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Research Article

Assessment of Surface Roughness and Color Stability of Glazed Versus Polished Pressed Zirconia Reinforced Lithium Silicate Restorations Under Different pH Oral Media (An *In-Vitro* Study)

Samar Fathy Fathy El Shihy[1]*, Reham Said El Basty[2] and Gihan Abd Elhady El Naggar[3]

[1]Fixed Prosthodontics Department, Faculty of Dentistry, Cairo University, Cairo, Egypt and A. Lecturer, Fixed Prosthodontics Division, School of Dentistry, Newgiza University, Giza, Egypt

[2]A. Professor, Fixed Prosthodontics Department, Faculty of Dentistry, Cairo University, Cairo, Egypt and Head of Fixed Prosthodontics Division, School of Dentistry, Newgiza University, Giza, Egypt

[3]Professor, Fixed Prosthodontics Department, Faculty of Dentistry, Cairo University, Cairo, Egypt

***Corresponding Author:** Samar Fathy Fathy El Shihy, Fixed Prosthodontics Department, Faculty of Dentistry, Cairo University, Cairo, Egypt and A. Lecturer, Fixed Prosthodontics Division, School of Dentistry, Newgiza University, Giza, Egypt

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Abstract

Aims: Assess the effect of the surface finish protocol of pressed zirconia reinforced lithium silicate ceramics on the surface roughness and color stability under different pH oral media.

Methodology: A total of 30 samples were used in the present study. The samples were divided into two main groups according to the surface finishing protocol (n = 15); glazed and polished group. Then, subdivided into three subgroups according to the media tested: coffee, carbonated orange juice and artificial saliva as the control group. The surface roughness was measured using optical profilometer quantitatively and with scanning electron microscope qualitatively. Also, Color parameter was measured using spectrophotometer. Afterwards, the discs were immersed in the aging solutions for 12 days simulating 1 year in-vivo. Then, the surface roughness and color change were remeasured after aging.

Result: For surface roughness results, regardless of surface finish protocol the difference between immersion solutions was statistically non-significant where (Coffee ≥ Fanta ≥ AS). Also, irrespective of immersion solutions the difference between surface finish protocol was statistically non-significant where (Glazed ≥ Polished). Regarding the color stability results, regardless of surface finish protocol the difference between immersion solutions was statistically non-significant where (Coffee > Fanta > A.S), furthermore, irrespective of immersion solutions the difference between surface finish was statistically significant where (Glazed > Polished).

Conclusion: Surface finish protocol (glazed and polished), as well as immersion media, showed no effect on the surface roughness of zirconia reinforced lithium silicate restorations. Also, Color stability was significantly affected by the surface finish protocol, while the pH immersion media showed no effect on the color stability.

Keywords: Zirconia Reinforced Lithium Silicate; Celtra Press; Surface Finish; Surface Roughness; Color Stability; Optical Profilom-

Abbreviation

SEM: Scanning Electron Microscope;AS: Artificial Saliva; STL: Standard Tessellation Language; ZLS: Zirconia Reinforced Lithium Silicate

Introduction

The use of all-ceramic restorations has evolved because of their excellent aesthetics, biocompatibility, and enhanced strength. Celtra Press is available nowadays which is a new class of zirconia-reinforced lithium silicate material for pressing. It is a multiphase ceramic characterized by high strength of about 500 MPa and excellent flow properties during pressing consisting of a glass matrix and lithium silicate crystals having a crystal length of about 1.5 µm plus Nano-scale lithium phosphate and about 10% zirconia ($ZrO2$), which is dissolved completely in the glass.

Surface roughness of ceramic restoration is a character of surface texture that must be kept to a minimum; however, it is unavoidable. Since surface roughness may increase during fabrication process, laboratory procedures or chair side adjustments, therefore, Glazing and polishing are two finishing protocols that are commonly used to provide a smooth surface of all ceramic restorations. The effect of glazing and polishing on the surface roughness of dental ceramics is still controversial.

Color stability of the restoration depends on maintaining its color intraorally with non-perceivable change. The color of an aesthetic restoration and its stability is important for its long-term success. Color of ceramics is generally affected by intrinsic factors such as the ceramic composition, the glaze layer, and the surface texture. Whereas food, liquid colorants are extrinsic factors that can influence color stability.

Commonly consumed beverages can affect the surface roughness and color stability of dental restorations due to the colouring agents in these beverages and their acidic content that may affect the surface roughness and may also increase the stainability of this rough surface. Limited evidence is available regarding the alterations caused by acidic agents on the surface of glazed and polished zirconia reinforced lithium silicate ceramics. Hence, the aim of the study was to assess the effect of the surface finish protocol (glazing vs. polishing) of zirconia reinforced lithium silicate ceramics on the surface roughness and Color stability under different pH oral media. (Artificial saliva, Coffee, Carbonated orange juice)

Materials and Methods

Sample size

This *in-vitro* study was conducted in the laboratory of Fixed Prosthodontics Department, Faculty of Dentistry, Cairo University, Egypt according to the recommendations and approval of the ethics committee on in-vitro research. Sample size calculation was performed using G*Power version 3.1.9.22 then 4 samples were added to the total sample size to give sum of 30 tested surfaces to be divided evenly to each subgroup. Based on a study by Lauvahutanon., *et al*. 2017 [1] using an alpha (α) level of 0.05, a beta (β) level of 0.20. The sample size (n) was calculated with a total of 22 samples i.e. (11) for each group. Sample size was increased by 20% to be a total of 26 samples i.e. (13) for each group. Sample size calculation was performed then 4 samples were added to the total sample size to give sum of 30 samples to be divided evenly to each subgroup.

Sample grouping

A total of 30 samples were used in the present study. The samples were divided into two main groups according to the surface finishing protocol.

- **Group I:** Glazed Zirconia reinforced lithium silicate: Celtra Press (n = 15)
- **Group II:** Polished Zirconia reinforced lithium silicate: Celtra Press (n = 15)

Each subgroup was further subdivided into three subgroups according to the media tested as follows:

- **Subgroup A:** Samples immersed in artificial saliva (n = 5)
- **Subgroup B:** Samples immersed in Coffee (n = 5)
- **Subgroup C:** Samples immersed in Carbonated orange Juice (n = 5)

Samples preparation

A 10 mm diameter and 6.5 mm thickness cylinder was designed using Exocad software, saved as STL File. The STL file was transferred to the K5+ five-axis milling machine (VHF Cam facture AG, Germany) and was milled from CAD wax (BiLKim Co. Ltd., Turkey) producing a wax cylinder with predetermined dimensions. Pressing of the cylinders was done according to manufacturer's instructions using Celtra press MT Shade A2 ingots (Dentsply Sirona, Germany) in Ivoclar Programat EP 3010 Furnace (Ivoclar Vivadent Liechtenstein, Germany). Divesting was done by 110-micrometer aluminium oxide. Each ceramic cylinder was cut into 3 discs of a thickness (1.5mm±0.2mm) using IsoMet4000 (Buehler, Lake Bluff, USA). The measurements were then confirmed with a Digital caliper giving a sum of 15 ceramic discs. All discs were subjected to finishing using fine grit diamond stone according to manufacturer instructions. Then according to the protocol suggested by, [2,3]. The discs were glazed on one side and polished on the other side resulting in 15 glazed and 15 polished surfaces. A marginal notch with a diamond stone was made on the side that will receive polishing to be able to differentiate between the polished and the glazed sides. For glazing, a considerable amount of Celtra glaze (Dentsply Sirona, Degu Dent GmbH, Germany) was placed on a mixing palette and diluted with glaze liquid to obtain a thinner consistency. An evenly covering layer of glaze was applied to the entire single surface of each disc using a clean brush. The discs were then placed on a honeycomb firing tray in Ivoclar programmat CS 3 for glazing. Disc polishing was done with a dental surveyor '' surveyor II '' (Saeshin Precision Co., Ltd., Korea) for standardization. A cast model was designed to be fixed on the surveyor during the polishing of the discs. This cast has an empty mold inside to fit a silicone mold that will hold the discs during polishing (Figure1). A gypsum base was fabricated to hold the cast parallel to the straight handpiece. The selected polishing instrument was mounted in a straight low-speed handpiece of the micromotor that was fixed to the upper member of the surveyor in such a way that the strokes of the stone attached to the handpiece were parallel to the long axis of the disc (Figure 2). Using Vita Suprinity Polishing Technical Set (VITA Zahnfabrik H. Rauter GmbH and Co. KG, Germany), the discs were polished with pink pre-polishing stones with a speed of 10.000min$^{-1.}$ High gloss grey polishing stones were used with a speed of 6.000 min^{-1} according to the manufacturer's instructions to produce high surface lustre.

Figure 1: Cast model with the silicone mold inside to hold disc during polishing.

Baseline measurements

Glazed and polished discs were measured before immersion in aging solutions to give baseline readings. Regarding surface roughness, Measurements were done using optical profilometry (Guangdong, China) that tends to fulfil the need for quantitative characterization of surface topography without contact. Qualitative evaluation of the surface morphology of the discs was done

Figure 2: The whole assembly on the surveyor.

using a Scanning electron microscope Quanta™ FEG (FEI Company, United states). For Color change measurements, the disc color was measured using a reflective spectrophotometer model RM200QC (X-Rite GmbH, Neu-Isenburg, Germany) that was calibrated before each measurement. Three measurements were taken for each disc and the average was recorded.

Immersion in different oral pH media

The discs from each tested group were randomly divided into 3 subgroups (n = 5) according to the immersion medium (artificial saliva, coffee, and carbonated orange juice). The pH of the solutions was measured using a pH meter (Szeged, Hungary) and determined to be 6.8, 5.4 and 3.5 for artificial saliva, the coffee solution, and the carbonated orange juice, respectively. The discs were immersed individually in closed vials containing 5 mL of each immersion medium and stored in an incubator(Medical Equipment, Torre de' Picenardi, Italy) at 37°C for 12 days which was equivalent to 1 year of consumption of beverages according to the protocol followed by [4,5]. The solutions were changed every 12 hours to avoid yeast or bacterial contamination. To reduce the precipitation of particles in the staining solutions, the solutions were stirred twice a day. At the end of the immersion period, the discs were rinsed with distilled water and wiped with gauze.

Post-immersion measurements

Both the surface roughness and the color of each disc were assessed after employing different aging protocols as described for the baseline measurements. Data were collected, tabulated, and statistically analysed.

Results

Results of the effect of immersion solution on the surface roughness of both tested surface finish groups (Ra difference)

Regardless to surface finish protocol, the difference between the effect of immersion solutions was statistically non-significant (P = 0.2059 > 0.05) as indicated by two-way ANOVA where (Coffee ≥ Fanta ≥ AS)

Results of the effect of surface finish protocol (glazed versus polished) on the surface roughness when immersed in each solution

Irrespective of immersion solutions the difference between surface finish protocol was statistically non-significant (P = 0.5209 > 0.05) as indicated by two-way ANOVA where (Glazed ≥ Polished).

Variables		Surface finish								Statistics	
		Polished				Glazed				t-test	
		Mean	SD	95% CI		Mean	SD	95% CI		t- value	P value
				Lower	Upper			Lower	Upper		
Immersion solutions	Coffee	0.0011	0.0017	-0.0009	0.0032	0.0051	0.0024	0.0021	0.0080	1.95	0.06ns
	Fanta	0.0007	0.0031	-0.0031	0.0046	0.005	0.0102	-0.0076	0.00176	0.8	0.24ns
	A. S	-0.0003	0.0014	-0.0021	0.0014	0.0006	0.0009	-0.0005	0.0017	1.6	0.193 ns
Statistics	ANOVA	F value		0.6		F value		0.9			
		P value		0.5724 ns		P value		0.4277 ns			

Table 1: Results of the surface roughness average change (Mean ± SD) for both groups as a function of immersion solutions.

Different superscript capital letter in the same column indicating a statistically significant difference between different immersion solutions (p < 0.05) CI; confidence intervals. *; Significant (p < 0.05) ns: Non-Significant (p > 0.05).

Figure 3: Column chart of the mean values of surface roughness average change (Ra difference) for both groups as a function of immersion.

Results of the effect of immersion solution on the color stability for each surface finish tested groups (ΔE)

Regardless to surface finish protocol group, the difference between immersion solutions was statistically non-significant (P = 0.1811 > 0.05) as indicated by two-way ANOVA where (Coffee > Fanta > A.S).

Results of the effect of surface finish protocol (glazed versus polished) on the color stability when immersed in each solution

Totally, irrespective of immersion solutions the difference between surface finish protocol was statistically significant (P = 0.0004)

D images of Optical Profilometer Surface Roughness

3D images of the surface roughness change pre and post immersion in aging solution were captured where the changes were seen in the difference in heights of the peaks and depth of the valleys across the surface. In both glazed and polished surfaces, discs immersed in coffee gave the highest roughness followed by Fanta and Finally the lowest roughness difference in the artificial saliva immersed discs. However, these differences were statistically insignificant (Figure 5 and 6)

Results of SEM testing

The SEM image of the glazed sample before immersion (a) showed homogenous surface morphology due to the glaze layer covering

Variables		Surface finish								Statistics	
		Polished				Glazed				t-test	
		Mean	SD	95% CI		Mean	SD	95% CI		t- value	P-value
				Lower	Upper			Lower	Upper		
Immersion solutions	Coffee	4.35^{A}	1.36	2.6	6.05	5.4^{A}	1.1	4.07	6.78	3.8	0.02*
	Fanta	3.21^{AB}	0.82	2.19	4.23	3.42^{B}	1.41	1.67	5.17	0.5	0.64 ns
	AS	1.9^{B}	0.3	1.6	2.25	2.55^{B}	0.37	2.09	3.01	7.8	0.001 *
Statistics	ANOVA	F value		8.6		F value		9.8			
		P value		0.004*		P value		0.003*			

Table 2: Results of color change (Mean ± SD) for both groups as a function of immersion solutions.

Different superscript capital letter in the same column indicating statistically significant difference between different immersion solutions. (*p* < 0.05)CI; confidence intervals *; Significant (*p* < 0.05) ns; Non-Significant (p > 0.05).

Figure 4: Column chart of the mean values of color change (ΔE) for both groups as a function of immersion solutions.

the surface entirely which made the crystal hardly distinguished. Sample immersed in artificial saliva (b) demonstrated the least surface morphology change compared to sample imaged before immersion. While regarding sample immersed in coffee(C), it showed obvious surface change seen as numerously scattered patches and small pores that might indicate high surface roughness, followed by sample immersed in Fanta (d) that showed dispersed porosities all over the image. However, the quantitative surface roughness values were statistically insignificant (Figure 7).

Also, the SEM of the polished samples showed that the crystals are clearly seen in this magnification (a). The lowest surface morphological change was seen in the sample immersed in artificial saliva (b). Regarding the sample immersed in coffee(c), the morphology showed some patches and numerous porosities dispersed all

Figure 5: Images showing Surface roughness of glazed samples a) before A.S immersion, b) after A.S immersion, c) before coffee immersion, d) after coffee immersion, e) before Fanta immersion and f) after Fanta immersion.

Figure 6: Images showing surface roughness of polished samples a) before A.S immersion, b) after A.S immersion, c) before coffee immersion, d) after coffee immersion, e) before Fanta immersion and f) after Fanta immersion.

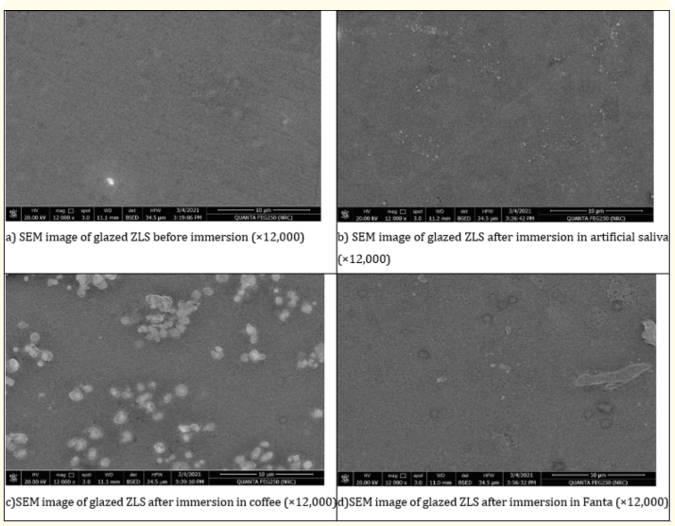

Figure 7: Scanning electron microscopy images of Glazed pressed ZLS. a) Before immersion, b) After Artificial saliva aging, c) After coffee aging and d) After Fanta aging. (Magnification × 12,000).

over the surface, fewer pores are seen on the sample immersed in Fanta (d). However, the quantitative surface roughness values were statistically insignificant (Figure 8).

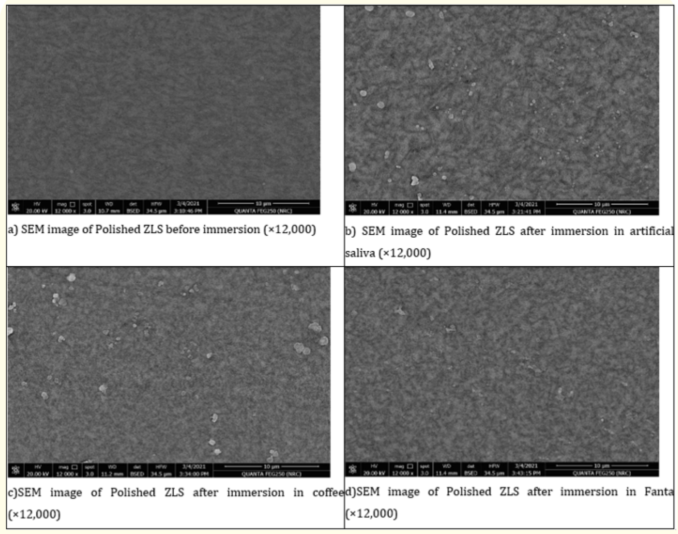

Figure 8: Scanning electron microscopy images of polished pressed ZLS. a) Before immersion, b) After Artificial saliva aging, c) After coffee aging and d) After Fanta aging. (Magnification × 12,000).

Discussion

The use of all-ceramic restorations increased owing to their excellent physical and chemical properties, [6]. Surface roughness and color stability play an important role in the success of different dental restorations, however, there are concerns about their long-term survival in the oral environment [7].

In the present study, Celtra press ceramic was selected as it was claimed to combine the excellent aesthetics of glass-ceramics with the strength of zirconia which enhance the material mechanical and optical properties [8]. Celtra Press is supplied as ingots for a hot-pressing technique which is considered a relatively inexpensive technique and dental technicians are familiar with it. Also, several studies stated that pressed restorations are characterized by decreased porosity, increased flexural strength and excellent marginal fit [9,10]. Surface finishing protocols are important for reducing the roughness of adjusted ceramic surfaces decreasing the plaque retention, prevent discolouration and wear of opposing teeth which in turn will maintain the longevity and natural appearance of the restorations [11].

One of the factors that might affect the quality of the restorations is the consumption of beverages such as coffee, tea, soft drinks. Many studies have shown that liquids consumed because of dietary habits cause a color change in restorative materials and affect surface roughness at different rate [12].

In the present study, wax cylinders were milled from CADwax blank which show advantages of being efficient and standardized method for wax pattern production [13]**.** The diameter of the wax cylinders was chosen to be 10 mm to be easily handled during glazing and polishing steps [14,15].

A 4 mm tapered sprue was attached at 90°angle in the direction of flow and at the thickest point of the cylinder to ensure complete pressing according to manufacturer instructions, Then, the patterns were heat pressed according to the protocol mentioned by the manufacturer's instructions. The samples were sectioned using Isomet 4000(Buehler, Lake Bluff, USA) with speed of 2,500 rpm under water coolant that offers low kerf loss and standardized thickness of each sample [8]. All the discs were glazed on one side and polished on the other side which make use of both surfaces without affecting color and surface roughness measurements [3].

The Vita Suprinity Polishing Sets was used as it offers reliable, efficient, and material-specific surface treatment of zirconia reinforced lithium silicate ceramic restorations. To achieve standardized polishing, a dental surveyor was used with a low-speed straight handpiece that allowed the strokes of the stone attached to the handpiece to be parallel to the long axis of the sample. Moreover, the arm of the surveyor that holds the straight handpiece was moved in such a way that the stone was kept touching the surface of the specimen for 60 seconds to perform polishing. The samples were polished in one direction by using the same low-speed straight handpiece [16,17].

Immersion in commonly consumed staining solutions including Coffee, carbonated orange juice as an aging method was carried out to simulate the physiological aging of biomaterials. Specific interest in selecting these agents (Coffee and Fanta) because they are the most consumed soft and hot beverages. Fanta consists of carbonic and citric acid and is a highly acidic soft drink, these acids can decalcify the tooth structure with time causing degradation, ionic dissolution and release of alkaline lithium and aluminium ions, also, result in dissolution of the ceramic silicate network that directly increase the stainability of the restorations in conjugation with the coloring pigments on the beverage [18]. Whereas coffee consists of acidic contents that might cause elemental dissolution of ceramics due to their chelating effect [19]. Both beverages cause significant changes in the color and surface roughness of different restorative materials as reported by several invitro studies [20]. Several reports have investigated different polishing techniques for ceramic restorations to create smooth surfaces, such as glazing, and support the use of polishing as an alternative to glazing [21]. In the present study, artificial saliva was selected over distilled water as a storage medium to simulate the oral environment.

An optical profilometer is an appropriate instrument used for the quantitative evaluation of surface roughness. Scanning electron microscope has been advocated to complement the qualitative analysis of the surface roughness [22]. The mean roughness value (Ra) was the parameter mostly used by many authors evaluating the effect of the different protocols of ceramic finishing [23].

For the color assessment, spectrophotometer was used as it eliminates the subjective interpretation of visual color comparison and can detect any slight color changes in dental materials during clinical work and in research [24]. The CIEL*a*b* system was selected as it is commonly used and reported in the literature, allowing for comparison with previous similar studies on color of dental materials [25]**.**

The samples were stored at a constant temperature of 370c in an incubator to simulate the temperature in the oral cavity. The samples were immersed in these different solutions for 12 days, simulating one year of clinical aging according to the protocol introduced by [4].

The first null hypothesis stating that there would be no difference in the effect of the tested surface finish protocol (glazing and polishing) and pH oral media on the surface roughness of zirconia reinforced lithium silicate ceramics was accepted.

The second null hypothesis stating that there would be no difference in the effect of the tested surface finish protocol (glazing and polishing) and pH oral media on the color stability of zirconia reinforced lithium silicate ceramics was partially accepted.

Regarding the surface roughness results, there was a statistically non-significant difference in the mean Ra value of glazed and polished groups immersed in the tested oral media. These findings agreed with [26] who reported that the difference between polishing and glazing of zirconia reinforced lithium silicate groups on surface roughness was statistically non-significant.

On the contrary [18] found that glazed samples of pressed zirconia reinforced lithium silicate had lower surface roughness before and after immersion, compared with polished samples. The variation in the results might be due to the difference in methodology as the specimens were thermo-cycled in orange, cola and black tea, also due to difference in immersion time''125 hours''. Moreover [3] found that the glazed groups were reported with higher surface roughness and lower microhardness when compared to the polished groups before and after thermocycling for zirconia reinforced lithium silicate glass-ceramic, lithium disilicate glass-ceramic, and feldspathic glass-ceramic and this was related to an initial higher roughness value of the glazed samples.

The collected data must be related to clinical requirements for better outcome understanding. Superficial roughness greater than 0.5 μm can be detected by the sensorial fibres of the tongue, resulting in discomfort for the patient [27]**.** So, in the present study, polishing and glazing allowed ZLS samples to be imperceptible by the tongue [16]**.** When referring to the clinical acceptability of the finished surfaces, all the Ra values measured in the present study were far below the abrasive wearing threshold (1.5 μm), [28].

The results of color change in this study revealed that regardless to surface finish protocol the difference between immersion solutions was statistically non-significant ($P = 0.1811 > 0.05$) as indicated by two-way ANOVA where (Coffee > Fanta > A.S). The insignificant results of the effect of immersion media on the zirconia reinforced lithium silicate in the present study could be attributed to the relatively small sample size, the insignificant change in the surface roughness and the short immersion time as prolonged immersion time in different pH media of ceramics could produce a more pronounced effect on the color stability. The amount of color change (ΔE) values greater than 1 were considered perceptible, whereas values greater than 3.7 were considered clinically unacceptable, according to the 50:50% threshold**.** [29,30], While according to [31,32] delta E of 5.5 were considered clinically acceptable.

These findings agreed with [33] who found that no significant difference was observed between the color stability values of e-max (control group), Vita Suprinity and Enamic after immersion in coffee. This insignificant difference may be attributed to the fact that Enamic contains 86% ceramic filler which may render the material to act like ceramic material than a resin material, as well as the Suprinity which contains only 10% zirconia filler. also, due to nearly similar immersion time used in the present study (15 days in this study).

Our findings disagreed with [25]. who stated that the color change (ΔE) values of lithium disilicate and zirconia reinforced lithium silicate increased significantly because of storage in different pH media ''tea and coffee''. this might be attributed to different immersion time used (2 months which corresponds to 5-year clinical usage of the ceramic *in-vivo*)

Also, the results of the present study revealed that irrespective of immersion solutions the difference between surface finish was statistically significant ($P = 0.0004 < 0.05$) as indicated by two- way ANOVA where (Glazed ≥ Polished).

This could be attributed to the deterioration occurred to glaze layer that may increase the stainability of the restoration. With the most considerable color change was in the glazed groups immersed in coffee, this might be explained by the low pH as coffee contains approximately 22 types of acids including citric acid, acetic acid, malic acid, and other high molecular weight acids, the acidic pH of coffee solution might dissolve silica, with a consequent loss of alkaline ions and corrosion of the surface, which might have potentiated the degradation of the glaze that subsequently increases the stainability of the ceramics [34]**.**

This was in agreement with [35] who found that glazed vita Suprinity samples had higher mean value of ΔE in comparison to polished samples stored in citric acid and this was claimed to occur because glazing produce rougher surface than polishing pre and post immersion that increase the stainability of the ceramics subsequently.

However, our findings were in disagreement with [14] who found that the color changes of polished samples were more recognized than those of glazed samples for all types of ceramics (Zirconia, Emax Cad, Emax Press). They attributed such findings to the polishing techniques that might leave the porcelain surface rougher and more porous which could lead to more significant color changes caused by staining beverages such as Coca-Cola.

Color change ΔE values above 3.7 were recorded in samples immersed in coffee which is clinically perceivable by human eye and thus is clinically unacceptable and unesthetic according to [36] while it was considered clinically acceptable according to the values suggested by [37].

Regarding our study, some limitations were noticed as the sample size was relatively small, it is an in-vitro study which does not simulate all clinical conditions.

Conclusion

Within the limitations of the present in-vitro study the following conclusions were drawn

- Surface finish protocol (glazed vs. polished) as well as tested immersion media (artificial saliva, coffee and carbonated orange juice) showed no effect on the surface roughness of zirconia reinforced lithium silicate ceramics.
- Color stability of zirconia reinforced lithium silicate ceramics was significantly affected by the surface finish protocol. While the pH immersion media showed no effect on the color stability.
- Coffee produced significant color change with the glazed ZLS group compared to polished group, both values were above the clinically acceptable limit (ΔE > 3.7).

Recommendations

Further investigations are required to

- Evaluate the behavior of Celtra press in-vivo as the oral environment presents much more complex environment, the continuous change in temperature, pH, and different types of abrasive food, all that can affect the color and surface topography of the material.
- Evaluate the surface roughness and color stability of glazed versus polished heat pressed zirconia reinforced lithium silicate restorations after a more prolonged aging time to simulate more years of clinical severability of the restoration.

Conflict of Interest

The authors have no conflict of interest to disclose.

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