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Research Article

Effect of Surface Finish and Acidic pH Media on the Surface Roughness and the Color Stability of Zirconium Reinforced Lithium Silicate Glass Ceramics (An *In-Vitro* Study)

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Abstract

Purpose: The aim of this study was to determine the effect of surface finish (Polishing and Auto-glazing) and the effect of acidic pH media on the surface roughness and color stability of Vita Suprinity.

Materials and Methods: A total of 76 Vita suprinity plates of 3mm thickness were cut by an isomet cutting machine. All specimens were sintered and auto-glazed according to manufacturer's instructions. Half of the specimens was finished using a coarse grit diamond stone and then polished using the special polishing kit for Vita Suprinity. The other half was left with its glazed surface without any surface treatment. The samples were stored in 2% citric acid solution and artificial saliva as a control for 8 hours and 14 days respectively in an incubator at 37°C where the saliva was changed every 2 days. Color stability and surface roughness were measured before storage as baseline readings and after storage where the color stability was measured by using spectrophotometer. Surface roughness was calculated by using stylus profilometer.

Results: It was found that: Glazed samples stored in artificial saliva have lower mean value of ΔE and higher mean of surface roughness than polished samples stored in artificial saliva; Glazed samples stored in citric acid have higher mean value of ΔE and higher mean of surface roughness than polished samples stored in citric acid.

Conclusions: From the findings of our study, Ageing of Vita suprinity in acidic pH media (Citric acid solution) negatively affected both the color stability and surface roughness of Vita Suprinity. Surface finish was greatly affecting the surface roughness of Vita Suprinity.

Keywords: Vita Suprinity; Color Stability; Surface Roughness; Citric Acid; Surface Finish

Abbreviations

ZLS: Zirconia-Reinforced Lithium Silicate Glass Ceramic; CAD/CAM: Computer Aided Designing/Computer Aided Manufacturing; SD: Standard Deviation; CI: Confident Interval; IBM SPSS: International Business Machines Statistical Package for the Social Sciences; P value: Probability; Ra: Roughness Average; SEM: Scanning Electron Microscope; EDXA: Energy Dispersive X-Ray Analysis

Introduction

The increase in patient esthetic demands resulted in the development of number of metal-free fixed prosthesis. Zirconia based ceramics have good mechanical properties and biocompatibility. However their optical behavior is still controversial. Initially, zirconium oxide was considered as an opaque material, due to its high refractive index and high opacity in the visible and infrared regions of the spectrum. Thus the surface finish and the acidic pH represent important factors affecting the color matching to natural dentition. Little is known about the surface characteristics of monolithic ceramic materials in glazed and polished forms, and after their surface finishing and polishing with different intraoral systems. Glass ceramic restorations are popular amongst clinicians because of their superior aesthetic properties. In the last decade, zirconia has generated tremendous interest due to its favorable mechanical and biological properties. However, zirconia lacks the translucency that lithium disilicate materials possess and therefore has limitations in its use, especially in esthetically demanding situations. There has been a great thrust in research towards developing translucent zirconia materials for dental restorations [1,2]. Currently, there are several ceramic materials, including glass based ceramics, crystalline dominated ceramics, polycrystalline ceramics and hybrid ceramics that meet these requirements. Each type has different physical and optical properties. Among dental ceramic materials available nowadays is the Vita Suprinity which is used for the construction of single restorations together with long span bridges. This material facilitates and enables the production of esthetical, high-quality dental prosthesis, with satisfactory results for both the patients and the clinicians. Vita suprinity is a zirconia-reinforced lithium silicate glass ceramic (ZLS) for dental CAD/CAM applications for the fabrication of inlays, onlays, partial crowns, veneers, anterior, posterior crowns, anterior and posterior single tooth restorations on implant abutments.

Material and Methods

Samples preparation: A total of nineteen (19) blocks of Vita Suprinity (Figure 1) were sectioned under water coolant with a precision cutting machine Isomet 1000 (Buehler, Lake Bluff, USA) into seventy six (76) plates with dimensions 3 x 12 x 14 mm.

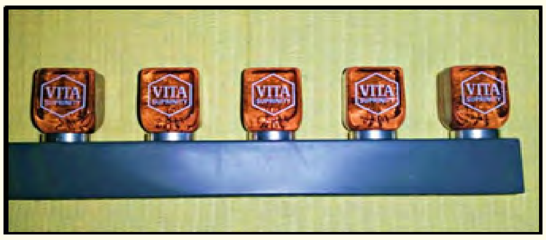

Figure 1: Vita Suprinity blocks.

Group allocation: 76 samples were divided into to 2 main groups according the type of surface treatment (auto-glazed or polished). Each main group was divided into 2 subgroups according to the type of the storage media (2% citric acid or artificial saliva).

- **Group 1 (control group):** Includes 19 auto-glazed vita suprinity samples that will not receive surface finish and stored in artificial saliva.
- **Group 2:** Includes 19 vita suprinity samples that will receive surface finish (selective grinding then polishing) and then stored in 2% citric acid solution.
- **Group 3:** Includes 19 auto-glazed vita suprinity samples that will not receive surface finish but only stored in 2% citric acid solution.
- **Group 4:** Includes 19 vita suprinity samples that will receive surface finish (selective grinding then polishing) and then stored in artificial saliva.

Preparation for surface treatment: All the specimens were sintered and then auto-glazed according to manufacturer's instructions [3]. All samples were numbered randomly from 1 - 76 with the aid of the website (http://www.random.org) to avoid bias. To stimulate intra-oral adjustment procedures, half of the specimens was finished using a coarse grit diamond stone and then polished by a single operator using 2-steps diamond polishing system (Vita suprinity polishing set clinical, VITA Zahnfabri, Germany) using a low-speed handpiece according to the manufacturer's instructions. The specimens were washed using distilled water in a digital ultrasonic cleaner. The other half was left with its glazed surface without any surface treatment.

Preparation for storage: Group 2 and Group 3 were stored in 2% citric acid solution for 8 hours which simulates two years *in vivo* [4] while Group 1 and Group 4 were stored in artificial saliva as a control for 14 days in properly sealed testing tubes an incubator at 37oC where the saliva was changed every 2 days. The pH was measured with a pH meter (WTW Inolab pH meter Level 1, Wissenschaftlich-Technische-Werkstätten Company, Germany). All the pH measurements were performed three times [5]. The artificial saliva recorded average pH of 7 while the 2% citric acid solution recorded pH of 2. After storage, the specimens were rinsed with distilled water for 5 minutes and blotted dry with tissue paper before surface micro-hardness and fracture toughness measurement.

Measurement of color stability and surface roughness

A spectrophotometer was used to measure the shade of glazed vita suprinity samples; we measure the shade of plates before any finishing or polishing and before storage in any aging media resembling baseline reading. Then the shade of vita suprinity samples for each group was measured after surface finishing and polishing and after storage in aging media. (E) > 2.5 will be considered a color change that can be precepted by the naked eye [6]. All measurements were performed twice for reproducibility and standardization.

Figure 2: Spectrophotometer.

A stylus profilometer (Figure 3) was used to measure arithmetic mean roughness (Ra) of the surfaces. (Ra) is the arithmetic average height of roughness-component irregularities (peak heights and valleys) from the mean line, measured within the sampling length [7]. With a diamond stylus radius 5 m, angle of stylus 90o perpendicular to the specimens, and a cut off length of 0.25 mm. Roughness was measured at five different points and the mean surface roughness measurement was calculated for each sample. Surface roughness measurements for glazed and polished samples were performed before and after immersion in artificial saliva and citric acid.

Figure 3: A stylus profilometer.

Results

Statistical Analysis: Numerical data were explored for normality by checking the distribution of data and using tests of normality (Kolmogorov-Smirnov and Shapiro-Wilk tests). Color change data (ΔE), (ΔL) and (Δb) data showed parametric distribution while (Δa) data showed non-parametric distribution. Data were presented as mean, median, standard deviation (SD), minimum, maximum and 95% Confidence Interval (95% CI) for the mean values. For parametric data, repeated measures ANOVA test was used to compare between storage media as well as color parameters after immersion and after polishing. Tukey's post-hoc test was used for pair-wise comparisons when ANOVA test is significant. For non-parametric data, Mann-Whitney U test was used to compare between the two storage media. Wilcoxon signed-rank test was used to compare between (Δa) after immersion and after polishing. The significance level was set at P ≤ 0.05. Statistical analysis was performed with IBM SPSS Statistics Version 20 for Windows.

Color change (ΔE)

Comparison between storage media

Whether after glazing or after polishing, artificial saliva showed statistically significantly lower mean ΔE than citric acid.

- Glazed vita suprinity samples stored in citric acid solution (pH = 2) record significantly higher ΔE than glazed vita suprinity samples stored in artificial saliva (p < 0.001).
- Glazed followed by finishing and polishing vita suprinity samples stored in citric acid solution (pH = 2) record significantly higher ΔE than glazed followed by finishing and polishing vita suprinity samples stored in artificial saliva (p < 0.016).

Comparison between surface finish (glazed vs finished and polished)

As regards artificial saliva, there was a statistically significant increase in mean ΔE after polishing.

While for citric acid, there was a statistically significant decrease in mean ΔE after polishing.

- Glazed vita suprinity samples stored in citric acid solution (pH = 2) record significantly higher ΔE than glazed vita suprinity samples stored in artificial saliva (p < 0.001).
- Glazed followed by finishing and polishing vita suprinity samples stored in citric acid solution (pH = 2) record significantly higher ΔE than glazed followed by finishing and polishing vita suprinity samples stored in artificial saliva (p < 0.001).

Storage media	Surface finish	Mean	SD	Median	Minimum	Maximum	95% CI	
							Lower bound	Upper bound
Artificial saliva	After glazing	0.73	0.11	0.73	0.54	0.89	0.66	0.81
	After polishing	1.27	0.12	1.27	1.07	1.48	1.18	1.35
Citric acid	After glazing	2.03	0.15	2.11	1.72	2.20	1.93	2.13
	After polishing	1.41	0.13	1.41	1.22	1.62	1.33	1.50

Table 1: Descriptive statistics of ΔE values.

Roughness

For Glazed group

The highest mean ± SD values of roughness were recorded for base line group (0.25377 ± 0.0009 Ra) followed by citric acid group mean ± SD values (0.25245 ± 0.0010 Ra) meanwhile the lowest mean ± SD value was recorded with artificial saliva group (0.25197 ± 0.0008 Ra). The difference between aging solution groups was statistically significant as indicated by one way ANOVA (P = 0.0003 < 0.05). Pair-wise Tukey's post-hoc test showed non-significance between artificial saliva and citric acid groups as shown in in table 2.

For polished group

The highest mean ± SD values of roughness were recorded for citric acid group (0.25192 ± 0.0017 Ra) followed by artificial saliva group mean ± SD values (0.25167A ± 0.0012 Ra) meanwhile the lowest mean ± SD value was recorded with control group (0.25023B ± 0.0009Ra). The difference between aging solution groups was statistically significant as indicated by one way ANOVA (P = 0.0096 < 0.05). Pair-wise Tukey's post-hoc test showed non-significance between artificial saliva and citric acid groups as shown in in table 2.

Variables	Surface finish		Statistics
	Glazed	**Polished**	**P value**
Base line	0.25377^A^± 0.0009	0.25023^B^± 0.0009	< 0.0001*
A. saliva	0.25197^B^± 0.0008	0.25167^A^± 0.0012	0.4974 ns
Citric A.	0.25245^B^± 0.0010	0.25192^A^± 0.0017	0.3795 ns
Statistics (P value)	0.0003*	0.0096*	

Table 2: Roughness results (Mean values± SDs) for both surfaces finish groups as function of aging solution.

Discussion

Although Vita Suprinity may have many promising features, it is not fully tested yet. It was claimed that the ceramics were used for its unexceptional chemical stability. However, previous studies found out that the consumption of certain beverages such as acidic food, coffee, tea, soft drinks, alcoholic beverages and even fluoridated water may affect the quality of the restorations. The effect of these beverages on the color stability of the restorative materials varies depending on its intrinsic feature such as their chemical composition.

In this study, citric acid was used as an ageing solution because of its frequent consumption in daily life as it presents in many foods and beverages as lemon, limes, oranges, grapefruits, kiwis, strawberries, apples, pears, cherries and raspberries and vegetables such as mushrooms, potatoes, tomatoes, peas and asparagus. The storage period for citric acid was 8 continuous hours with 2% concentration at 37oC, while it was 14 days for artificial saliva as it simulates 2 years *in vivo* [4].

Although water has been a commonly employed storage medium in *in-vitro* studies, in this study artificial saliva was selected over distilled water as a storage medium to simulate the oral environment and provide data closer to reality and reproduce clinical situations [8].

In the present study the specimens were sectioned with the Isomet 4000 [9,10]. The saw cut the materials with minimal specimen deformation and low kerf loss that can ensure standardized thickness for all 76 specimens, thus overcome any optical alterations that could occur due to change in thickness [11].

Polishing was performed for the specimens according to manufacturer's instructions, and regarding the evidence proving that stain resistance and color stability is improved with properly polished surfaces [12,13]. It was also reported that materials with different microstructures require different polishing techniques [14], considering the difference between both groups being tested. Each specimen was polished with the polishing kit recommended by the manufacturer to mimic the clinical situation.

The Vita suprinity was polished via its Vita suprinity polishing kit recommended by the manufacturer for a total of 60 seconds per surface following the protocol followed by Flury., *et al* [15]. The tested parameters were evaluated by immersion in citric acid and artificial saliva as a control. These solutions are frequently ingested by the society and have different staining and erosive capabilities. Citric acid with very low pH value (2) was reported to cause surface degradation of resin matrix and surface erosion of the filler particles [16], and for glass ceramics. Citric acid has also been proven to have more titrable acidity than other acidic commonly consumed beverages such as Coca Cola (the more titrable the acidity, the faster the saliva can neutralize it) [17].

Artificial saliva was chosen instead of human whole saliva to minimize the inter-individual variation in salivary protein content, and their instability extra-orally [18]. Artificial saliva was accurately proportioned according to the protocol followed by Wongkhantee., *et al* [19].

Instrumental measurements eliminate the subjective interpretation of visual color comparison [20]. Many devices are used for color measurements. Such as the digital devices, the colorimeters and the spectrophotometers. Some digital devices may have good reliability in shade recording, but there exists high variability among their accuracy, also may show low reliability under different illuminants [21]. The colorimeter is a relatively simple device. Based on three axes by using a filter simulating the human eye.

Spectrophotometer can analyze the principle components of series of spectra and have the ability to convert spectrophotometric measures into varies of measures. They have good reproducibility as opposed by colorimeters that are affected by the aging of their filters. Spectrophotometers have been used as a viable instrument in dentistry to obtain the proper shade selection [22,23] and in research to assess color stability of dental restorations, and have proven to be a rather sophisticated and accurate color measuring device [24] for this reason the spectrophotometer was used in this study.

The total immersion time was 8 continuous hours which is equivalent to 2 years of consumption of the beverages [25,26]. According to the protocol followed by Ertas., *et al.* [27] according to the manufacturer of the citric acid, the average consumption time for one cup of citric acid is 15 seconds, and among citric acid drinkers the average consumption quantity is 3.2 cups per day.

The samples were stored at a constant temperature of 370c in an incubator to simulate the temperature in the oral cavity, and prevent any alterations in the staining potency of the solutions due to temperature changes [25].

Douglas., et al. [28] reported that 50% of dentists could perceive color difference at 2.6 Δ E units, other studies reported that at Δ E = 5.5 50% of patients would require change of the restoration [25], other studies reported clinically acceptable differences to fall under Δ E = 3.7 [29].

However more recent studies reported color changes below Δ E = 1 are unperceivable to the naked eye, and 1 > Δ E > 3.3 require a skilled person to detect the difference, thus gaining clinical acceptance [28]. Regarding the fact that the tested materials are indicated for laminate veneer construction, which are deemed as highly esthetic restorations, Δ E = 3.3 was regarded as the cutoff point in this study for clinical acceptance.

Regarding samples within artificial saliva table 2, there was statistically significance difference in Δ E as it was significantly lower than Δ E of citric acid. This may be attributed to that the staining susceptibility of ceramics is directly related to the degree of water sorption [9]. Their ability to absorb water can also absorb fluid with pigments, thus acting as a vehicle for stain penetration into the bulk of the material [30]. Regarding samples within citric acid, Ruyter., *et al.* (1991) [31] showed that citric acid contained yellow colorants with lower polarity leading to discoloration due to adsorption and absorption of the pigments into material [29].

Surface roughness

In this study the stylus profilometer was used in measurement of surface roughness due to its surface independence as contacting the surface is often an advantage in dirty environments where non-contact methods can end up measuring surface contaminants instead of the surface itself and so this method is not sensitive to surface reflectance or color. Its resolution is excellent as the stylus tip radius can be as small as 20 nanometers so can measure thin films, significantly better than white-light optical profiling. Vertical resolution is typically sub-nanometer as well. Stylus profilometer is a direct technique so no modeling is required. It allows creation of clear wave profile and long distance measurement up to 200mm to analyze thin film stress.

Stylus profilometer also allows excellent measurement repeatability, ease of use (fast, simple, step heights), lower cost, long life, durable and upgradeable.

According to storage media [32], the glazed vita suprinity samples stored in citric acid (pH = 2) record higher surface roughness than glazed vita suprinity samples stored in artificial saliva. Also glazed followed by finishing and polishing vita suprinity samples stored in citric acid record higher surface roughness than glazed followed by finishing and polishing vita suprinity samples.

This weight loss was confirmed by the quantity of ions released in the solutions. At acidic pH = 2, there was a significantly greater ion release from finished and polished samples than from glazed samples. This included network modifiers (Ca, Zn, Li) as well as Si^{+4}, the primary network former. The concentrations of Li, Zn, and Si released from the glaze were similar at acidic pH 2. The release of Ca from the glaze did not follow this trend. In fact, the release rate of Ca at a pH of 2 was comparable with the release rate of Si.

Conclusion

Within the limitations of this present *in vitro* study the following conclusions were drawn:

1. The color and surface roughness were altered by the type of storage media and surface finish.
2. Irrespective to the type of storage media glazed followed by finished and polished vita suprinity samples was greatly affected than glazed vita suprinity samples in terms of color change.
3. Acidic pH solutions (pH = 2) affect the color stability of vita suprinity samples than artificial saliva.
4. Irrespective to the type of storage media glazed vita suprinity samples was greatly affected than glazed followed by finishing and polishing vita suprinity samples in terms of surface roughness.

Recommendations

- The role of saliva in diluting the pH value of the solutions was not considered, furthermore, the oral environment presents much more challenges and a much more complex environment, the continuous change in temperature, pH, different types of abrasive food, all that can affect the color and surface topography of the material. Therefore further studies are needed to evaluate the behavior of Vita suprinity ceramic *in vivo*.
- Energy dispersive x-ray analysis (EDXA) is required to detect the leached ions before and after storage.
- Further studies are needed to evaluate the surface roughness of vita suprinity samples by using scanning electron microscope (SEM).

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