of appearance match by visual observation and clinical colorimetry. *J Dent Res*. 1989 May;68(5):819-22.
23. Da Silva JD, Park SE, Weber HP, Ishikawa-Nagai S. Clinical performance of a newly developed spectrophotometric system on tooth color reproduction. *J Prosthet Dent*. 2008;99(5):361-8.
24. Chevalier J, Gremillard L, Virkar AV, Clarke DR. The Tetragonal-Monoclinic Transformation in Zirconia: Lessons Learned and Future Trends. *J Am Ceram Soc*. 2009;92:1901-20. doi:10.1111/j.1551-2916.2009.03278.x.
25. Wang L, D'Alpino PHP, Lopes LG, Pereira JC. Mechanical properties of dental restorative materials: relative contribution of laboratory tests. *J Appl Oral Sci*. 2003;11(3):162-7.